AutoPore IV 9500

Operator's Manual
V1.09

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2. If an instrument or product is found defective during the warranty period, replacement parts may, at the discretion of MICROMERITICS, be sent to be installed by the purchaser, e.g., printed circuit boards, check valves, seals, etc.

3. Expendable items, e.g., sample tubes, detector source lamps, indicator lamps, fuses, valve plugs (rotor) and stems, seals and O-rings, ferrules, etc., are excluded from this warranty except for manufacturing defects. Such items which perform satisfactorily during the first 45 days after the date of shipment are assumed to be free of manufacturing defects.

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PRECAUTIONS

It is important that you read these precautions before operating the AutoPore IV Porosimeters.

» Generation of hydraulic pressures in excess of 60,000 psia (414 MPa) on the 9510 and 9520 and 33,000 (228 MPa) psia on the Models 9500 and 9505 should never be attempted, even though all critical components are designed to withstand slightly higher pressures. Two features are built in to limit operation to, or near, the design level. First, the control module is programmed to terminate pressure generation at 60,000 psia on the Models 9510 and 9520, and 33,000 psia on the Models 9500 and 9505. Second, a relief valve is incorporated in the high-pressure system. These two safety provisions must not be altered; otherwise, damage to the instrument and physical danger to the operator and those in the immediate area of the instrument could result.

» Do not attempt to unscrew a high pressure chamber closure without first releasing its vent valve. Sufficient negative pressure could be generated to cause leaking around an internal seal.

» Do not attempt to open a high pressure chamber or release the vent valve on top of the presssure chamber when the internal pressure is greater than atmosphere. Doing so will result in a discharge of hydraulic fluid with considerable force.

» Accumulated mercury vapor is considered potentially harmful. The AutoPore IV is provided with an exhaust port that draws air across the work space and from within its cabinet when attached to a duct which pulls air to an exhaust system.

» Any mercury spilled on the counter tray should be wiped into the drain hole in the tray, from which it will fall into a collector and be covered by a layer of oil.

» The instrument must be unplugged before any panel is removed to avoid the hazards of high voltages which are present.

» No situation is known where pressure has caused an explosion or other dangerous reaction in a material while being evaluated by mercury porosimetry. Nevertheless, it is well to be aware of such a possibility should azides or perchlorates, for example, be considered for testing.

» When performing a low-pressure operation, make sure all retaining knobs are securely tightened. It is also recommended that capacitance detectors be installed on *all* ports, used and unused. This is to prevent the possibility of the penetrometer being expelled when the system pressure is raised to as high as 50 psia.

» Should a penetrometer be broken and mercury spilled in a high pressure chamber, the glass and mercury should be removed immediately.

» Should operator error or malfunction draw mercury toward the vacuum system, the mercury will be collected in a protecting reservoir with a capacity sufficient to retain all the mercury in the system at one time. A warning buzzer will signal that mercury transfer has occurred. This reservoir should be drained immediately. If, instead, more mercury is added and the error persists, subsequent quantities of mercury cannot be retained. The vacuum pump and other components will then be subject to damage.

» The mercury supply reservoir located between the two high pressure chambers is sealed by a stopper cap. The cap must always be securely in place during a test; otherwise the run cannot be completed.

» The capacitance detectors used in the preparation and testing of samples are calibrated according to position of attachment and are not interchangeable.

» Solids are compressible in varying degrees, but the AutoPore IV makes no attempt to compensate for this factor. It is therefore possible for an indication of significant porosity to be generated in comparatively “soft” samples when, in fact, no pores actually exist.

» Operation in the manual mode permits great latitude; however, it must not be used without forethought and attention to consequences.

Additional cautions and warnings are included throughout this manual as follows:

Designates information concerning the possibility of bodily injury.

Designates information concerning the possibility of damage to the instrument or other property.

CHAPTER 1

GENERAL INFORMATION

- Organization of the Manual
- Conventions
- Equipment Description
- Overview of the AutoPore IV Analysis Process
- The Windows Interface
- Specifications

GENERAL DESCRIPTION

Organization of the Manual

This manual describes how to install, operate, and maintain the AutoPore IV Mercury Porosimeters with Windows software. The manual is divided into the following chapters:

Chapter 1 Provides a general description and specifications for the AutoPore IV.

Chapter 2 Provides unpacking and inspection information, and instructions for installing the software and hardware.

Chapter 3 Provides general operating instructions and instructions for working in the Windows software environment.

Chapter 4 Provides an introduction to the AutoPore software and instructions for creating sample and parameter files.

Chapter 5 Provides step-by-step instructions for performing low- and high-pressure analyses, and describes how to monitor analysis status.

Chapter 6 Describes the low and high pressure systems and their components. Also provides instructions for operating the AutoPore in manual mode, and for calibrating the instrument.

Chapter 7 Provides instructions for generating reports.

Chapter 8 Describes troubleshooting and maintenance operations.

Chapter 9 Gives details for ordering parts, supplies and accessories.

Appendix A Provides forms that may be copied and used to assist in input data preparation.

Appendix B Discusses the mercury porosimetry theory used by the AutoPore IV.

Appendix C Provides information on the proper handling of mercury.

Appendix D Gives information on the AutoPore's method for data reduction.

Appendix E Provides the format of exported data.

Appendix F Explains the maximum intrusion volume option and its use.

Appendix G Provides information on blank and sample compression corrections.

Appendix H Gives information on pore surface area computation.

Appendix I Lists error messages and gives probable cause(s) and corrective action(s) for each.

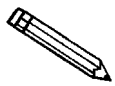
Appendix J Provides instructions for converting nonWindows Sample files to Windows-compatible files.

Appendix K Provides guidelines for choosing the proper pump-down rates for an unfamiliar sample material.

Appendix L Provides guidelines for setting up an analysis to acquire data for computing the volumetric compressibility of a sample material.

Conventions

This manual uses the symbols shown below to identify notes of importance, cautions, and warnings.



Notes contain important information pertinent to the subject matter.

Cautions contain information to help you prevent actions which could damage the instrument.

Warnings contain information to help you prevent actions which could cause personal injury.

Equipment Description

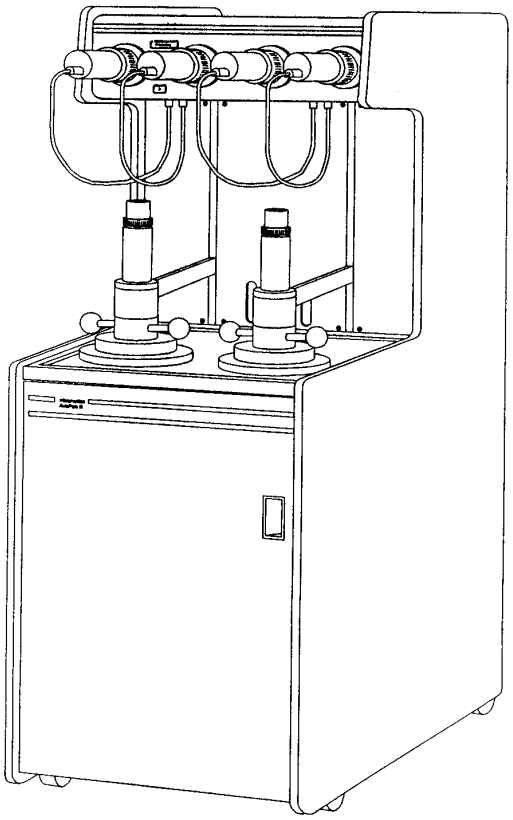


Figure 1-1. AutoPore IV Analyzer

The AutoPore IV Mercury Porosimeter is available in four configurations:

- **AutoPore IV 9520** is a 60,000 psia (414 MPa) mercury porosimeter covering the pore diameter range from approximately 360 to 0.003 µm. This model has four built-in low-pressure ports and two high-pressure chambers.
- **AutoPore IV 9510** has the same performance as the AutoPore 9520 except it features two low-pressure ports and one high-pressure chamber.
- **AutoPore IV 9505** is a 33,000 psia (228 MPa) mercury porosimeter covering the pore diameter range from approximately 360 to 0.005 µm. This model has four built-in low-pressure ports and two high-pressure chambers.
- **AutoPore IV 9500** has the same performance as the AutoPore 9505 except it features two low-pressure ports and one high-pressure chamber.

All aspects of low-pressure and high-pressure analyses, as well as data collection, reduction, and display are processed by the computer. The software and operator's manual are the same for all instruments, although the manual focuses on operation of the 9520. After initial setup of AutoPores 9510 and 9500, the software removes reference to the two unused low pressure ports and the unused high pressure chamber. Likewise, you should disregard references to the unused ports and chamber in the operator's manual.

Overview of the AutoPore IV Analysis Process

Before you can analyze a sample, you must (1) weigh the sample and record the weight and (2) create a sample information file 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. The convenient Windows software makes it easy to create and maintain sample files.

To begin the analysis, you load the sample into a penetrometer, then install the loaded penetrometer in a low pressure port. The first phase of the low pressure analysis is the evacuation of gases from the penetrometer. The penetrometer is then backfilled automatically with mercury. The second phase of the low pressure analysis is the collection of data at pressures up to the last low pressure point specified in the pressure table.

When the low pressure analysis is complete, you remove the penetrometer from the low pressure port and install it in a high pressure port. The high pressure analysis collects data at pressures indicated in the high pressure portion of the pressure table (up to 60,000 psia for Models 9520 and 9510; 33,000 psia for Models 9505 and 9500).

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 intrusion). Intrusion of different size pores occurs at different pressures. (The greater the pressure, the smaller the pore diameter into which the mercury can be forced.) Because mercury has a high surface tension and is nonwetting to most materials, its angle of contact and radius of curvature can be used to calculate the pore diameter into which it intrudes at a given pressure.

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

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

A thorough discussion of the theory of porosimetry is given in Appendix B.

The Windows Interface

The AutoPore IV Series of Mercury Porosimeters feature a Windows interface. Windows makes operation easier and allows you to run other applications on your computer while analyses are in progress. Two AutoPores can be operated from a single computer.

Specifications

The AutoPore IV has been designed and tested to meet the specifications listed below.

Characteristic	Specification
Low Pressure:	
Measurement:	0 to 50 psia (345 kPa)
Resolution:	0.01 psi (69 Pa)
Pore Diameter:	360 to 3.6 μm
Transducer Accuracy:	± 1% of full scale (transducer manufacturer's specifications)
High Pressure:	
Measurement:	
Models 9520/9510	From atmospheric pressure to 60,000 psia (414 MPa)
Models 9505/9500	From atmospheric pressure to 33,000 psia (228 MPa)
Resolution:	
Models 9520/9510	0.3 psi (2070 Pa) from 5000 psia (34 MPa) to 60,000 psia (414 MPa), and 0.1 psi (689 Pa) from atmospheric pressure to 5000 psia (34 MPa)
Models 9505/9500	0.2 psi (1400 Pa) from 3000 psia (21 MPa) to 33,000 psia (228 MPa), and 0.1 psi (689 Pa) from atmospheric pressure to 3000 psia (21 MPa)
Pore Diameter:	
Models 9520/9510	6 to 0.003 μm
Models 9505/9500	6 to 0.005 μm
Transducer Accuracy:	± 0.1% of full scale (transducer manufacturer's specifications)
Intrusion:	
Resolution:	Better than 0.1 μL
Accuracy:	±1% of maximum penetrometer stem volume
Penetrometers:	
Capillary Stem Intrusion Volumes:	0.38, 1.1, 1.7, 3.1, and 3.9 cm^3
Sample Size:	Maximum: a cylinder 2.5 cm diameter by 2.5 cm long (1 in. diameter by 1 in. long)

Characteristic	Specification
Utility Requirements:	
Voltage:	100/120/220/240 VAC ± 10%
Frequency:	50/60 Hz
Power:	600 VA maximum
Gas:	Nitrogen or other clean, dry gas at 50 psig (345 kPa)
Physical:	
Height:	143 cm (56.25 in.)
Width:	54.3 cm (21.38 in.)
Depth:	78 cm (30.75 in.)
Weight	250 kg (550 lb)
Computer Hardware and Software:	
Minimum Requirements:	Pentium 333 MHz or equivalent One CD ROM drive 64 megabytes of RAM 1-gigabyte hard drive 800 x 600 video display capability Windows NT (V4.00 or newer)

CHAPTER 2

INSTALLATION

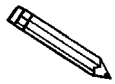
- Unpacking and Inspection
- Selecting the Location
- Installing the Hardware
- Filling the Mercury Reservoir
- Turning the System On and Off
- Installing the Analysis Program
- Interruption of the Program or Loss of Power

INSTALLATION

This chapter describes how to unpack and inspect the AutoPore IV. Your instrument will be installed and verified for operation by a Micromeritics representative or a representative of a Micromeritics distributor. A suitable location provided with proper utilities is required prior to installation.

Unpacking and Inspection

When you unpack the shipping cartons, carefully compare the packing list with the equipment actually received and check the equipment for any damage during shipment. Be sure to sift through all packing materials before declaring equipment missing.



If you need to declare equipment as damaged or lost, save the shipping cartons. The claims investigator must examine the cartons in order to complete the inspection report.

Equipment Damage or Loss During Shipment

If equipment is damaged or lost in transit, you are required to make note of the damage or loss on the freight bill. The freight carrier, not Micromeritics, is responsible for all damage or loss occurring during shipment. If you discover damage or loss of equipment during shipment, report the condition to the carrier immediately.

Equipment Return

Micromeritics strives to ensure that all items arrive safely and in working order. Occasionally, due to circumstances beyond our control, a customer may receive equipment which is not in working order. When equipment has been damaged (either during shipment or in use) and you wish to return the equipment to Micromeritics for repair or replacement, follow the steps below:

1. Pack the instrument in its original shipping carton if possible. If the original carton is unavailable, for a nominal fee, Micromeritics can provide another carton for your use.

Failure to package your instrument properly may result in shipping damage.

2. Tag or otherwise identify the defective equipment, noting the defect and, if possible, the circumstances under which the defect occurs.

3. Make reference to the sales order or purchase order for the equipment, and provide the date the equipment was received.

4. Notify the Micromeritics Service Department of the defect and request shipping instructions. The Service Department will assign a Return Material Authorization (RMA) number to your return and provide shipping information.

Selecting the Location

A floor area of at least 40 ft^2 (3.7 m^2) is recommended for the AutoPore to allow free access on all sides. This space might be allocated as 5 ft x 8 ft (1.5 m x 2.4 m).

The computer requires a space at least 36 in. (92 cm) wide x 23 in. (58 cm) deep x 18 in. (46 cm) high. This space will also accommodate the printer and may be as far as 6 1/2 ft (2 m) from the AutoPore.

The area should be well ventilated. Access to an exhaust hood or other external ventilation is strongly recommended. Flexible ducting of 5 in. (13 cm) inside diameter, similar to that often used with a home clothes dryer, is required if you want to vent the AutoPore to a suction fan. An optional exhaust fan kit is available (refer to **Chapter 9**, **Ordering Information**).

A nearby sink will make cleaning the penetrometer after analyses easier. Ready access to an analytical balance (with 0.1 mg resolution) for weighing filled penetrometers of up to 500 grams, and a drying oven for sample preparation is advantageous.

A gas supply (nitrogen or argon is recommended) with a regulator to provide up to 50 psig (345 kPa) is required.

Do not locate the instrument where air conditioning or heating vents can blow air directly on the instrument or where it is exposed to direct sunlight.

Installing the Hardware

These hardware installation instructions are not complete. They are not intended for first-time installation. They are intended for situations such as relocating the system. Contact your Micromeritics service representative for first-time installation.

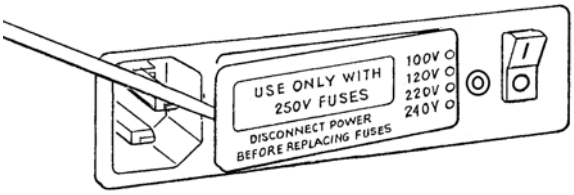
Selecting the Voltage

AutoPore

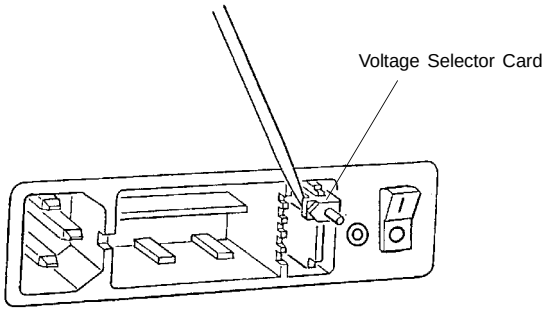
The AutoPore leaves the factory set for 120 VAC and with the line fuse removed. The correct setting of the universal power entrance must be checked and the appropriate fuse installed before the AutoPore can be operated. The AutoPore is designed to operate with either 100, 120, 220, or 240 VAC at 50 or 60 Hz. Voltage selection and fusing are made at the power connector located on the rear panel of the analyzer.

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.

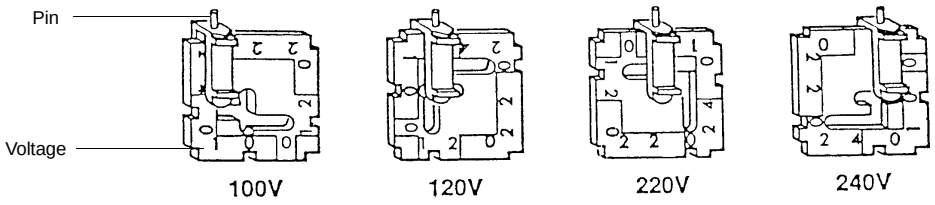
1. Make sure the power cord is disconnected from the analyzer.
2. Check the voltage setting on the rear panel of the analyzer; if the voltage is at the correct setting, skip to Step 9.
3. Using a pointed object, remove the fuse block and cover assembly from the power connector at the rear of the analyzer.



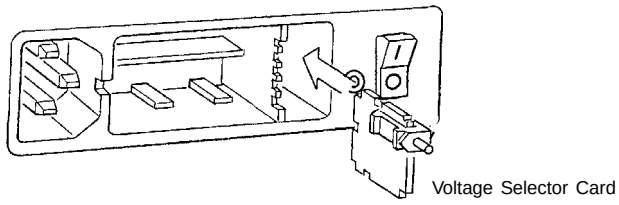
4. Pull the voltage selector card straight out of the power connector housing.



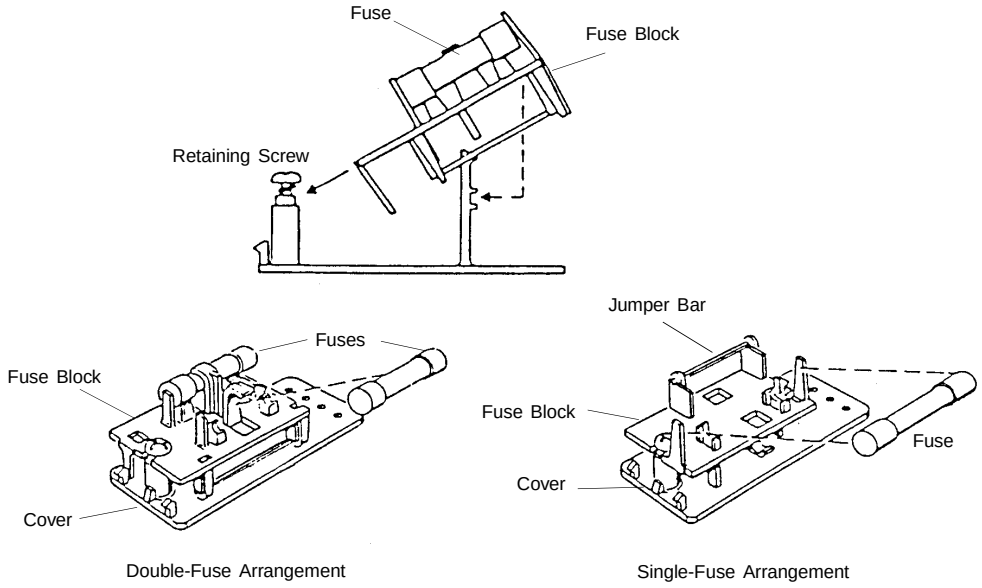
5. Orient the voltage selector card so that the desired voltage is indicated at the bottom. Orient the indicator pin so that it points upward as shown in the following illustration.



6. Insert the voltage selector card into the power connector housing with the edge containing the desired voltage first and with the printed side facing the power cord connector.



7. 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, as shown in the following illustration.



Double-Fuse Arrangement

Single-Fuse Arrangement

The power cord should be disconnected from the analyzer when installing or replacing fuses. Failure to do so could result in electrical shock.

a. Observe the position of the fuse block, using the previous figure for reference. If the single-fuse arrangement is desired, 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 desired, position the fuse block so that the side with the double-fuse slots is away from the cover.

b. If the fuse block is positioned properly for the fusing desired, proceed to Step c.

   If the fuse block is not positioned properly for the fusing desired:

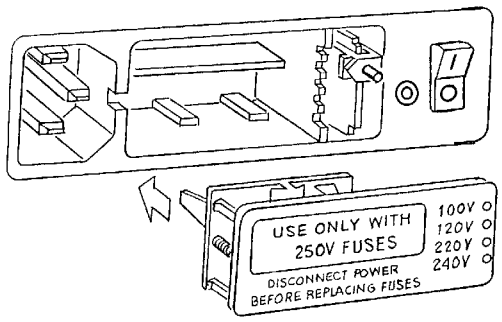
   1) Remove the fuse block retaining screw.
   2) Lift the fuse block from the cover.
   3) Rotate the fuse block.
   4) Mount the fuse block to the cover.
   5) Replace the retaining screw.

c. Insert appropriate fuse for the input power source. Refer to the chart below for the appropriate fuse rating.

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

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

8. Insert fuse block and cover assembly into input power connector (as shown in the following illustration) 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.



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

Computer

The line voltage of the computer must be set to match the input power source (from the wall outlet). The computer operates with either 100-120 VAC or 200-240 VAC at 50 or 60 Hz. Refer to the instruction manual supplied with your computer for instructions on selecting power input.

Do not connect the computer power cord to a power source until the proper voltage selection is made. Doing so could result in electrical shock and/or damage to the computer.

Connecting the Power Cables

For each device, connect the female end of the power cable to the power entrance. The location of the power entrance of the analyzer is shown in Figure 2-4. Plug the other end of each cable into an appropriate power source.

Connecting the Vacuum Pump

The AutoPore requires a vacuum pump for operation. The instructions provided here are for installation of the vacuum pump available from Micromeritics (refer to Chapter 10 for ordering information). These instructions may vary slightly if you are providing your own vacuum pump.

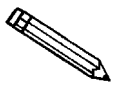
Perform the following steps to install the vacuum pump:

1. Prepare the vacuum pump (check oil, install exhaust filter, etc.) following the instructions provided in the vacuum pump manual.

2. Verify that the pump voltage (located on the side of the pump motor) matches the voltage of the power outlet to which the instrument will be connected. If the voltage does not match, contact your Micromeritics service representative for the appropriate action.

3. Place the vacuum pump on the vacuum pump tray in the lower right side of the analyzer. DO NOT connect the vacuum pump hose to the analyzer at this time.

4. Plug the vacuum pump power cord into the power outlet on the rear panel of the AutoPore (shown below).



Some vacuum pumps or vacuum pump cords may contain an ON/OFF switch. If this is the case, ensure that the switch is in the ON position.

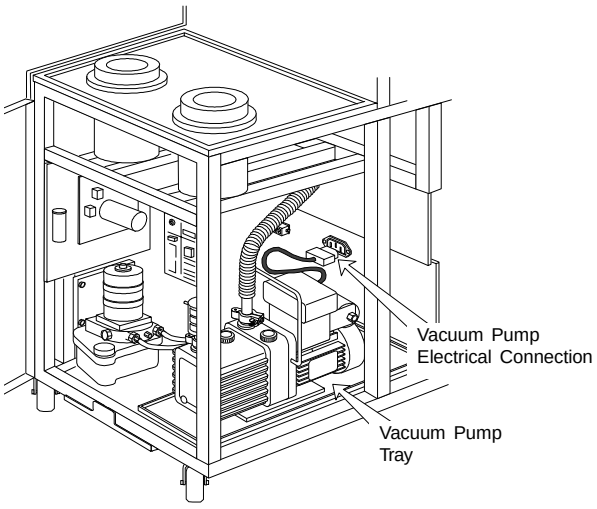


Figure 2-1. Vacuum Pump Connection

5. Connect the vacuum hose from the pump to the analyzer.

Connecting the Gas Supply

The AutoPore requires a supply of clean, dry air or gas, such as nitrogen or argon, at 50 psig. Regulators compatible with Micromeritics instruments can be ordered from Micromeritics. See Chapter 9 for ordering information.

1. Attach an appropriate regulator to the gas bottle. Leave the gas bottle shut-off valve closed until instructed otherwise.

2. If the regulator has a 1/8-in. Swagelok™ compression fitting on the outlet, proceed to the next step. If the regulator has a 1/4-in. compression fitting, attach the reducer fitting to the outlet of the regulator shut-off valve.

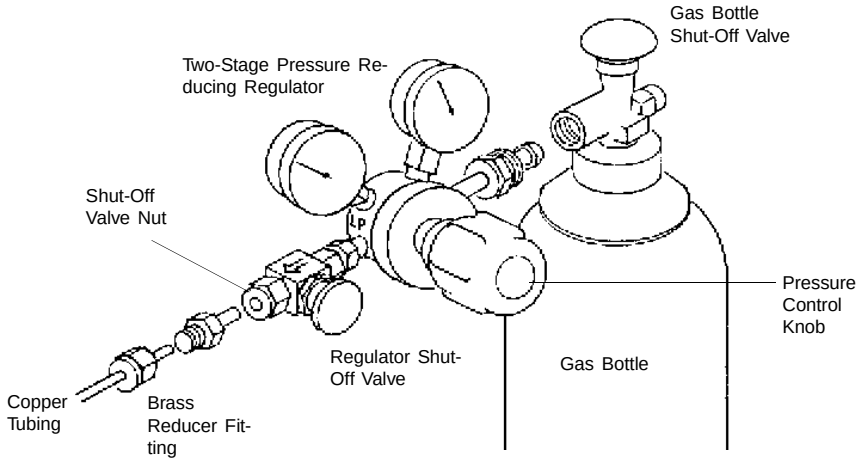


Figure 2-2. Typical Gas Pressure Regulator Assembly

3. Attach the copper tubing to the regulator or reducer fitting.

Do not over-tighten the fittings. Doing so could collapse the brass fitting or copper tubing and cause a leak.

4. Test the regulator for leaking as follows:

   a. Close the regulator shut-off valve by turning it clockwise.

   b. Turn the pressure regulator control knob fully counterclockwise.

   c. Open the gas bottle valve by turning it counter-clockwise; then close the gas bottle valve.

   d. Observe the high pressure gauge. If the pressure decreases, tighten the nut connecting the regulator to the gas bottle. If the pressure is stable, proceed to next step.

e. Turn the pressure regulator control knob clockwise until the outlet pressure gauge indicates 45-50 psi (310- 345). Open the regulator shut-off valve by briefly turning it counterclockwise, then close the valve.

f. Make sure the gas bottle valve is completely closed.

5. Attach the other end of the copper tubing to the gas port on the back of the analyzer.

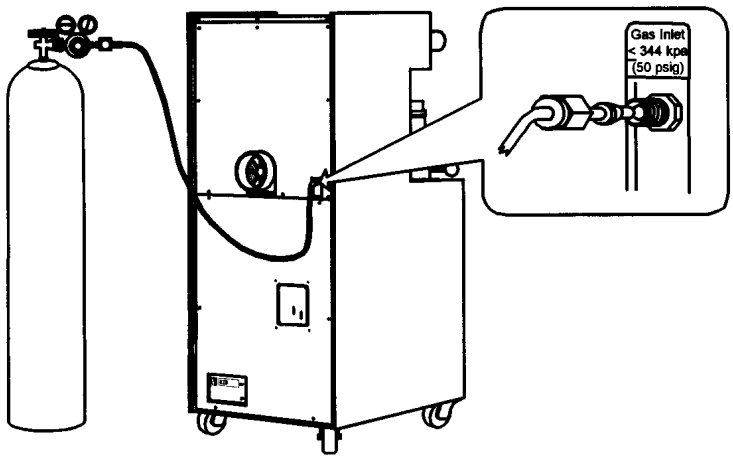


Figure 2-3. Gas Inlet Port

6. Open the regulator shut-off valve and the gas bottle valve.

Attaching the Computer

Connecting the Computer to the Analyzer

1. Connect one end of the communication cable to the connector labeled **COM 1** (serial port) on the computer; tighten the retaining screws.

The communication cable has 9-pin connectors on both ends. If your computer does not have a 9-pin connector, use an adapter to make the connection.

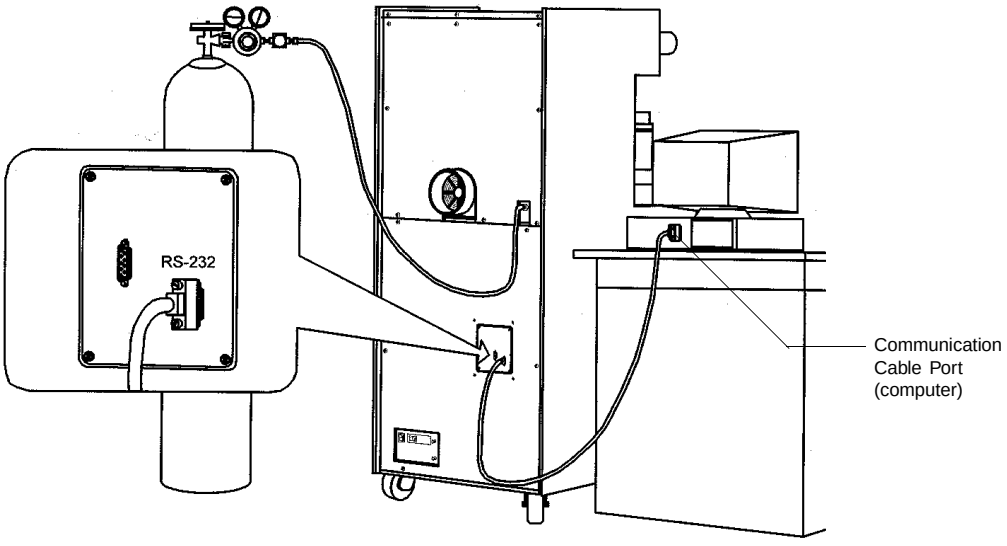


Figure 2-4. Connecting the Cables to the AutoPore IV

2. Connect the other end of the communication cable to the connector labeled **RS232** on the analyzer; tighten the retaining screws.

Connecting the Computer to the Monitor, Keyboard, and Printer

Follow manufacturer's instructions shipped with each device. The following are guidelines. Figure 2-5 shows typical connections.

1. Plug the keyboard cable into the keyboard input connector on the rear panel of the computer.

2. Plug the video monitor cable into the monitor connector on the rear panel of the compuer.

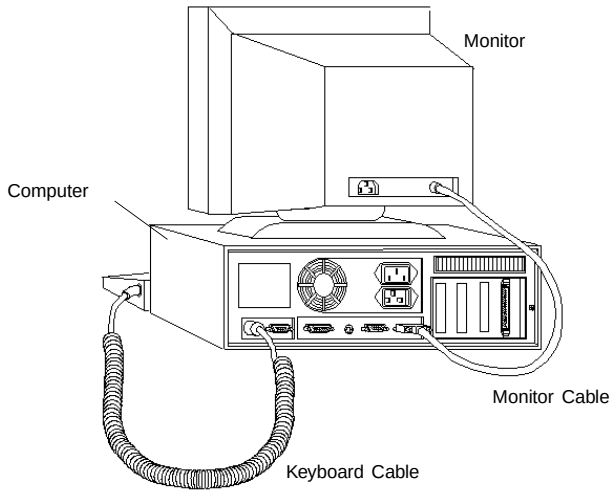


Figure 2-5. Keyboard and Video Monitor Connections

3. Plug the mouse cable into the mouse connector on the rear panel of the computer (not shown).

4. Plug the monitor and computer power cords into an appropriate power source.

5. Connect one end of the printer cable into the input connector at the rear of the printer. Connect the other end of the printer cable to the output connector labeled **LPT1 (PRINTER)** at the rear of the computer. The printer output connector at the rear of the computer may be labeled **PARALLEL** instead of **LPT1**.

Connecting the Exhaust Duct (optional)

If the location of the instrument is not well ventilated, you should vent the exhaust port to a ventilation system or to the outdoors. Connect the AutoPore exhaust port to your forced-air venting system as follows:

1. Connect one end of the 5-in. (13-cm) flexible duct (similar to the type used for home clothes dryers) to the exhaust port on the rear panel of the AutoPore; secure with a hose clamp.

2. Connect the other end of the flexible duct to the venting system and secure with a hose clamp.

Since the AutoPore does not force air, your venting system must pull air from the AutoPore. The air should be pulled at a rate of approximately 50 liters/sec (100 cfm).

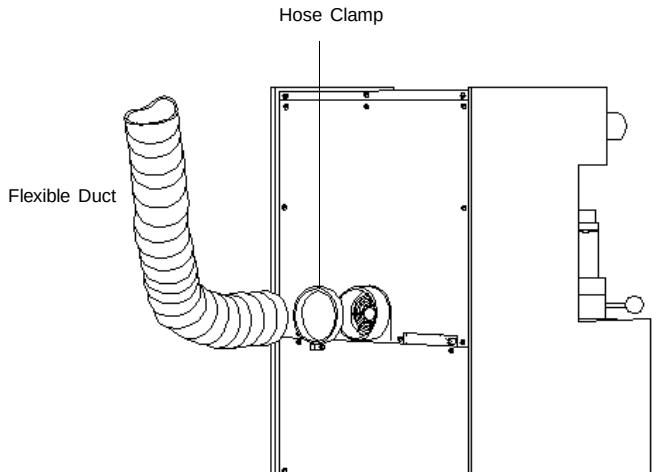


Figure 2-6. Venting the AutoPore IV

Installing the Capacitance Detectors

Capacitance detector cables should be unplugged using the 4-pin connectors at the ends of the cable. The connectors are designed to retract as you pull them from the receptacle. Pulling on the cable itself does not cause the connector to retract, so it cannot be pulled free of the receptacle without damaging the cable.

There are six capacitance detectors for the 9520 and 9505 (two are internal on the high pressure vessels), and three for the 9510 and 9500 (one is internal). Each low-pressure external capacitance detector has a label inside, identifying the po crt to which it should be connected.

Installing Cables on Capacitance Detectors

Attach a cable to each capacitance detector by inserting the cable's 4-pin connector into the receptacle on the back of the detector.

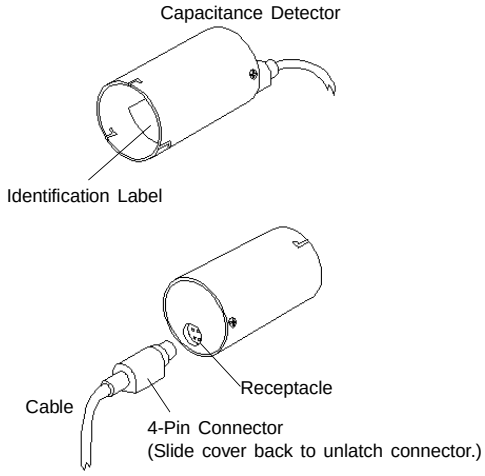


Figure 2-7. Capacitance Detector Cable Installation

Installing Capacitance Detectors on the Low Pressure Ports

1. Install each low pressure capacitance detector on the appropriate low pressure port. The correct port is printed on the identification label inside each detector.

Each capacitance detector is calibrated to its specific port; therefore, it is important to install each detector on the correct port.

2. Plug the free end of each cable into the corresponding outlet.

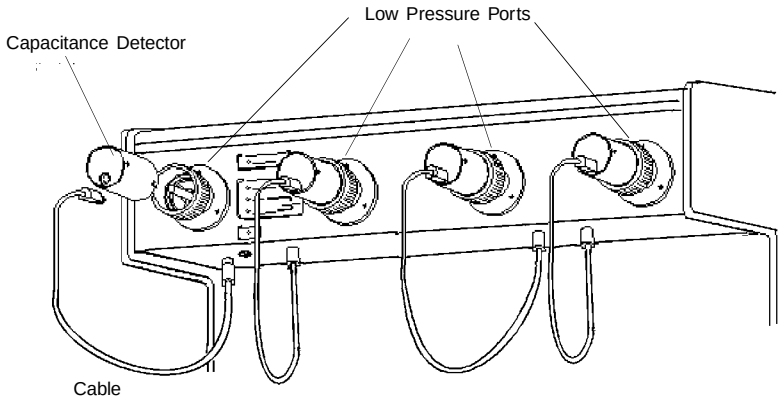


Figure 2-8. Installing Low Pressure Capacitance Detectors

Installing Capacitance Detectors on the High Pressure Ports

1. Open the analyzer cabinet's front panel. Install each high pressure capacitance detector on the appropriate high pressure port. (The correct port is printed on the identification label inside each detector.) The high pressure detectors fit on the underside of each port.

2. Plug the free end of each cable into the corresponding receptacle. Both receptacles are located on a panel on the upper right side of the high pressure compartment. Be sure to plug the left and right port cables into the corresponding receptacles. The receptacles are labeled for left and right ports.

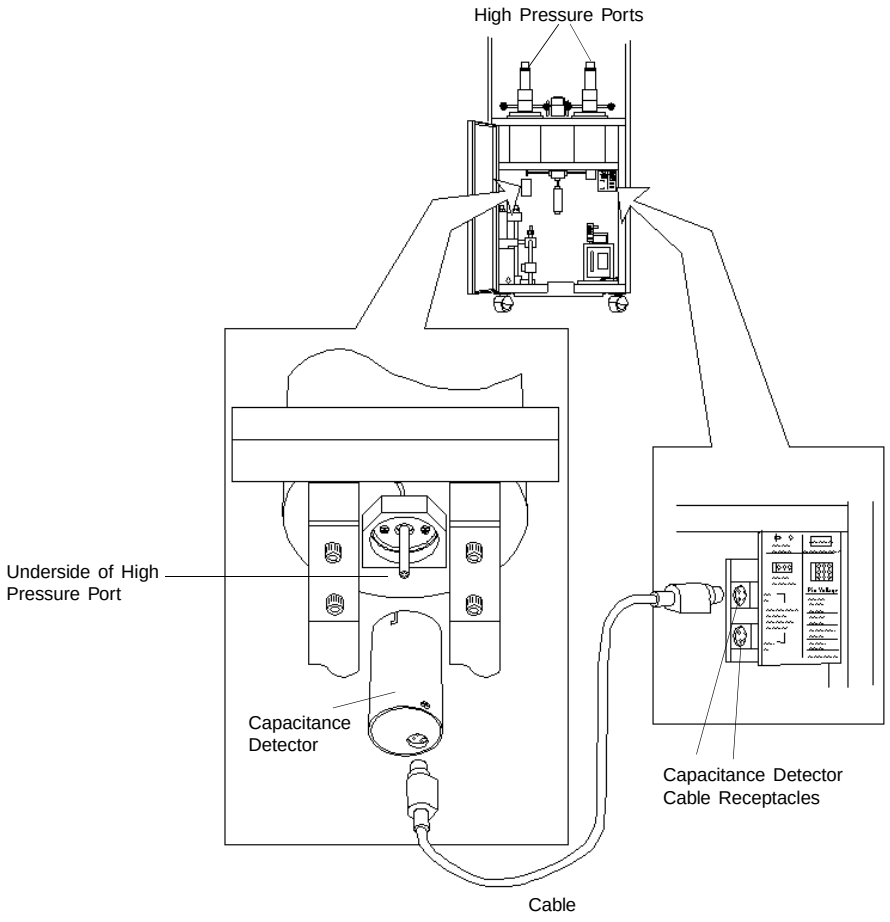


Figure 2-9. Installing a High Pressure Capacitance Detector

Filling the Mercury Reservoir

The AutoPore requires approximately 5 lbs of mercury (minimum) to begin analyses; do not use more than 10 lbs. The mercury level should be approximately 0.5 to 1.0 in. (1 to 3 cm) from the top of the mercury viewing window; it should never be above the window. Fill the mercury reservoir following the instructions provided in *Maintaining Mercury Level* in Chapter 8.

A spilled-mercury container located in the center of the AutoPore work surface 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.

Turning the System On and Off

Turning On the System

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

1. Place the analyzer's power switch in the ON position. The Power indicator on the upper front panel is illuminated.
2. Turn on the video monitor, printer, and computer.
3. Double-click on the AutoPore icon or select the AutoPore progran from the Windows Start menu to start the AutoPore program.

Turning Off the System

Always make sure you exit the program and/or Windows before turning off the computer. Failure to do so could result in loss of data.

1. Select **Exit** from the **File** menu. If you exit the AutoPore program with analyses in progress, the analyses continue and data are collected. Reports that are queued under the Print Manager will print.
2. Place the computer, monitor, and printer ON/OFF switches in the OFF position.
3. Place the analyzer Main Power switch in the OFF position.

Installing the Analysis Program

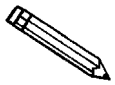
Your system must meet or exceed the following minimum requirements before you can install the AutoPore software:

- Pentium 333 MHz computer (or equivalent) with 64 megabytes of main memory and 200 megabytes of free disk space
- Windows NT (4.0 or newer)
- CD ROM drive
- 3 1/2-in. disk drive
- SVGA monitor (800 x 600 minimum resolution)

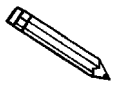
You can also install the AutoPore analysis program on a computer other than the one controlling the analyzer, allowing you to:

- create or edit sample and parameter files
- generate reports on completed sample files

Review the Micromeritics PROGRAM License Agreement for restrictions on the use of another copy of the analysis program.



Power management features should be disabled so that Micromeritics applications can communicate properly with the instrument during operation. These features can be disabled in the computer setup configuration or through a utility supplied by your computer manufacturer.



It is recommended that you close all programs before beginning installation of the AutoPore software.

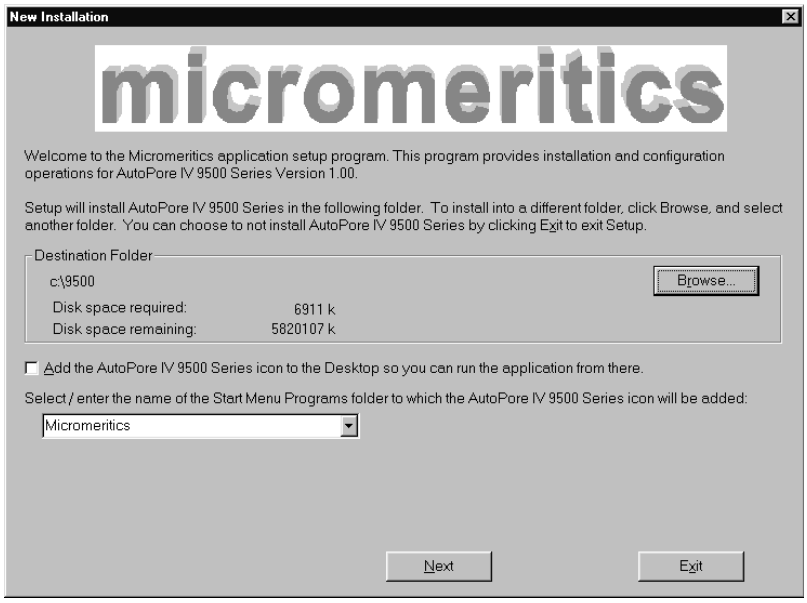
Initial Installation

The AutoPore IV analysis program is supplied on a CD. Install the program as follows:

1. Turn on the AutoPore analyzer.
2. Insert the AutoPore CD into your CDROM drive.
3. Select **Start** from the Status bar.
4. Select **Run** from the Start menu.
5. Enter the drive designator for the CDROM, followed by **setup**; for example:

   e:setup

6. Click OK ; the New Installation dialog is displayed.

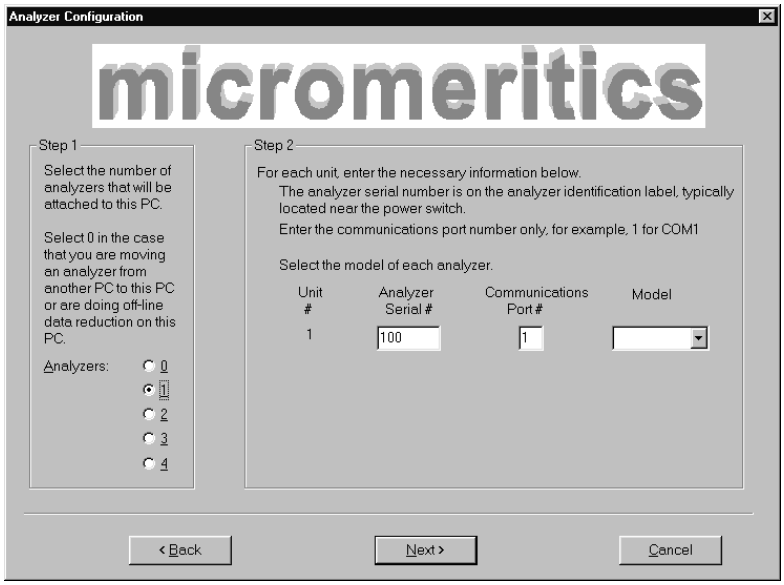


7. The Destination Folder portion of the dialog displays the amount of current disk space, the amount of disk space required for the analysis program, and the directory into which the application will be installed. If you want to install the application into another directory, click Browse to select a directory.

8. If you want to add the AutoPore IV icon to your desktop so you can run the application from there, click the checkbox.

9. Enter or select from the drop-down list the folder to which the AutoPore IV icon will be added.

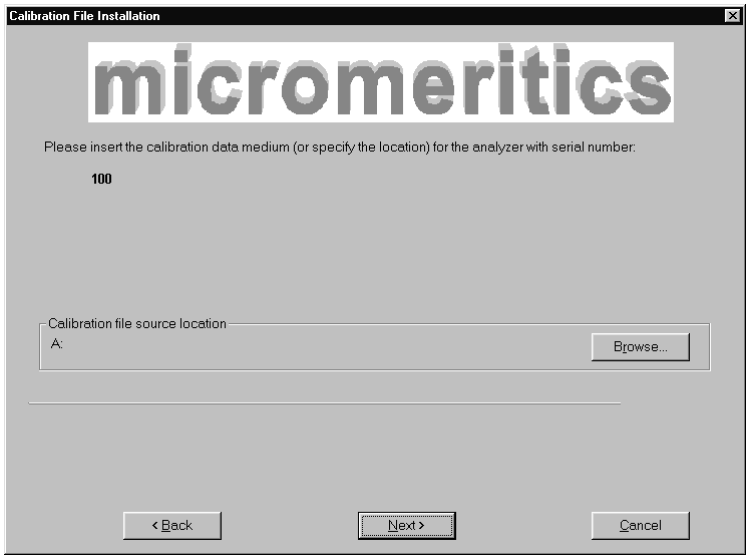
10. Click Next ; the Analyzer Configuration dialog displays.



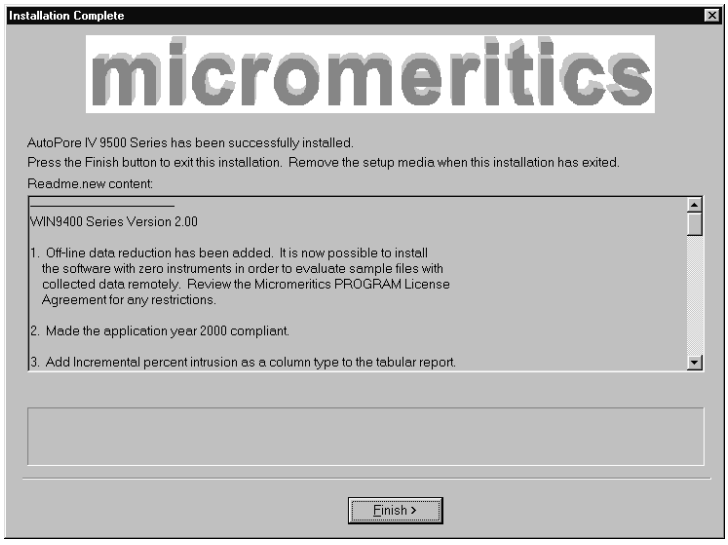
11. Click the radio button for the number of analyzers to be attached to this computer.

12. For each analyzer, enter the analyzer's serial number and communications port number, then select its model number from the Model drop-down list.

13. Click Next ; the Calibration File Installation dialog is displayed.



14. Select the location of the calibration source files. If the calibration files are located in a directory other than the one displayed, click Browse to select the directory. Click Next ; the application installs the calibration files and displays the Installation Complete dialog.



15. The Readme file is displayed. Use the scroll bar if you want to read the contents of the file. Click Finish to complete the installation.

16. Remove the program CD and store in a safe place. The original program CD contains the calibraiton files specific to your instrument. Upgrade CDs do not contain calibration files. Therefore, it is important that you store the original program CD in a secure location in the event calibraiton files need to be reinstalled.

Subsequent Installation

After initial installation, you can use the application setup program to:

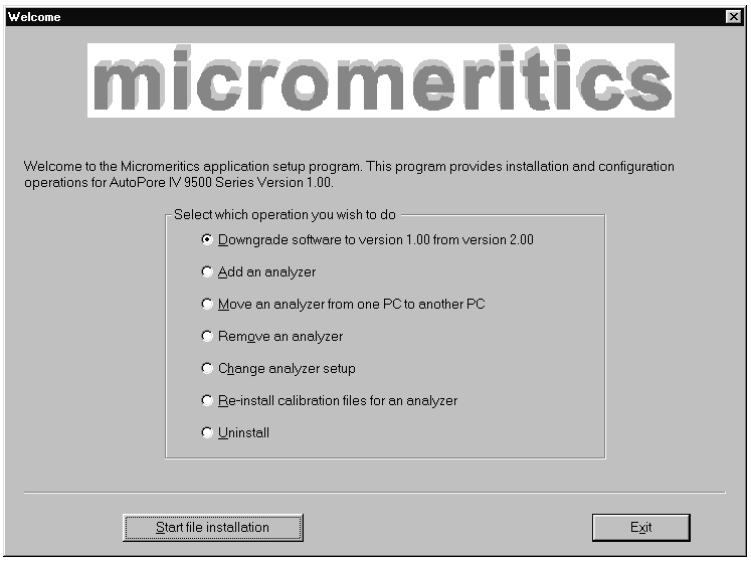
- Upgrade software
- Add an analyzer
- Move an analyzer from one computer to another
- Remove an analyzer
- Change an analyzer's setup
- Reinstall calibration files for an analyzer
- Uninstall the analysis program

To run the application setup program:

1. Turn on the AutoPore analyzer.
2. Insert the AutoPore CD into your CDROM drive.
3. Select **Start** from the Status bar.
4. Select **Run** from the Start menu.
5. Enter the drive designator for the CDROM, followed by **setup**; for example:

   e:setup

6. Click OK; the Setup Welcome screen is displayed.



7. Click the radio button for the operation you wish to perform. Instructions for each operation are included in the following sections.
8. Click Exit to close the Welcome screen.

Installing a Software Upgrade

When you install a software upgrade, the system installs all the application files and any status files that do not already exist on the computer. Existing analyzer status files are not affected and default and data files are not overwritten. There are three cases for this operation:

- The software version controlled by the setup program is a later version than the version installed on the computer.
- The software version controlled by the setup program is the same version as the version installed on the computer.
- The software version controlled by the setup program is an earlier version than the version installed on the computer.

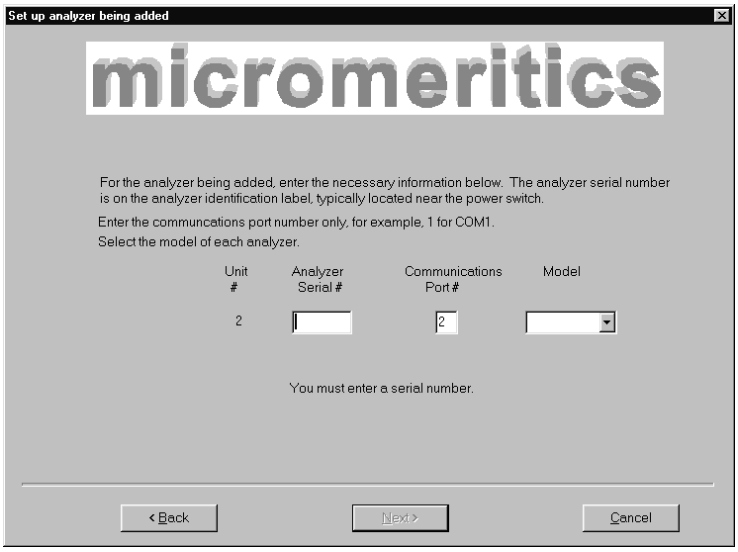
The setup program detects which type of Upgrade applies and customizes the selections in the Setup dialog accordingly.

1. In the Setup dialog, select:
   - Upgrade software to version [number] from version [number], or
   - Reinstall software version [number], or
   - Downgrade software to version [number] from version [number]
2. Click **Start file installation**. The application installs the software and displays the Installation Complete dialog.
3. Click **Finish** to complete the installation.

Adding an Analyzer

You can add an analyzer to an existing application as follows:

1. In the Setup dialog, select **Add an analyzer**, then click Next. The Setup dialog displays.



2. Enter the analyzer serial number and model number and the communications port number.

3. Click Next; the Calibration File installation dialog is displayed. Select the location of the calibration source files. If the calibration files are located in a directory other than the one displayed, click Browse to select the directory.

4. Click Next; the application installs the calibration files and displays the Installation Complete dialog.

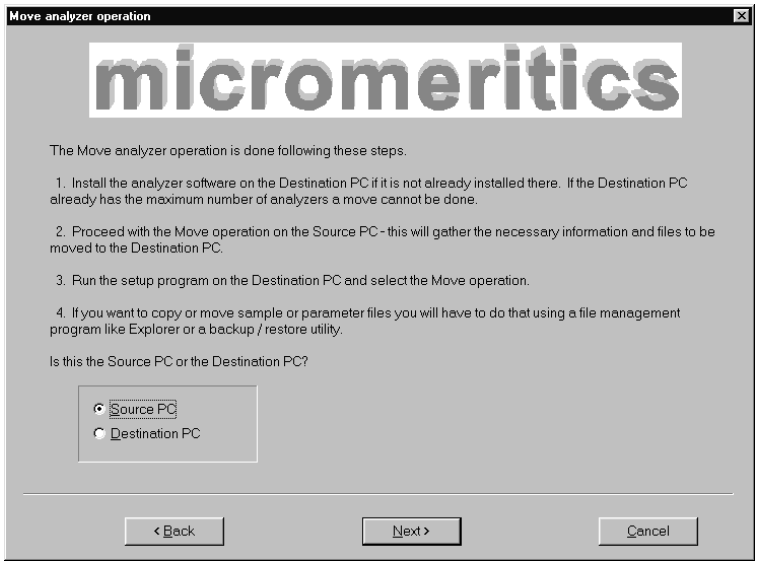
5. Click Next. The application adds the new analyzer and displays the setup Welcome screen.

Moving an Analyzer from One Computer to Another Computer

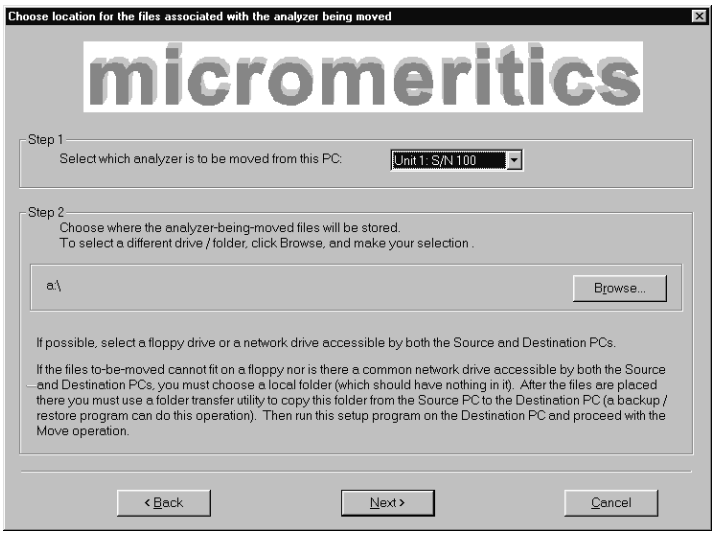
You can move an analyzer, along with its status and calibration files, from one computer (Source PC) to another computer (Destination PC) as follows.

This operation does not move sample or parameter files. To move these files, use a file management program such as Explorer or a backup/restore utility.

1. Install the AutoPore IV software on the destination computer. Refer to *Initial Installation* earlier in this chapter.
2. Run the application setup program on the source computer. Refer to *Subsequent Installation* earlier in this chapter.
3. In the Setup dialog, select **Move an analyzer from one PC to another PC**, then click Next. The Move analyzer operation dialog box displays.



4. Select **Source PC,** then click Next ; the following dialog is displayed.



5. Select the analyzer that is to be moved from this computer from the drop-down list.

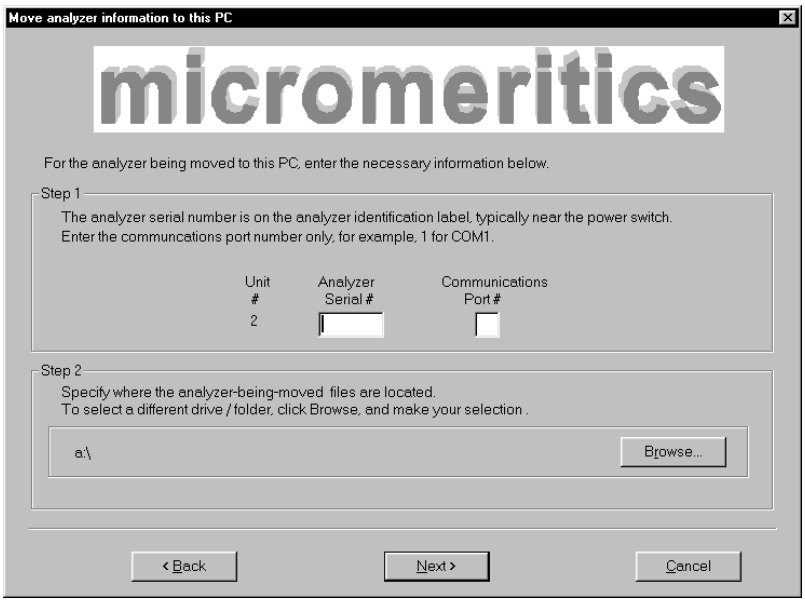
6. Select the location in which the files to be moved will be stored. If possible, the location should be a floppy drive or a shared network drive. If this is not possible, select a local folder. After the files are placed there, use a folder transfer utility to copy this folder from the Source PC to the Destination PC.

7. Click Next ; the files are moved and the setup Welcome screen is displayed.

8. Run the application setup program on the destination computer. Refer to *Subsequent Installation* earlier in this chapter.

9. In the Setup dialog, select **Move an analyzer from one PC to another PC**. The Move analyzer operation dialog box displays (shown on previous page).

10. Select **Destination PC,** then click Next. The following dialog is displayed.



11. Enter the serial number and model number of the analyzer that is being moved and the communications port.

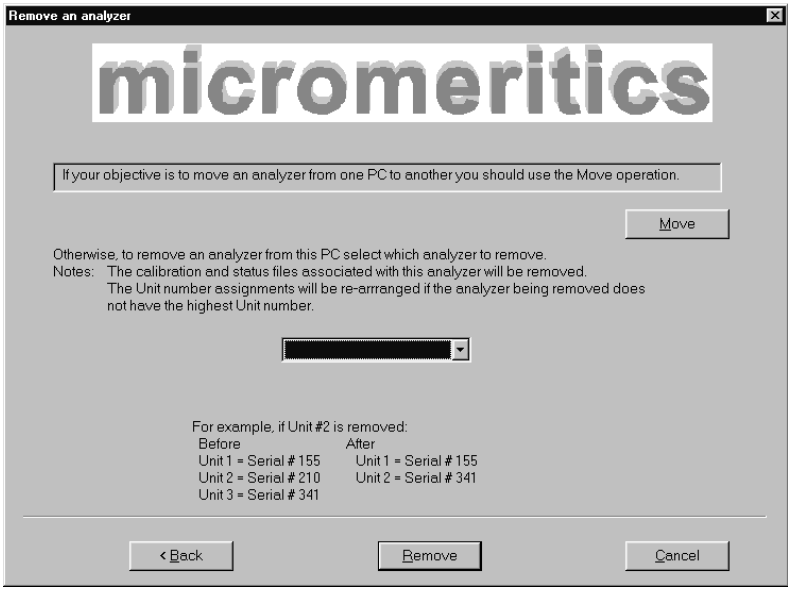
12. Enter the location of the files that were stored previously (step 6 above).

13. Click Next; the files are moved and the setup Welcome screen is displayed.

Removing an Analyzer

You can remove an analyzer from the system as follows. When you do so, the system removes the calibration and status files from the computer.

1. In the Setup dialog, select **Remove an analyzer**, then click Next. The Remove an analyzer dialog displays.



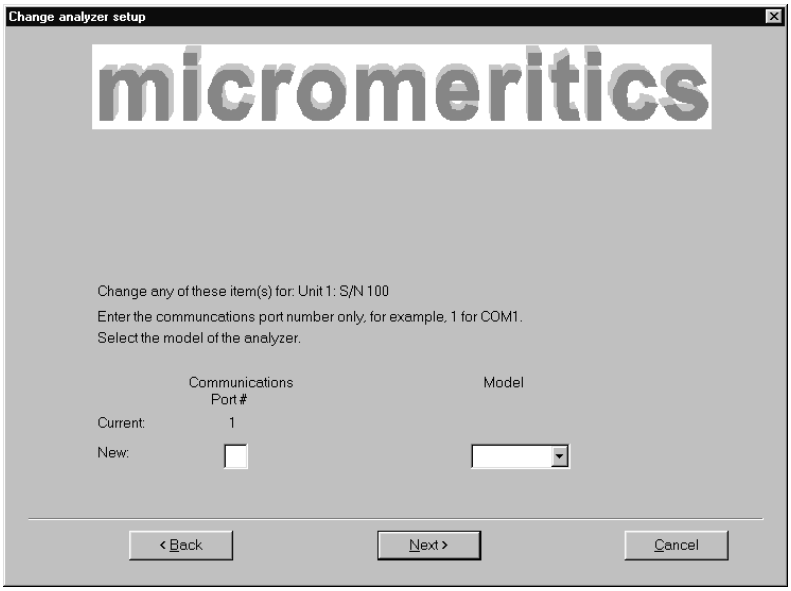
2. Select the serial number of the analyzer you want to remove.

3. Click Remove. The application removes the analyzer and returns to the setup Welcome screen.

Changing an Analyzer Setup

You can change the setup of an analyzer as follows:

1. In the Setup dialog, select **Change analyzer setup**; the Change analyzer setup dialog displays.



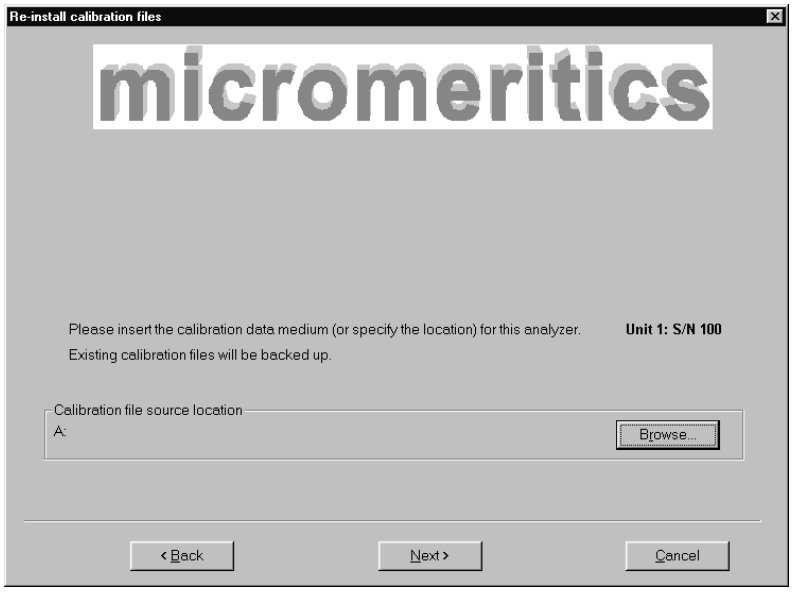
2. Select the analyzer's communications port and model.

3. Click Next. The application changes the analyzer's setup and returns to the setup Welcome screen.

Reinstalling the Calibration Files

You can reinstall the files containing an analyzer's factory calibration data as follows:

1. In the Setup dialog, select **Re-install calibration files for an analyzer**, then click Next. The Re-install calibration files dialog displays.



2. Select the analyzer whose calibration files you want to reinstall from the drop-down list.

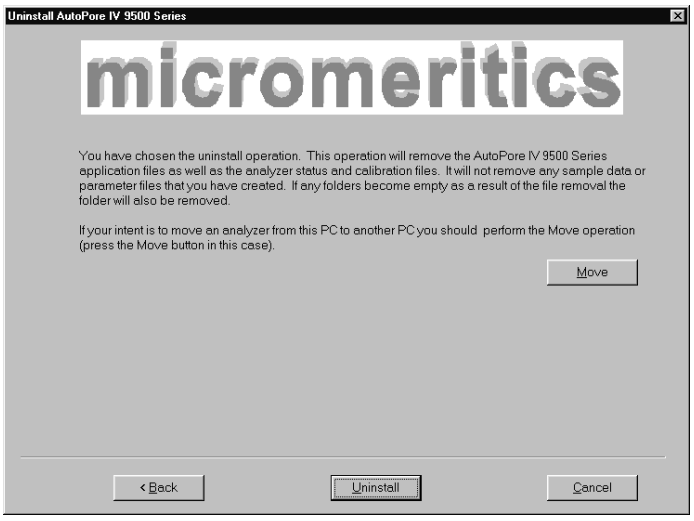
3. Insert the disk containing the calibration files or select the location of the calibration files if they are stored in another location.

4. Select Next. The application reinstalls the calibration files and returns to the setup Welcome screen.

Uninstalling the Analysis Program

You can remove the AutoPore IV analysis program as follows. When you perform this operation, the application removes the application, status files, analyzer setup files, and resulting empty directories. It does not remove data files.

1. In the Setup dialog, select **Uninstall**, then click Next. The Uninstall dialog displays.



2. Click Uninstall. The Select Uninstall Method dialog displays.

3. Choose one of the following:

   - Select **Automatic**, then click Next to have the system uninstall the 9500 software automatically.

   - Select Custom, then click Next to choose which modifications are to be made during the uninstall. The system displays a series of dialog boxes that allow you to select the files and directories to be removed.

Interruption of the Program or Loss of Power

If you exit the AutoPore program with analyses in progress, the analyses continue and data are collected by the instruments. Reports which were already queued under Print Manager will print. However, reports for new data collected after exiting the program will not print because the program cannot pass the new data to the Print Manager.

If power is interrupted to the computer only, analyses continue, and data are collected, but not permanently saved on the computer disk. When power is restored to the computer, any data collected after power was interrupted are permanently saved on the computer disk, and the instrument can be operated as usual. As with virtually all computer applications, changes you have made to open files (but have not yet saved) are lost when power is interrupted.

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

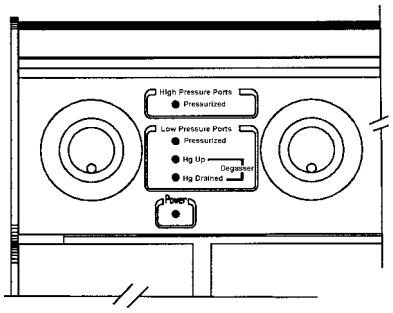
CHAPTER 3

GENERAL OPERATING INSTRUCTIONS

- Front Panel Description
- Rear Panel Description
- Using the Software
- Menu Structure
- Selecting (Opening) Files
- Displaying the Unit Configuration
- Displaying the Instrument Log
- Changing System Options Settings

GENERAL OPERATING INSTRUCTIONS

Front Panel Description



High Pressure Ports Pressurized — Illuminates when the high pressure system is pressurized, when the computer system is not connected, or when the computer is connected but the software is not running.

Low Pressure Ports Pressurized — Illuminates when the low pressure system is pressurized, when the computer system is not connected, or when the computer is connected but the software is not running.

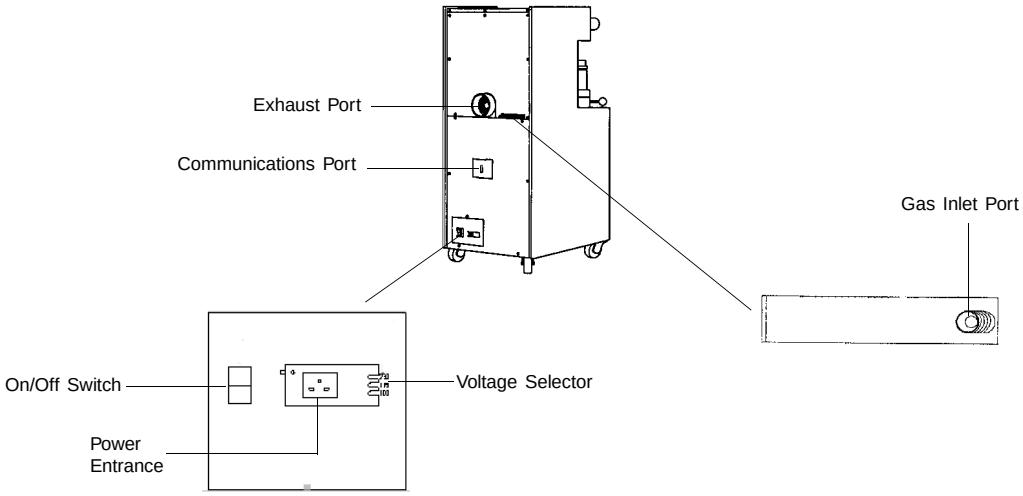
Hg Up — Illuminates when the amount of mercury in the low pressure system is sufficient to fill the penetrometers.

Hg Drained — Illuminates when mercury has been drained from the low pressure system.

Never remove a penetrometer or blank plug from the low pressure port when the Hg Drained indicator is not illuminated. Doing so could allow mercury to spill from the low pressure port.

Power — Illuminates when power is supplied to the instrument.

Rear Panel Description



Exhaust Port — Provides ventilation for the analyzer. This exhaust can be vented to the outside using flexible tubing similar to the type used on a home clothes dryer, and a vent system which pulls air from the exhaust port.

Communication Port — Used for connecting the computer to the analyzer.

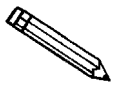
Gas Inlet Port — Used for connecting an external gas supply to the analyzer when generating pressures in the low pressure system.

On/Off Switch — Turns the analyzer on and off. It also provides electrical overload protection.

Voltage Selector — Used to set the analyzer for the correct incoming AC line voltage.

Power Entrance — Used for connecting the analyzer to the electrical supply.

Using the Software



The AutoPore program operates in the Windows environment. You should be familiar with standard Windows operations, such as using the mouse, menus, and dialog boxes, before attempting to use the AutoPore software. This manual provides no instructions for such standard operations. Consult your Windows User's Guide for more information.

This section describes the specific applications of certain software conventions to AutoPore IV.

Keyboard

Shortcut (mnemonic) keys can be used instead of, and in addition to, a mouse. Instead of opening a menu and clicking on an item, just press the appropriate shorcut keys. The following table describes the shortcut keys used to perform AutoPore commands. To access a function using the keys, press Alt plus the underlined letter. Refer to your Windows manual for more information about using keys.

Table 3-1. Shortcut Keys

Key(s)	Function
F2	Open a sample information file
F3	Open an analysis conditions file
F4	Open a penetrometer properties file
F5	Open a report options file
F6	Tile windows
F7	Cascade windows
F8	Start report
F9	Cancel report
Alt + F4	Exit the AutoPore analysis program
Ctrl + F6	Shift between windows
Ctrl + S	Turn on/off grid display for graphs
Ctrl + T	Indicate that you are using threaded penetrometer closures
Ctrl + R	Present pore size data in radius measurements
Ctrl + D	Present pore size data in diameter measurements
Ctrl + N	Present pore size data in nanometers
Ctrl + M	Present pore size units in micrometers
Ctrl + A	Present pore size units in Angstroms
Ctrl + I	Present pressure data in psia
Ctrl + P	Present pressure data in Megapascals
Shift + F2	List sample information files
Shift + F3	List analysis conditions files
Shift + F4	List penetrometer files
Shift + F5	List report options files

Browse Button

Some screens include a Browse... button. Browse... opens a window that lets you choose from the files available for the current field. You can select a choice from the list by double-clicking on it.

Next and Previous Keys

On some screens, you can click on Next>> and <<Prev to move from one file or part of a file to another.

Replace Button

The Replace button allows you to copy file values from an existing file into the one you are creating. You can then edit the values in the new file without changing the original file. To use this button:

- Click Replace.
- Select a file from the drop-down list. Choose from the sample files supplied with your system or from files you have created.
- Click OK, and values in the file you are copying appear in the new file you are creating.
- Changes affect the new file only, not the original file.

Red Field Markers

If you enter unacceptable data in a field, the field is shaded red. You cannot save a screen that contains a red-shaded field. If you move to another window without correcting the field, the word “Error” may appear in red letters on the window containing the red-shaded field.

To correct data in a field that is shaded red, click the cursor in the shaded field, then read any instructions that appear at the bottom of the window. You may also wish to consult the instructions for the field or window in the operator’s manual.

Menu Structure

Main Menu Bar

All of the functions in the system are accessed from the Main Menu, which contains the following choices:

Menu Item	Description
File	Allows you to manage sample and parameter files.
Unit [n]	Enables you to perform analyses and other instrument operations.
Reports	Enables you to generate and close reports.
Options	Allows you to make a variety of choices about your system's defaults and configuration.
Windows	Enables you to select and manage the application's windows.
Help	Displays online help for the AutoPore program and information about the AutoPore program.

File Menu

The File menu lets you manage files containing sample and parameter information. The functions on this menu are discussed in Chapter 4.

Open — Allows you to select an exising file to open for editing, or to create a new one.

Save — Saves changes to an open file.

Save As — Saves the open file under a different name or as a different type.

Save All — Saves all open files.

Close — Closes the file in the active window.

Close All — Closes all open files.

Print — Prints the contents of sample or parameter files. Report destination is selected from the print dialog window, which opens when you select a print option.

List — Generates a directory listing of sample or parameter files.

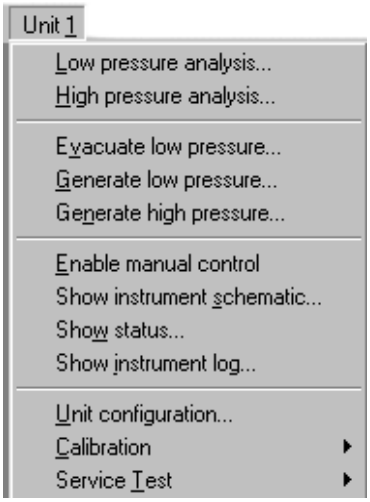
Export Exports the contents of a sample or parameter file (ASCII format).

Average Allows you to create a sample file containing the averages of up to four sample files.

Convert Converts a nonWindows sample file for the 9220, 9320, or 9420 to a sample file that is compatible with Windows (refer to Appendix J).

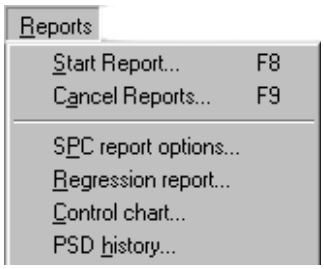
Exit Exits the application.

Unit Menu



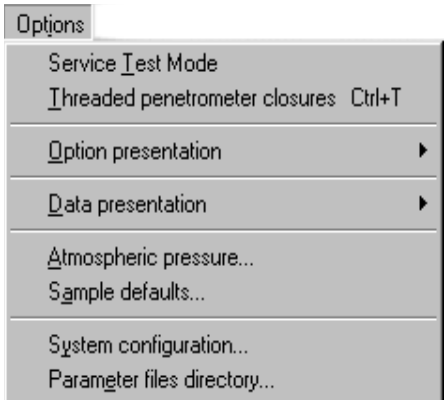
The Unit menu allows you to start a low-pressure or high-pressure analysis; manually control the instrument; display the system schematic, status, log, and configuration; and calibrate the instrument. The functions on this menu are discussed in Chapters 5 and 6.

Reports Menu



The Reports menu allows you to select, modify, and start reports, or to cancel a report in progress. The functions on this menu are discussed in Chapter 7.

Options Menu

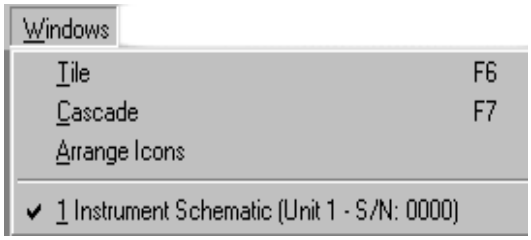


The Options Menu allows you to make a variety of choices about your system's defaults and configuration.

The data presentation submenu of the options menu allows you to customize your data.

These options are discussed later in this chapter.

Windows Menu



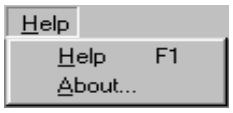
Tile — Resizes all open windows and arranges them side by side so that the contents of all open windows are visible.

Cascade — Resizes all open windows and arranges them in a stacked fashion. The active window is positioned on top of the stack. Each window's title remains visible, making it easy to select other windows.

Arrange Icons — Arranges all minimized icons in an orderly manner.

The windows menu also displays all open files; the active window is preceded with a check mark.

Help Menu

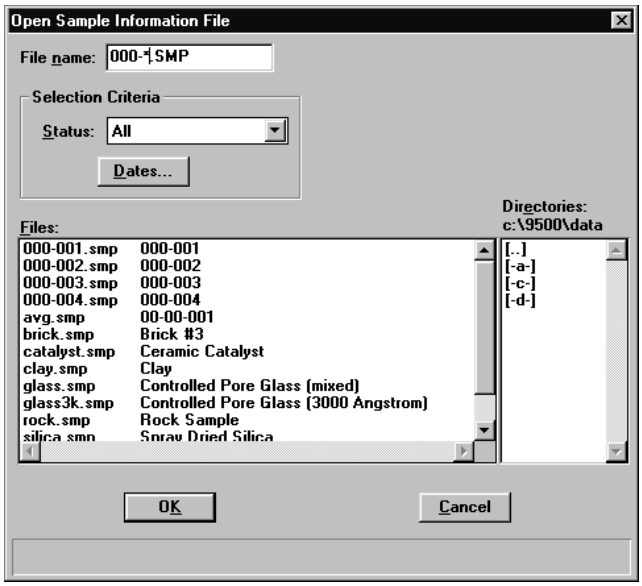


Help — Lists help topics for the AutoPore analysis program.

About — Displays information about the AutoPore IV program.

Selecting (Opening) Files

It is important to be familiar with opening new and existing files using the Open File windows. An Open File window appears when you select an operation that requires you to open a file. Many AutoPore operations require you to select a file before beginning the operation. The window below is an example:



In some instances, a default file name appears in the File Name box. You may accept the default by pressing Enter, or you may change the file name.

- To create a new file, type a new file name.
- To open an existing file, select the file's name from the list of available files in the Files box.

You may change the current directory by choosing another directory from the Directories list box or by typing a new directory into the File name field.

Controlling/Limiting the Available Files

You can limit the list of files displayed in the Files box in three ways.

- Use wildcard characters in the path name entered in the File name field.

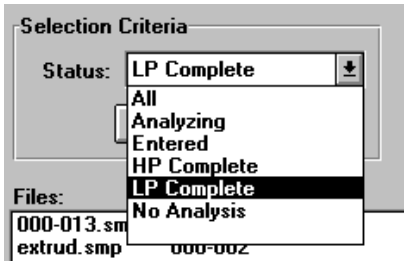
  Standard wildcard characters (such as * and ?) can be used to filter file names. For example, in the dialog box shown above, the list of files is limited to files containing 000- as the first four characters of the file name.

- Enter a range of dates. To enter a range of dates, click on Dates... . The Select Dates window appears:

  Enter the beginning and ending date. The format of the date is set using the International Date Format function on the Windows control panel. The range of dates remains the default until you change the dates or select Show All Dates. When entering dates, you may use the following function keys. (These keys' usual functions are replaced with the following when the Select Dates window is open.)

  F2 Clears the field.
  F3 Inserts the current date.
  F4 Displays a calendar from which you may select a date.

- Limit the available file choices by status.



  From the drop-down list, select the status to which you wish to limit the file choices. The following table describes file types.

Table 3-2. Sample Information File Status and Description

Status	Description	Operations That May be Initiated on This Sample
All	All sample information files in the specified directory and within the specified range of dates.	As per following descriptions.
Analyzing	An analysis is currently being performed using this sample file.	Generation of partial reports
Entered	This sample file contains manually entered data.	Report generation
LP Complete	A low pressure analysis has been performed using this sample file. (Only one low pressure analysis may be performed on each file.)	High pressure analysis Generation of low pressure reports
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.	Report generation High pressure analysis
No Analysis	No analysis has been performed using this sample file.	Low pressure analysis

File Name Conventions

For sample information files, a default file name (which contains the next available sequence number) and a default extension appear. For analysis conditions, penetrometer properties, and report options, a wildcard character and a default extension appear.

The default file name extensions appear below.

File Type	Extension
Sample information	SMP
Analysis conditions	ANC
Penetrometer properties	PEN
Report options	RPO
Export to disk (ASCII)	RPT
Convert nonWindows sample files to Windows-compatible files	DAT
Report to disk	RPT
List to disk	RPT

File Names *vs.* File Identifiers

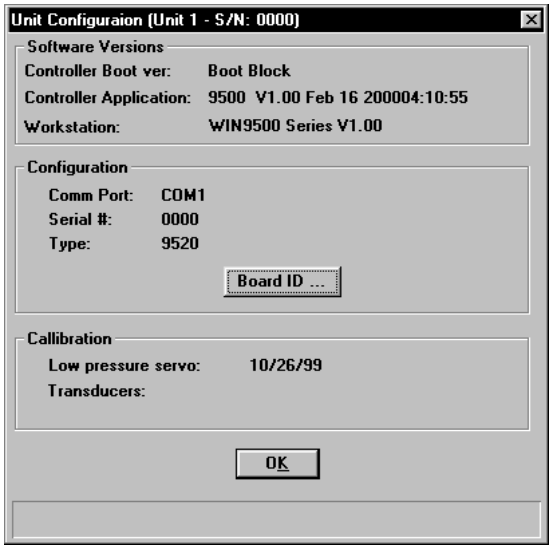
When you create a sample file, or a part of a sample file, such as an analysis conditions file, penetrometer properties file, or report options file, you are asked to name the file in the File Open window.

When the data entry window for the file opens (for example, the Basic Sample Information window for creating a sample file), a long field is available for you to type a name that makes it easy for you to identify the file. Sometimes, the file name defaults into the file identifier field. You may accept the file name or type an identifier that gives a more intuitive description of the file's contents.

The default identifier for a sample file can be controlled from the Sample defaults option under the Options menu.

Displaying the Unit Configuration

You can display the software/hardware configurations for the AutoPore analyzer as described below. From the Main Menu, select **Unit**, **Unit configuration** to display the Unit Configuration window.



Software Versions — Displays the software versions being used by the instrument.

Configuration — Displays the instrument's communications port, serial number, and type.

Board ID... — Click to display the Board ID dialog box, which displays information about the board selected in the Board drop-down list.

Calibration — Displays the dates of the last high and low pressure servo calibrations, as well as transducer calibrations.

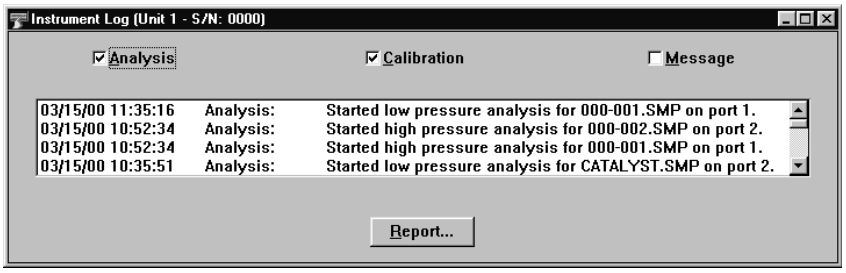
Displaying the Instrument Log

The instrument log contains the following information about the analyses and calibrations performed on the selected instrument:

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

. To display the log, follow these procedures.

1. From the Main Menu, select **Unit, Show instrument log**. The Instrument Log dialog is displayed.



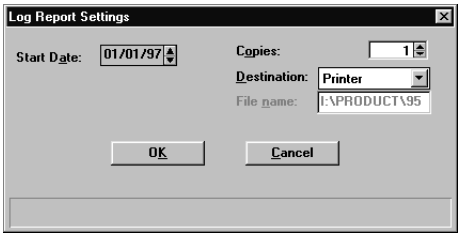
2. The check boxes enable you to select the type of information to display.

   - Click **Analysis** to display the date and time analyses were started on the instrument.

   - Click **Calibration** to display the date and time calibrations were started on the instrument.

   - Click **Message** to display the date, time, and content of messages generated during operation.

3. If you want to save to a file or print the contents of the log, select **Report**. The Log Report Settings dialog box is displayed.



4. Select the start date for the report.

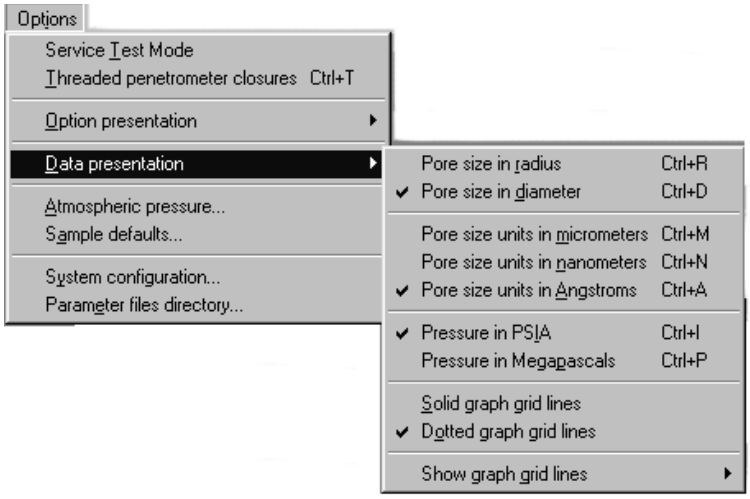
5. Select the destination from the drop-down list. The choices are: File, Printer, Printer/Plotter, and Screen.

   - If you choose File, enter the file name.
   - If you choose, Printer or Printer/Plotter, select the number of copies you wish to print.

6 Click OK to start the report.

Changing System Options Settings

System options settings are found on the Options drop-down menu (on the Main Menu). Instructions for editing most of these settings are included in this section. For those options described elsewhere in this manual, the chapter and/or section is provided.



Service Test Mode

Various service tests are included in the AutoPore operating program. These tests can be performed only with the assistance of a trained Micromeritics service representative. When you select **Service Test Mode** from the Options menu, a dialog prompting you to enter a password is displayed. You will not be able to perform these tests without his guidance. After Service Test Mode has been activated, the tests can be accessed from the Unit menu.

Threaded penetrometer closures

The system cannot generate pressures above 30 psia (0.2068 MPa) unless you are using a penetrometer with threaded closures. Select this option if you are using threaded penetrometer closures.

Option presentation

Allows you to choose the format for sample editing dialogs.

Choices: Advanced, Basic, or Restricted

Advanced displays all parts of the sample information file in a tabbed dialog similar to that of an index card file. Clicking on the tab opens an associated dialog. This format is used to customize files.

Basic displays all parts of the sample file as a single dialog. Sample files are created by selecting predefined parameter files. This format also allows you to switch to the Advanced format if editing is desired.

Restricted displays in the same manner as the Basic format. Certain menu options, however, become disabled and you cannot switch to the Advanced format.

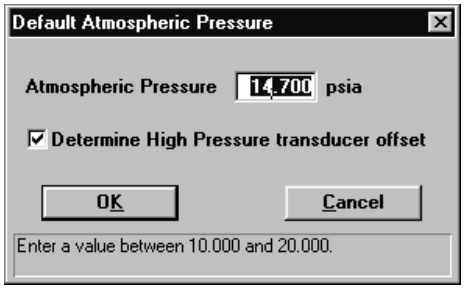
Data presentation

The data presentation pull-down menu lets you specify units of measure for pore size and pressure and make choices about other display options.

Select the options you wish to use:

- Pore size in radius or diameter
- Pore size units in micrometers, nanometers, or Angstroms
- Pressure in psia or Megapascals
- Solid graph grid lines or dotted graph grid lines
- Show graph grid lines enables you to choose from the following:
  None, Vertical, Horizontal, Both

Atmospheric pressure: Displays the Default Atmospheric Pressure dialog.



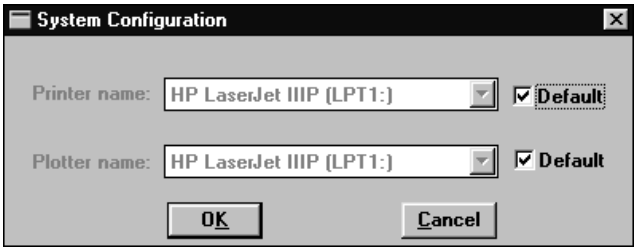
For accurate analysis, the system must adjust pressure measurements to account for variations in local atmospheric pressure (barometric pressure).

Enter the current atmospheric pressure or accept the system default of 14.7 psia (0.1014 MPa). We recommend entering the value on the low pressure display when one port is open. The system uses this value until you change it. We recommend entering 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.

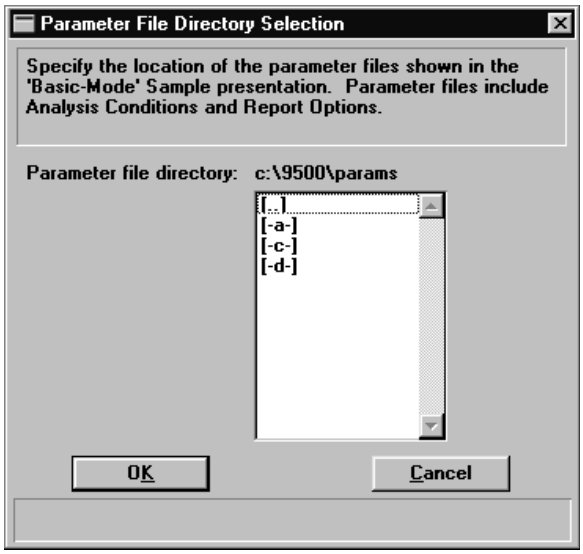
Sample defaults Opens the windows necessary to specify sample defaults (refer to *Editing Sample Defaults* in Chapter 4.).

System configuration Displays the System Configuration dialog, enabling you to specify the printer(s) and/or plotter(s) to which reports will be sent.



Parameter files directory

Displays the Parameter File Directory Selection dialog, enabling you to specify the directory whose files will appear in drop-down lists on the Basic Sample Information window.



This directory selection is independent of the directory selectors on the File Open windows. The File Open windows' directory selectors allow an advanced user to open parameter files from any directory. On the Basic Sample Information window, however, it is impossible to change the directory. Therefore, the files that appear in the drop-down lists are those contained in the directory specified under this option.

CHAPTER 4

CREATING SAMPLE AND PARAMETER FILES

- The Analysis Process - An Overview
- Baseline Error Correction Methods
- Where Should I Begin?
- Calibrating Penetrometers
- Creating and Using Blank Correction Files
- Editing Sample Defaults
- Creating Sample Files
- Creating Penetrometer Properties Files
- Creating Analysis Conditions Files
- Creating Report Options Files

CREATING SAMPLE AND PARAMETER FILES

The Analysis Process - An Overview

The procedure for collecting data with the AutoPore IV can be broken down to two basic steps:

- Creating sample files
- Performing analysis runs

This chapter discusses sample files and gives instructions for creating them. Instructions for performing analyses are found in Chapter 5.

Customize Your Work Flow

The AutoPore IV allows you to customize your method of operation to fit your work flow. You can either create and save groups of sample files, then run groups of analyses, or you can run an analysis on each sample file immediately after you create it.

The Windows environment allows you to increase throughput by allowing you to continue entering sample files while an analysis is running.

Sample Information

Before you can analyze a sample, you must create a *sample information file* for it. The sample information file consists of five parts:

- Sample information
- Analysis conditions
- Penetrometer properties
- Report options
- Collected or Entered Data

The data portion of the sample file is created automatically by the AutoPore after data are collected if you select **Collected Data** for type of data.

Basic Sample Files - Overview

A basic sample file is created by completing a single Sample Information screen. To complete the screen, you select predefined penetrometer properties files and analysis conditions files from drop-down lists. Reports are selected from a check-list. (Complete instructions for creating a basic sample file are contained in this chapter.)

Selecting parameter files (analysis conditions and penetrometer properties files) from drop-down lists makes it easy to apply the same analysis parameters to many sample files.

After you select the parameter file you wish to use, that file's values replace the default values.

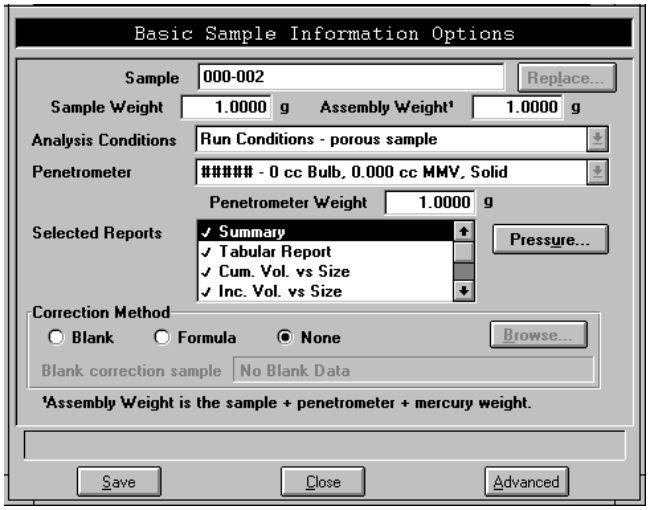


Figure 4-1. The Basic Sample Information Options Screen

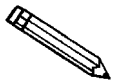
You may move from field to field, entering or changing data until the file is complete. Save the file, and it's ready for analysis.

Using independent parameter files allows one person (for example, a lab manager) to establish parameter files for use by others (such as lab technicians). Duplicate data entry is eliminated, and human error minimized.

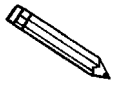
The system includes complete parameter files. You can use the system's parameter files or create your own. After you create a parameter file, it appears on the drop-down list, and can be selected when you create sample files.

For the fastest, easiest operation of your AutoPore IV software, customize the system's defaults to the values you use most. Then, creating a sample file may require only a few entries or adjustments. The system includes a complete set of sample defaults. If the defaults meet your lab's needs, you can immediately begin creating sample files using the instructions in this chapter.

Instructions for editing the sample default file (changing the values to those you expect to use most) and creating your own independent parameter files are also found in this chapter.



If you wish to create your own parameter files, then use them as your default parameter files, create them *before* you edit the defaults. After you create your own files, they appear in the drop-down lists and you can select them as your defaults.



Convenient worksheets to help you gather the information needed to create your samples files can be found in Appendix A.

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.

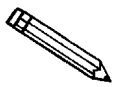
As a result, analysis data may show intrusion where none actually exists. For example, if the sample compresses sharply at a given pressure (compared to the compression of the mercury at that pressure), mercury moves from the penetrometer stem into the sample bulb to fill the space vacated by the shrinking 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. You indicate your choice of correction method in the correction box located on the Basic Sample Information window or the penetrometer properties portion of the advanced format sample information file.

Formula Correction

Through large numbers of blank runs using a variety of pressure tables and penetrometers filled with mercury, Micromeritics has developed a formula for correction of each penetrometer type. The AutoPore software provides this formula, so you simply click on the Formula button in the correction selection box.

If you plan to use formula correction, you should perform some trial blank runs to ensure that the formula provides an acceptable degree of correction for your sample type(s) and analysis conditions.



To achieve the most accurate formula correction, you must enter accurate values for Sample weight, Assembly weight, and Penetrometer weight. You must also provide the calibrated volume of the penetrometer. Either an Assembly weight or a Penetrometer volume of 1.0 disables the mercury compression form of formula correction.

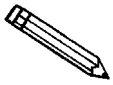
Blank Correction Files

A more accurate and useful correction technique is to use a blank correction file. A blank correction file contains analysis data on a run using one of the following:

- Mercury only (a blank run)
- A nonporous sample of the same weight and material as the samples you plan to analyze

Ideal data would show no intrusion or extrusion; any intrusion that does appear is attributed to baseline error and can be subtracted from subsequent sample runs, thus removing the baseline error from the sample data.

When you perform a blank run using mercury only, the data collected reveal baseline errors caused by the compressibility and thermal effects of the *mercury and penetrometer parts only*. Using a nonporous sample or a reference sample has the added advantage of compensating for the compressibilty and thermal effects of the *sample material*.



You can use the convenient High Pressure Differential Analysis option to create the blank correction file as you perform the sample analysis. A complete description of the High Pressure Differential Analysis is contained in this chapter.

An in-depth discussion of correction methods is contained in Appendix G.

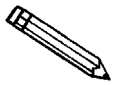
Where Should I Begin?

Use the outline below to decide which section to start with, based on your plans.

SECTION	BEGIN HERE IF
Calibrating Penetrometers	You wish the AutoPore to determine sample density, or if you plan to use correction by formula. » *To review the system's Penetrometer Properties files, select* ***Open***, ***Penetrometer properties*** *from the File menu. The system files are located in the PARAMS directory. Select the appropriate file(s) from the Files list box.*
Creating Blank Correction Files	You wish to use correction by blank file.
Editing Sample Defaults	You plan to use the system's Penetrometer Properties and Analysis Conditions files, but wish to set the sample file defaults to values you expect to use most often. » *To view the system's sample file defaults, select* ***Sample defaults*** *from the Options menu.*
Creating Sample Files	**Basic Format** You plan to use the Penetrometer Properties files, Analysis Conditions files and defaults provided with the system. **Advanced Format** You wish to customize penetrometer properties, analysis conditions, or report options.

SECTION	BEGIN HERE IF
Creating Analysis Conditions Files	You plan to create your own Analysis Conditions files or if you wish to enter your own pressure table(s), especially if you wish to use them as your default files. » *To review the system's Analysis Conditions files, select* ***Open****,* ***Analysis conditions*** *from the File menu. The system files are located in the PARAMS directory. Select the appropriate file(s) from the Files list box. System file names are int_only, bigpores, and refmtrl.*
Creating Penetrometer Properties Files	You plan to change the system penetrometer file names to reflect your penetrometers' serial numbers or if you wish to create your own Penetrometer Properties files, especially if you wish to use them as your default files. » *To review the system's Penetrometer Properties files, select* ***Open****,* ***Penetrometer properties*** *from the File menu. The system files are located in the PARAMS directory. Select the appropriate file(s) from the Files list box.*

Combining Penetrometer Calibration and Blank Correction File Creation



Calibrating your penetrometers and creating blank correction files both require you to perform a low pressure run using an empty penetrometer. You can avoid performing duplicate low pressure runs by combining these processes.

An overview for combining these procedures follows. Complete instructions for each step are found in subsequent sections.

1. Calibrate the penetrometer. To do so, you must create a sample file. Give this sample file the name you wish to use for your blank correction file. Use the system's penetrometer properties file that matches the penetrometer (size and type). Click **Pressure...** to edit the pressure table, if necessary. (You can create your own pressure tables in advance, by creating analysis conditions files that contain your pressure table. Follow the instructions in this chapter for creating analysis conditions files.) Use the analysis conditions and pressure table you plan to use when you analyze samples with this blank correction file.

2. After the low pressure run, do not empty the penetrometer; weigh it and install it in the high pressure port.

3. Create blank correction files by completing the high pressure run.

4. Create a new Penetrometer Properties file for this penetrometer. For the file name, use the six-digit, hyphenated number that is etched on each penetrometer. This number is the last two digits of the part number (which indicates the type of penetrometer), followed by a dash, then the four-digit serial number. When the Penetrometer Properties window opens, click **Replace** . From the File selection list, choose the default file for this type of penetrometer. Change the identifier to include the penetrometer's serial number. Enter the calibrated volume and save the file.

At this point, you have created a complete penetrometer properties file including calibrated penetrometer volume. You have also created a blank correction file for this penetrometer.

Creating Pressure Tables

Pressure tables are created as part of the analysis conditions files. Follow the instructions in this chapter for *Creating Analysis Conditions Files*.

Calibrating Penetrometers

If you wish the AutoPore to determine sample density, or if you plan to use blank correction by formula, the calibrated empty volume of each penetrometer must be entered in the penetrometer properties file. The file can then be selected and used for analyses.

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

Use the Penetrometer Volume Calibration form (a reproducible copy is found in Appendix A) as a worksheet and to maintain a record. This form prompts you through the process of calculating a penetrometer's volume three times, then provides space to record the average of these calculations. When you create the penetrometer properties file, enter the *average* volume.

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

1. Create a sample file. You must create a sample file to run a low pressure analysis, even though no sample is in place.

2. From the File menu, select **Open**, **Sample information**. Type the name you wish to use for this calibration. (If you plan to use blank correction files, see the earlier section on combining procedures.) Click OK, then Yes to create the file.

3. Follow the instructions later in this chapter for creating a sample file. Use zero for sample weight. Use the Penetrometer Properties file that corresponds to the size and type of penetrometer you are calibrating. Click Pressure... to edit the pressure table, if necessary. (If you do not plan to use this low pressure run as part of a blank correction file, set the filling pressure at 1 psia. Use one pressure point at 15 psia.)

4. Perform the low pressure analysis, following the instructions in Chapter 5. Clean the penetrometer (as described in Chapter 5) if you do not plan to perform the high pressure analysis and use these data for a blank correction file.

5. Repeat this process three times, using the worksheet. Average the volumes resulting from the low pressure runs.

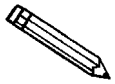
6. Create a penetrometer properties file for this penetrometer, using the instructions later in this chapter. Enter the calibrated volume in the "volume" field.

Creating and Using Blank Correction Files

Creating Blank Correction Files

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

- Mercury only (a blank run)
- A nonporous sample of the same weight and material as the samples you plan to analyze



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

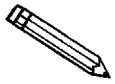
No matter which type of blank correction run you decide to use, the procedure is the same: you follow the instructions for creating a sample file (even if there's no sample at all), then run the analysis. (When you make the blank file's sample file, select *None* for correction method.)

1. Create a sample file. (You must create a sample file to run an analysis, regardless of whether you are using a non-porous sample or no sample at all.)

2. From the File menu, select **Open**, **Sample information**. Type the name you wish to use for this blank correction file. (If you plan to calibrate your penetrometers, see **Combining Penetrometer Calibration and Blank File Creation** earlier in this chapter.) Click OK, then Yes to create the file.

3. Follow the instructions later in this chapter for creating a sample file. Use zero for sample weight.

4. Perform the low pressure and high pressure analyses, following the instructions in Chapter 5.



An alternative method for creating the blank correction file is to perform a High Pressure Differential analysis. This method, described in Chapter 5, analyzes the blank and sample simultaneously.

If you wish, you may perform three blank runs and average their data (using **File**, **Average**) into a final blank correction file for each penetrometer. This is time-consuming, however, and most labs find correction using a single blank correction file to be accurate.

Using Blank Correction Files

When you create sample files for your samples, click the Blank radio button in the Correction box. Click Browse, then select the appropriate correction file from the list. (The Correction box is located on the basic sample information screen. If you are using advanced format, it is located on the Penetrometer Properties screen.)

Editing Sample Defaults

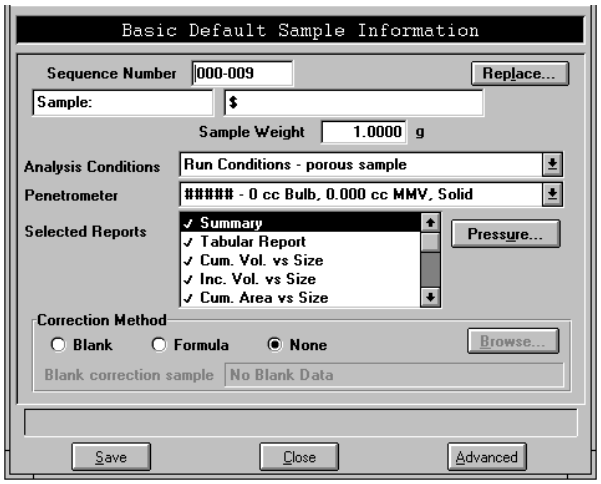
When you open a new sample file, a default value appears in each field. One complete set of default values (the default file) is included with your software. You can use the existing default values, or you can change them to values you expect to use most frequently.

If you plan to use only the default files (you do not plan to change any parameters from sample to sample) you can create sample files on a one-by-one basis as you start the low pressure run. Simply start the low pressure run as directed in Chapter 5; then, type a new sample file name in an available port. You are asked if you wish to create this file. Click OK **and a sample file is created using all default parameters.**

Basic Format

The following section gives complete instructions for changing the basic sample default file to include values you expect to use most frequently.

1. From the Main Menu, select **Options**, **Sample defaults**.



2. The window should be in basic format; if not, click **Basic** .

3. Examine each default value; if necessary, change the values to those you expect to use most frequently. Use the following directions:

   a. **Sequence Number:**

      Specify a default format for the sequence number. This is the sample file's name. The system uses this format to generate an incremented sequence number for each sample file you create.

      - Your sequence number format *must* contain at least three consecutive numbers, such as 1,2,3 or 1,4,8.
      - You may include additional letters, numbers, or some other printable characters, such as dashes.
      - The maximum number of characters is eight.
      - Do not use characters such as * or ?.

   b. **Sample:**

      The Sample field allows you to create a sample file name that provides a better description of the sample than the sequence number alone.

      In the left box, you can edit the *prompt* for Sample. For example, you may prefer "Lot number." The maximum number of characters is 20.

      In the box on the right, you can specify a *format* for the Sample identification.

      - You may use letters, numbers, or other printable characters, such as dashes.
      - If you want to include the automatically generated sequence number as part of the sample identification, enter the $ symbol where you want the sequence number to appear.
      - The maximum number of characters is 42, plus the $.

        For example, if the sequence number is 000-001, enter the sample identification as follows: **Lab #25 - $**

        The resulting sample identification for the first sample information file would be: **Lab #25 - 000-001**

        The sample identification for the second sample information file would be: **Lab #25 - 000-002**

        and so on.

c. **Sample Weight:**

Estimate the weight of the sample. When you create individual sample files, you must enter an exact sample weight for accurate analysis and reporting. However, you may enter an approximate weight for the default.

Range: 0.0000 to 500.0000 g

4. Select sets of penetrometer properties and analysis conditions from the drop-down lists. The files available in the lists are those included with the AutoPore software plus any independent files you have created. Select reports from the checklist.

5. **Correction method:**

Select the appropriate radio button. Choose *Blank, Formula,* or *None.*

If you select *Blank,* the Blank Correction Sample box becomes active.

- Click Browse, then select a blank correction file.
- The file name is then displayed in the Blank Correction Sample box.

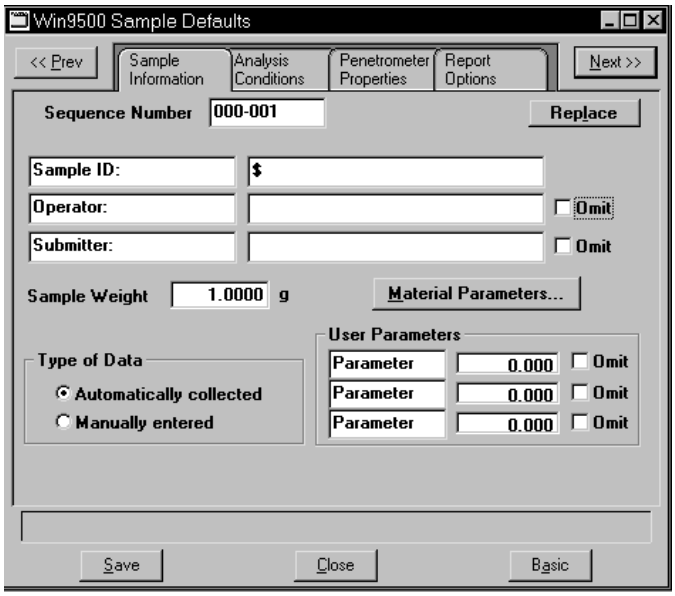
Advanced Format

The advanced sample default file contains basic sample information and sample parameters.

The advanced sample default window looks like a set of index cards. You can move from one set of parameters to another by clicking on the parameter name tabs at the tops of the cards. The types of parameters you can edit are: sample information, analysis conditions, penetrometer properties, and report options.

The following section gives complete instructions for changing the advanced default file to include values you expect to use most frequently. If you expect to use advanced format very rarely, it may not be worth the time to edit the defaults. If you plan to use advanced format frequently, however, you should edit the sample file defaults as presented in the advanced format.

1. Select **Options, Sample defaults** from the Main Menu.



2. If the advanced format is not open, click **Advanced**.

3. Examine the following default values, changing them where necessary.

   a. **Sequence Number:**

   Specify a default format for the sequence number. This is the sample file's name. The system uses this format to generate an incremented sequence number for each sample file you create.

   - Your sequence number format *must* contain at least three consecutive numbers.
   - You may include additional letters, numbers, or some other printable characters, such as dashes.
   - The maximum number of characters is eight.
   - Do not use characters such as * or ?.

   b. **Sample (identifier):**

   The Sample identifier field allows you to create a sample file name that provides a better description of the sample than the sequence number alone.

   In the left field, you can edit the *prompt* for **Sample**. For example, you may prefer "Lot number." The maximum number of characters is 20.

   In the field on the right, you can specify a *format* for the Sample identification.

- You may use letters, numbers, or other printable characters, such as dashes.
- If you want to include the automatically generated sequence number as part of the sample identification, enter the $ symbol where you want the sequence number to appear.

  For example, if the sequence number is 000-001, enter the sample identification as follows: **Lab #25 - $**

  The resulting sample identification for the first sample information file would be: **Lab #25 - 000-001**

  The sample ID for the second sample information file would be: **Lab #25 - 000-002**

  and so on.

- The maximum number of characters is 42.

c. **Operator (identifier):**

The Operator identifier field allows you to identify the person performing this analysis. If you mark the **Omit** box, neither the prompt nor the identifier field will appear on sample files based on this default file.

In the left field, you can edit the *prompt* for **Operator** identifier. For example, you may prefer “Employee number.” The maximum number of characters is 20.

In the field on the right, you can specify a *format* for the Operator identifier.

- You may use letters, numbers, or other printable characters, such as dashes.
- The maximum number of characters is 40.

d. **Submitter (identifier):**

The Submitter identifier field allows you to identify the department or customer associated with this sample. If you mark the “Omit” box, neither the prompt nor the identifier field will appear on sample files based on this default file.

In the left box, you can edit the *prompt* for **Submitter** identifier. For example, you may prefer “Customer.” The maximum number of characters is 20.

In the field on the right, you can specify a *format* for the Submitter identifier.

- You may use letters, numbers, or other printable characters, such as dashes.
- The maximum number of characters is 40.

e. **Sample Weight:**

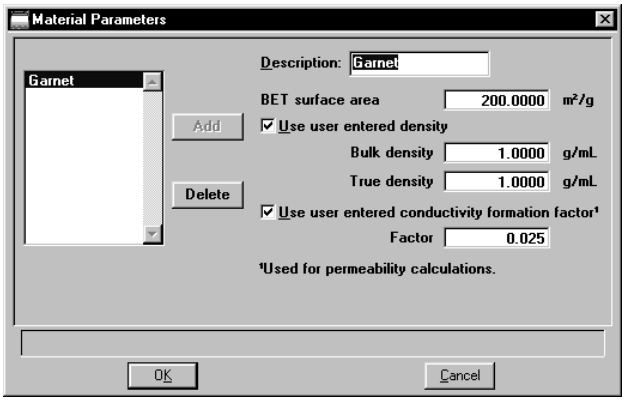
Estimate the weight of the sample. Entry of an exact sample weight is required for accurate analysis and reporting. However, you may enter an approximate weight in this default sample file, then enter the exact sample weight before you begin analysis.

Range: 0.0000 to 500.0000 g

f. **Type of Data:**

Mark a radio button to indicate whether the data will be automatically collected by this instrument during an analysis, or manually entered (perhaps using data collected by another instrument).

g. Click **Material Parameters** if you wish to enter the material information; the Material Parameters dialog displays:



A list of materials is displayed on the left side of the dialog. Select the material whose parameters you wish to edit from the list.

You can use **Add** and **Delete** to add or remove materials form the list.

BET surface area
Range: 0.5000 to 1500.0000 g/mL

Use external density
Check this box to use the entered bulk density and true density. if this box is not checked, the systemwill use values calculated from the intrusion data.

Range: 0.0010 to 50.0000 g/mL (Bulk and True Densities)

Use external conductivity formation factor
Check this box to use the entered conductivity formation factor. If this box is not checked, the systemwill use values calculated from the intrusion data.

Range: 0.001 to 1.000 (Factor)

h. **User Parameters**

User parameters are user-defined sets of data that will be included in SPC reports. You could use a parameter, for example, to specify the amount of time the sample was degassed. You can enter up to three parameters. If you mark the **Omit** box, the parameters will not appear on the SPC reports.

Enter data in each of the Parameter fields you wish to use.

Range: -1000.000 to 1000.000

4. You can edit any of the following analysis parameters by clicking the appropriate tab:

Analysis conditions (for instructions on editing analysis conditions, refer to *Creating Analysis Conditions Files*, beginning with step 5, in this chapter).

Penetrometer Properties (for instructions on editing penetrometer properties, refer to *Creating Penetrometer Properties Files*, beginning with step 5, in this chapter).

Report Options (for instructions on editing report options, refer to *Creating Report Options Files*, beginning with step 5, in this chapter).

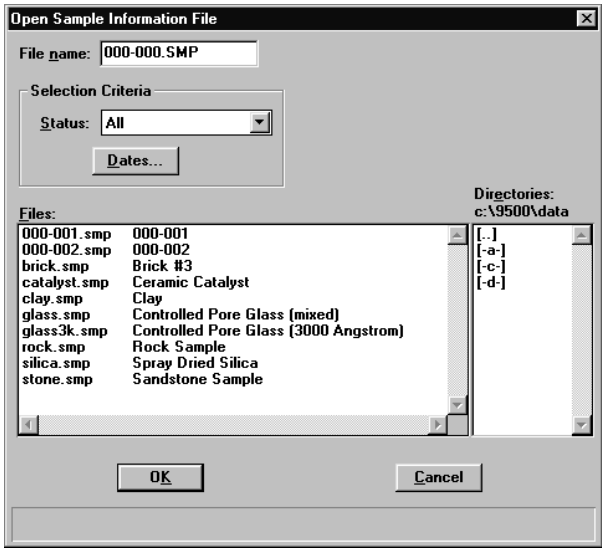
Creating Sample Files

Sample files may be created in three different formats: Basic, Restricted, and Advanced. In addition, you can create sample files in which the collected data contain the averages of the collected data in up to four similar analyses.

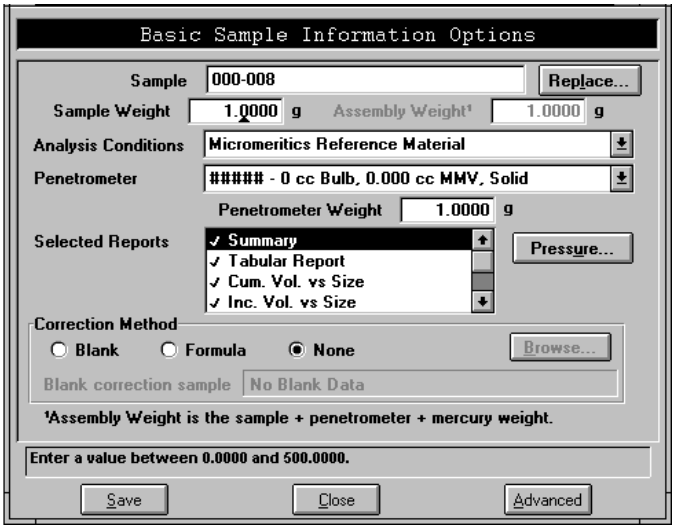
Basic and Restricted Formats

Sample files for the **Basic** and **Restricted** formats are created in the same manner. The only exception is that you cannot switch to the **Advanced** mode when using the **Restricted** format.

1. From the **File** menu, select **Open**, **Sample information**. The Open Sample Information File window is displayed. The system automatically generates the next sequenced file name (based on the format you specified in your sample defaults) and inserts it into the **File name** field. Enter a new file name or accept the default.



2. Click OK. If this is a new file name, click Yes when prompted; the Sample Information window containing the default file values is displayed.



3. Make any necessary changes using the following instructions:

   a. **Sample (identifier):**

      One line of information that further identifies the sample.

      Range: 0 to 40 alphanumeric characters, upper or lower case

   b. **Sample Weight:**

      Sample weight in grams.

      Range: 0.0000 to 500.0000 g

   c. **Analysis Conditions:**

      Select an analysis conditions file from the drop-down list. The list includes the files supplied with your system and any independent files you have created.

   d. **Penetrometer:**

      Select a penetrometer properties file from the drop-down list. The list includes the files supplied with your system and any independent files you have created.

   e. **Penetrometer Weight:**

      Enter the weight of the penetrometer. Instructions for obtaining the weight of the penetrometer (including the weight of the sealing grease) are found in Chapter 5.

f. **Selected Reports:**

Select reports from the list. The list includes the reports supplied with your system.

g. **Correction method:**

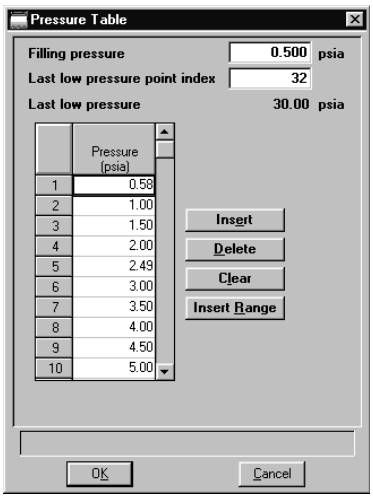
Mark a radio button to choose between:

- Blank correction file - click Browse , then select a specific file
- Formula correction
- No correction

(A complete discussion of correction methods is found in Appendix G.)

h. **Pressure:**

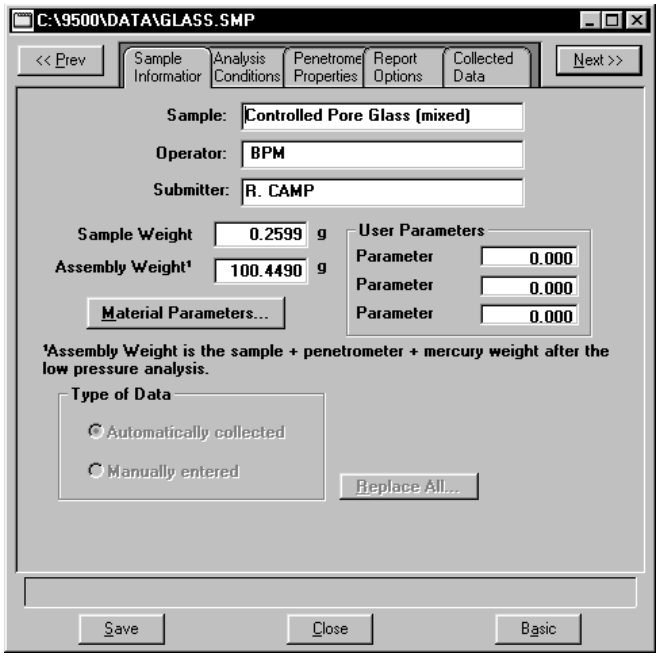
Click Pressure... to edit the pressure table to be used for this analysis.



The pressure table is created as part of the Analysis Conditions file. Complete instructions for editing pressure tables are included in *Creating Analysis Conditions Files* in this chapter.

Advanced Format

Although using the Basic format is the fastest and easiest method, the AutoPore software also allows you to create sample files by completing each part of the sample file individually. The “advanced” format sample windows present the same data contained in the basic format window. However, advanced format gives you complete access to all fields in each part of the sample file. In contrast, basic format offers greater speed and flexibility. Basic format also provides greater control by allowing one user, such as a lab manager, to set up standard defaults and analysis conditions files to be selected from drop-down lists by other users, such as lab technicians.



Use advanced format if:

- You wish to use custom reports.

  You can use the instructions in *Creating Report Options Files* in this chapter. Because basic format sample files do not allow you to select entire report options files (you simply select the reports you wish to include from the checklist on the sample file screen), you must use advanced format if you wish to use custom reports or report options files.

- You wish to have complete, ready access to all parts of the sample file.

  Advanced format sample dialogs are set up like a box of index cards. There is one “card,” or dialog for each part of the sample file: sample information, analysis conditions, penetrometer properties, report options, and data.

- You wish to enter data manually instead of allowing the AutoPore to collect data automatically during analysis.

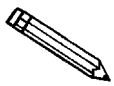
  If you have collected compatible mercury intrusion data from some other source, you may create a sample file and enter the data manually. The AutoPore can then perform data reduction and reporting. You should note that while it is possible to overlay manually entered data on reports of data collected by the instrument, there may be some variation between instruments, which may influence comparisons.

  The Data portion of the sample file is either *manually entered* or *automatically collected*. You specify how data are obtained by marking your choice on the Sample Information dialog. The *manually entered* option appears *only* on the Advanced format sample file screen.

  – If you mark *Automatically Collected*, the data portion of the sample file does not appear until the analysis has been completed (because there is no data until that time). If data are automatically collected, you may view data after they have been collected, but you can not edit them.

  – If you mark *Manually Entered* on the Sample Information dialog, you can enter and edit data in the data portion at any time. However, you may notice that certain fields in the sample file are not available. This is because, when data are to be manually collected, fields that control the automatic analysis process are not needed; only fields that are involved in data reduction are needed.

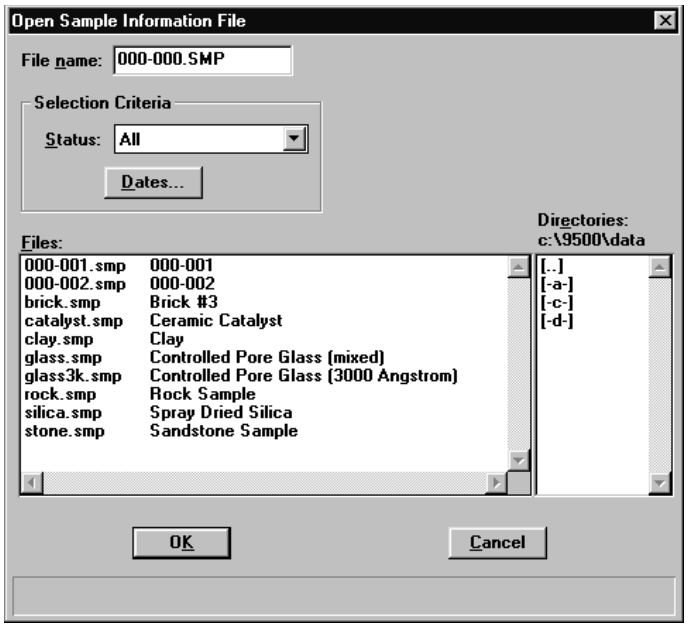
On screen, each advanced format sample file looks like a set of index cards. You move from one part of the file to another by simply clicking on the name tabs. You may continue moving from dialog to dialog, entering or changing data until the sample file is complete.



You can also move from dialog to dialog (within the sample file) by clicking on Next>> and <<Prev.

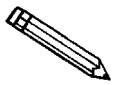
To create an advanced format sample file, follow these procedures.

1. From the File menu, select **Open**, **Sample information**. The Open Sample Information File window is displayed.

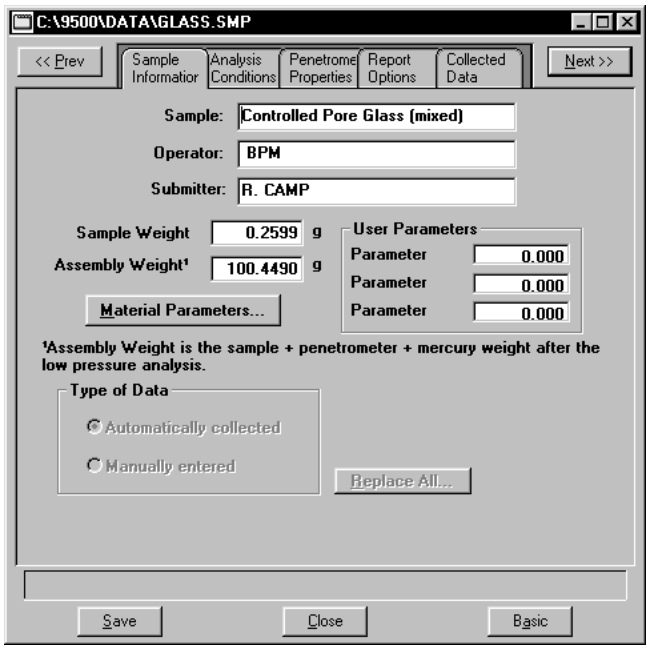


   The system automatically generates the next available file name (sequence number) based on the format you specified in your sample defaults. This file name appears in the **File:** field. Accept the default or enter a new name.

2. Click OK. If this is a new file, a message displays; click Yes. The Sample Information window appears containing default file values. If the window is in basic format, click Advanced to open the advanced format screen.



The Replace all button on the sample information screen replaces the *entire* sample file, including penetrometer properties, analysis conditions, and report options. Separate Replace buttons on the individual parameter screens replace the information on those screens individually.



3. Make any necessary changes to the variables:

   a. **Sample (identifier):**

      One line of information that further identifies the sample.

      Range: 1 to 42 alphanumeric characters, upper or lower case

One or both of the next two fields may not appear on your sample information screen, based on your default sample file.

   b. **Operator (identifier):**

      One line of information to identify the operator.

      Range: 1 to 40 alphanumeric characters, upper or lower case

   c. **Submitter (identifier):**

      One line of information to identify the submitter.

      Range: 1 to 40 alphanumeric characters, upper or lower case

   d. **Sample weight:**

      Sample weight in grams.

      Range: 0.0000 to 500.0000 g

e. **Type of Data:**

Select a radio button to choose between:

- automatically collected data: data is collected by the system during analysis.
- manually entered data: you may enter up to 2500 volume vs. pressure points that you have collected by some other method for data reduction to be performed by the AutoPore.

f. **User Parameters**

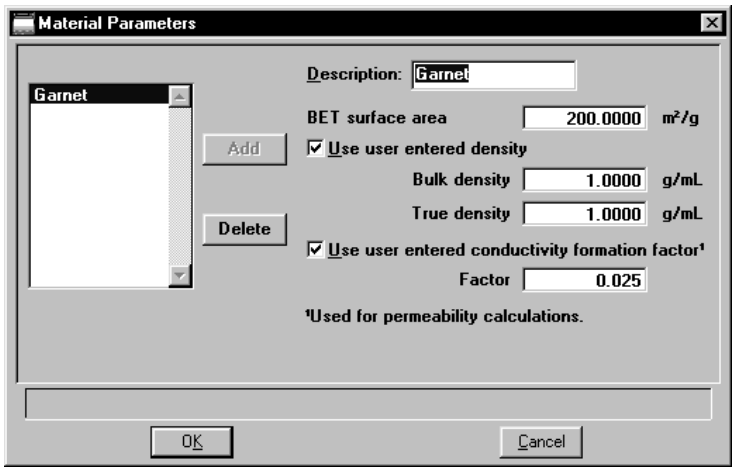
User parameters are user-defined sets of data that will be included in SPC reports. You could use a parameter, for example, to specify the amount of time the sample was degassed. You can enter up to three parameters.

Enter data in each of the Parameter fields you wish to use.

Range: -1000.000 to 1000.000

4. The bulk density, true density, and the conductivity formation factor can be calculated by the standard collected data, but those values are not as accurate as other means. Therefore, if this information is available, you can enter it to increase the accuracy of other data reductions.

Click **Material Parameters** if you wish to enter the material information; the Material Parameters dialog displays.



A list of materials is displayed on the left side of the dialog. Select the material whose parameters you wish to edit from the list.

You can use Add and Delete to add or remove materials from the list. If you change a parameter for a material in the list, Add changes to Change. Click Change to save the changes.

a. **BET surface area**

Range: 0.5000 to 1500.0000 m^2/g

b. **Use external density**

Check this box to use the entered bulk density and true density. If this box is not checked the system will use values calculated from the intrusion data.

Bulk density

Range: 0.0010 to 50.0000 g/mL

True density

Range: 0.0010 to 50.0000 g/mL

c. **Use external conductivity formation factor**

Check this box to use the entered conductivity formation factor. If this box is not checked the system will use values calculated from the intrusion data.

Factor

Range: 0.001 to 1.000

5. You can edit any of the following analysis parameters by clicking the appropriate tab:

Analysis conditions (for instructions on editing analysis conditions, refer to *Creating Analysis Conditions Files*, beginning with step 5, in this chapter).

Penetrometer Properties (for instructions on editing penetrometer properties, refer to *Creating Penetrometer Properties Files*, beginning with step 5, in this chapter).

Report Options (for instructions on editing report options, refer to *Creating Report Options Files*, beginning with step 5, in this chapter).

Switching Between Basic and Advanced Formats

Any time you wish to change formats for a given sample, you can just **c**lick on Basic or Advanced at the bottom of the Sample Information screen.

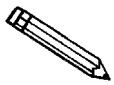
You can also change your default format to Advanced by selecting **Options**, **Data presentation**, **Advanced** from the Main Menu. If you will rarely be working in Advanced format, do not change your default.

Manually Entered or Collected Data

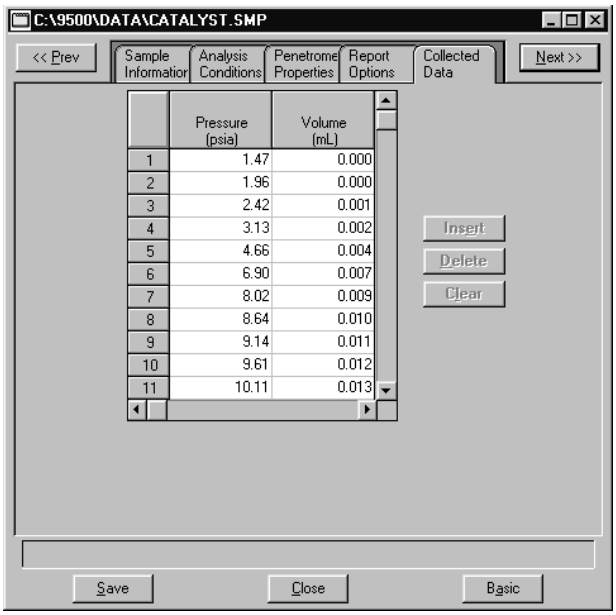
The Entered/Collected Data dialog displays up to 2500 data pairs (intrusion volume and pressure point).

- For advanced format sample files specifying automatically collected data, the Entered/Collected Data dialog can be opened and viewed, but not changed. Data pairs are recorded at the equilibration points at or near the pressure points specified in the sample file's pressure table.

- For those advanced format sample files specifying manually entered data, the Entered/Collected Data dialog allows you to enter up to 2500 pressure points and corresponding volume measurements collected by some other method. Data entered in this dialog will be divided by the weight entered for the sample.



You cannot enter data if the analysis program is being used for offline data manipulation on a computer other than the one controlling the analyzer.



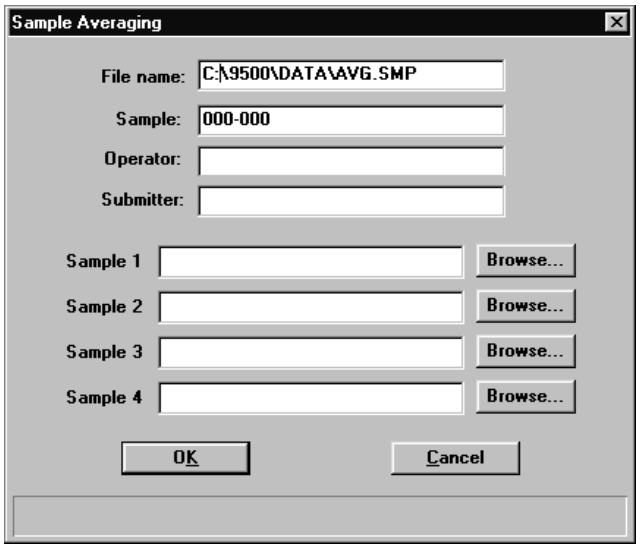
Pressure is displayed in the units you specified by selecting **Options, Data presentation**: either psia or megapascals. The Intrusion Volume column is displayed in mL.

Insert	**C**lick to insert a pressure point before a selected point.
Delete	Click to delete a selected point.
Clear	Click to clear the pressure table.
Ctrl + ↓	Adds data points incremented by the difference between the two previous points.

Sample Averaging

You can create a sample file in which the collected data are the average of up to four similar analyses. All the information in the created sample file will be the same as in the first selected file, except for the information entered in the Sample Averaging dialog. The collected data in the file will be the average of the data in the files selected.

1. Select **File, Average** from the Main Menu to display the Sample Averaging dialog.



2. Enter the following information:

   a. **File name:**

   The system automatically generates the next sequenced file name (based on the format you specified in your sample defaults) and inserts it into the **File name** field. Enter a new file name or accept the default.

b. **Sample (identifier)**:

One line of information that further identifies the sample.

Range: 1 to 42 alphanumeric characters, upper or lower case

c. **Operator (identifier):**

One line of information to identify the operator.

Range: 1 to 40 alphanumeric characters, upper or lower case

d. **Submitter (identifier):**

One line of information to identify the submitter.

Range: 1 to 40 alphanumeric characters, upper or lower case

3. Select up to four files for sample data averaging. You may enter the file names or use Browse to select the files. The system will create the file using an average of the files selected for averaging.

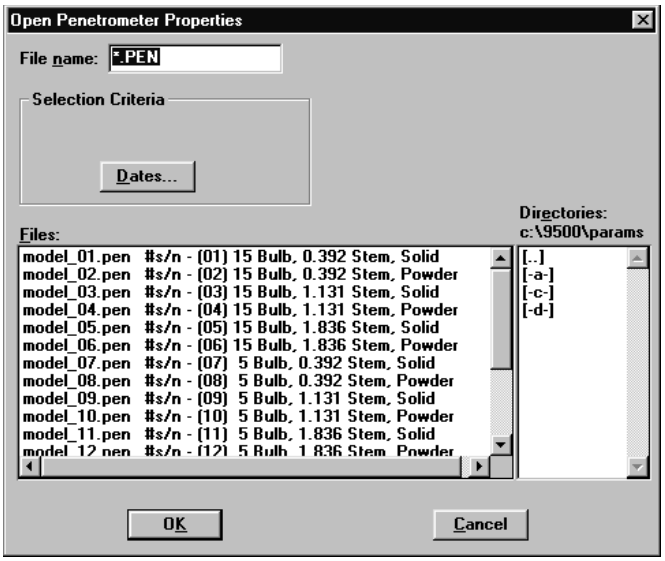
4. Click OK to close the dialog box.

Creating Penetrometer Properties Files

Penetrometer properties files contain the information about the penetrometer that affects the analysis. Penetrometer properties files are selected from the drop-down lists when you complete a basic sample information screen.

Use the following instructions to create a penetrometer properties file for each of your penetrometers. After you complete and save a penetrometer properties file, it will appear in the drop-down list each time you create or edit a sample file.

1. From the File menu, select **Open**, **Penetrometer properties**. The Open Penetrometer Properties dialog is displayed.



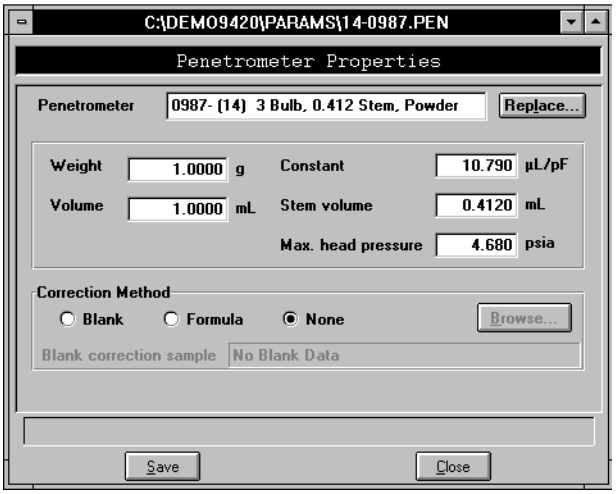
2. Enter a name in the **File name**: field. It is best to use the 7-character identifier etched on the penetrometer bowl. This number consists of the **last two digits of the base part number** and the **4-digit serial number** separated with a dash. For example, if the part number is 950-617**08**-00 and the serial number is **1420**; the etched number (and recommended file name) would be 08-1420.

3. Click OK. If this is a new file, the following message appears:

   File (file name) does not exist. Do you wish to create it?

4. Click Yes .

When you are creating penetrometer properties files, you should almost always use Replace . This button allows you to select the system penetrometer properties file that corresponds to your penetrometer's size and type. A copy of the system file is placed in the new file you are creating. This eliminates the need for you to enter much of the information about the penetrometer. You can then edit the new file, using values specific to this penetrometer.



5. Make necessary changes as follows:

a. **Penetrometer (identifier):**

Enter an identifier for this penetrometer file. We recommend that you use the penetrometer's type number and serial number (etched on the penetrometer) followed by the bulb volume, stem volume and type. If you have used Replace , all you will to do is replace the symbols at the beginning of the identification with the serial number.

Range: 40 alphanumeric characters, upper or lower case

b. **Weight:**

Weight of the empty, assembled penetrometer (not including the spacer). You may wish to simply accept the default, since this field should be completed (updated) when you load the penetrometer before each analysis.

Range: 1.0000 to 1000.0000 g

c. **Volume:**

Enter the volume of the penetrometer (optional). This is required in order to calculate density, or when using the blank correction formula. The instructions for determining the penetrometer's volume (*Calibrating Penetrometers*) are found earlier in this chapter.

Range: 1.0000 to 30.0000 mL

d. **Constant:**

Enter the penetrometer constant provided with your penetrometer. If you have used **Replace** to copy parameters from a previous file, the value defaulted in this field may be incorrect. Be sure to verify the value in this field with the one provided with your penetrometer.

Range: 1.000 to 50.000 µL/pF

e. **Stem volume:**

Enter the penetrometer stem volume from Table 5-1. If you have used **Replace**, the stem volume associated with this penetrometer is defaulted.

Range: 0.0200 to 10.0000 mL

f. **Max. head pressure:**

Enter the maximum head pressure from Table 5-1. If you have used **Replace**, the maximum head pressure associated with this penetrometer is defaulted.

Range: 3.000 to 5.000 psi or 0.021 to 0.034 MPa

g. **Correction method:**

Mark a radio button to choose between:

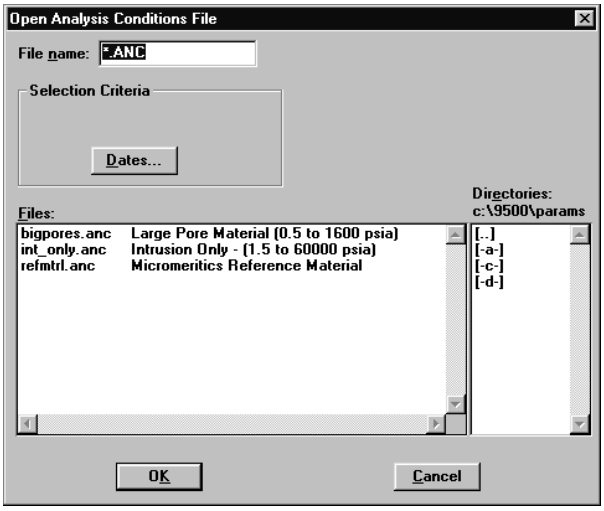
- Blank correction file — click **Browse**, then select a specific file
- Formula correction
- No correction

(Instructions for creating a blank correction file are found earlier in this chapter. A complete discussion of correction methods is found in Appendix G.)

Creating Analysis Conditions Files

Analysis conditions files specify the parameters used to guide an analysis, including the pressure table. Analysis conditions files are selected from the drop-down lists when you complete a basic sample information screen (described at the beginning of this chapter).

1. From the File menu, select **Open**, **Analysis Conditions**. The Open Analysis Conditions File window is displayed.

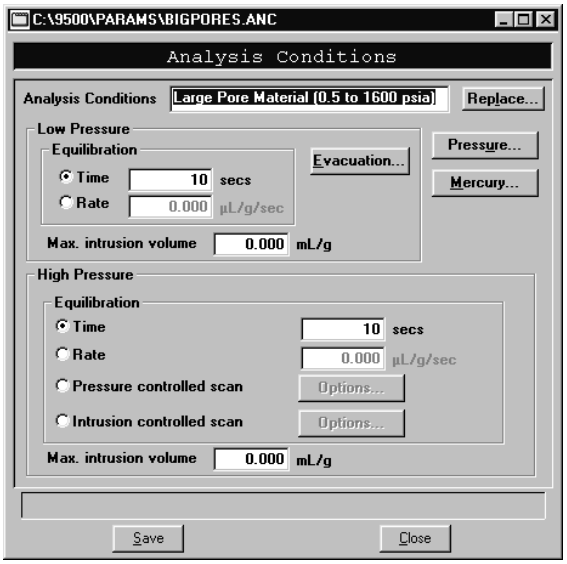


2. Create a name for this file and enter it in the **File name:** field.

3. Click OK. If this is a new file, the following message is displayed:

   File (file name) does not exist. Do you wish to create it?

4. Click Yes; the Analysis Conditions dialog box is displayed.



5. Make any necessary changes to the following variables:

   a. **Analysis conditions (identifier):**

      Enter an identifier for this file. It is recommended that you use an intuitive name that will help you identify the type of sample you plan to analyze under these conditions. Some users prefer naming their analysis conditions files to describe the pressure tables they contain.

      Range: 0 to 40 alphanumeric characters

   b. **Low Pressure Equilibration:**

      Select a radio button to indicate whether equilibration is to be based on elapsed time (seconds) or decrease in rate of intrusion (or extrusion) in µL/g per second.

      Range: Time: 0 to 1000 seconds
      Rate: 0.000 to 1000.000 µL/g per second

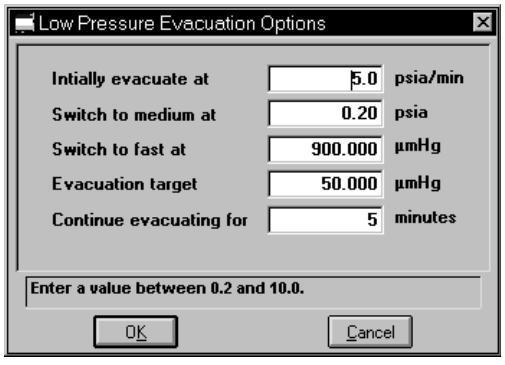
   c. **Low Pressure Max. intrusion volume:**

      The AutoPore automatically takes additional readings between points on the pressure table when an intrusion is detected. Enter the intrusion volume per gram of sample that must be reached in order for additional data pair readings (cumulative intrusion volume and absolute pressure) to be recorded. Use 0 to prevent readings between pressure points. Refer to Appendix F for additional information on choosing this option.

      Range: 0.000 to 100.000 mL/g

   d. **Evacuation:**

      Click Evacuation to open the Low Pressure Evacuation Options window.



      - **Initially evacuate at:** Enter the initial maximum evacuation rate.

        Range: 0.2 to 10.0 psia/min
        0.001 to 0.068 MPa/min

- **Switch to medium at:** Enter the pressure you wish the system to reach before medium evacuation begins.

  Range: 0.10 to 0.50 psia
  0.0007 to 0.0034 Mpa

- **Switch to fast at:** Enter the pressure you wish the system to reach before fast evacuation begins.

  Range: 100.000 to 900.000 µmHg

- **Evacuation Target:** Enter the evacuation pressure.

  Range: 15.000 to 500.000 µmHg

- **Continue evacuation for:** Enter the number of minutes you wish the evacuation to last.

  Range: 0 to 10000 minutes

Click OK to close the Low Pressure Evacuation Options dialog.

e. **High Pressure Equilibration:**

Mark a radio button to indicate whether equilibration is to be based on elapsed time (seconds), decrease in rate of intrusion (or extrusion) in µL/g per second, pressure controlled scan, or intrusion controlled scan.

- **Time** If you choose time, enter the number of seconds.

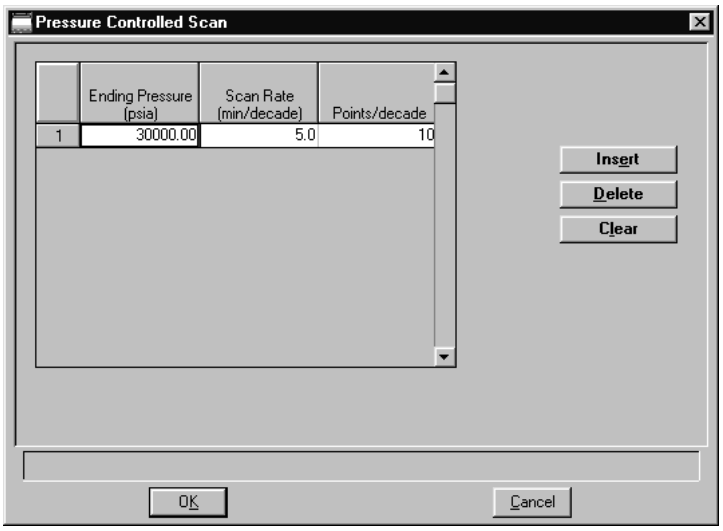
  Range: 0 to 10000 seconds.

- **Rate:** If you choose rate, enter the µL/g per second.

  Range: 0.000 to 10000.000 µL/g per second

- **Pressure Controlled Scan:** In pressure scanning mode, the instrument goes through a sequence of segments, with each segment starting at the end of the previous one. Each segment ends at the pressure you specify. The pressure is programmed to increase or decrease at a rate to give a constant time per decade of pressure. Along the way, the instrument takes intrusion points at the specified number of points per decade, ending at the specified ending pressure. Also, any points in the pressure table, as well as points separated by the maximum intrusion volume, are collected.

  If you choose pressure controlled scan, click Options to display the Pressure Controlled Scan dialog. This dialog contains a table of segments. Each row in the table contains the ending pressure, scan rate and points per decade for one segment.



Insert : Click to insert a row in the table.

Delete : Click to delete the selected row in the table.

Clear : Click to delete all the entries in the table.

Ending Pressure: Enter the ending pressure for this segment.

Range: 30 to 61000 psia
0.21 to 420.0000 MPa

Scan Rate (min/decade): Enter the minutes per decade for this segment.

Range: 1 to 180.0 min/decade

Points/decade: Enter the number of points per decade for this segment.

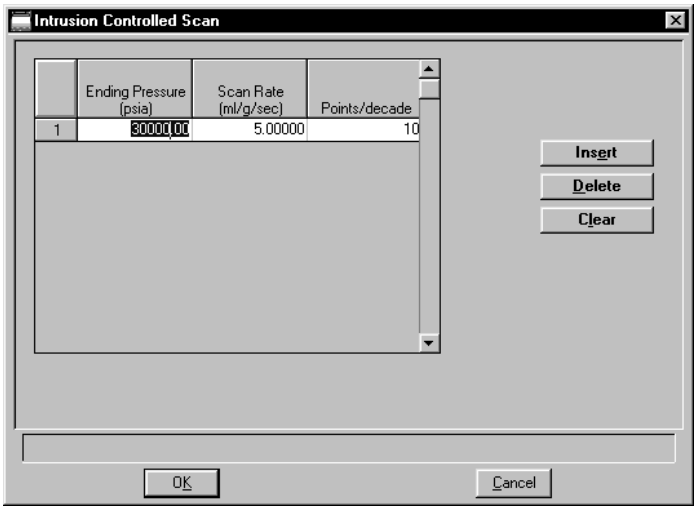
Range: 0 to 500

When you are finished editing the pressure controlled scan table, click **OK** to close the dialog box.

- **Intrusion Controlled Scan:** In intrusion scanning mode, the instrument goes through a sequence of segments, with each segment starting at the end of the previous one. Each segment ends at the pressure you specify. The pressure rate is themaximum achievable safe rate (up to 0.5 min/decade) and is programmed to increase or decrease at a rate to give a constant intrusion/extrusion rate. The instrument takes intrusion points at the specified number of points per decade, ending at the specified ending pressure. Also collected are points in the pressure table, as well as points separated by the maximum intrusion volume.

  If both high pressure ports are in use, they may have different intrusion rates. In this case, the left port (port 1) will determine when the data are collected. The right port will collect data at the same times as the left. This allows a differential analysis to be performed in this mode.

  If you choose intrusion controlled scan, click **Options** to display the Intrusion Controlled Scan dialog. This dialog contains a table of segments. Each row in the table contains the ending pressure, scan rate and points per decade for a segment.



Insert: Click to insert a row in the table.

Delete: Click to delete the selected row in the table.

Clear: Click to delete all the entries in the table.

Ending Pressure: Enter the ending pressure for this segment.

Range: 30 to 61000 psia
0.21 to 420.00 MPa

Scan Rate (mL/g/sec): Enter the intrusion rate to achieve for this segment.

Range: 0.00001 to 1.00000 mL/g/sec

Points/decade: Enter the number of points per decade for this segment.

Range: 0 to 500

When you are finished editing the intrusion controlled scan table, click OK to close the dialog box.

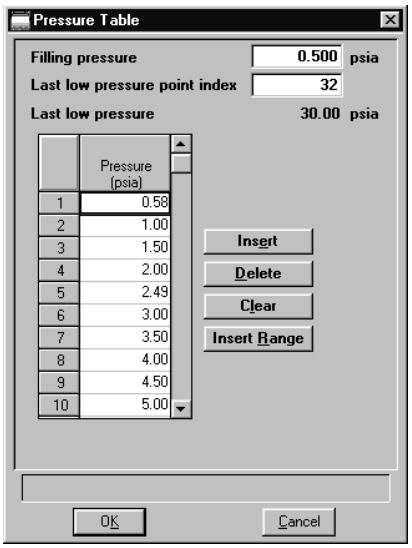
f. **High Pressure Max. intrusion volume:**

The AutoPore automatically takes additional readings between points on the pressure table when a significant intrusion is detected. Enter the intrusion volume per gram of sample that must be reached in order for additional data pair (cumulative intrusion volume and absolute pressure) readings to be recorded. Use 0 to prevent readings between pressure points. Refer to Appendix F for additional information on choosing this option.

Range: 0.000 to 100.000 mL/g

g. **Pressure:**

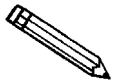
Click Pressure... to enter the pressure table data to be used for this analysis. During analysis, the AutoPore automatically records the corresponding volume of mercury intrusion at the equilibration point achieved for each pressure point you have included in the table. (See *Manually Entered or Collected Data* in this chapter for instructions on manual data entry.)



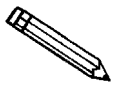
If you plan to generate pressures in the low pressure system above 30 psia (0.2068 MPa), you must use a penetrometer with threaded closures. BEFORE opening the pressure table, make sure that **Threaded Penetrometers** is selected on the Options pull-down menu.

- **Filling pressure:** The penetrometer is filled with this pressure before data are collected. Generally, it is recommended that you set the filling pressure slightly lower than your first low pressure point on the pressure table.

  Range: 0.200 to 20.000 psia
  0.001 to 0.14 MPa

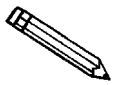


A filling pressure of at least 0.5 psia is recommended. Because mercury generates pressure and because fill pressures less than 0.5 psia can fail to fill the corner radii and gaps between the glass and sample in the penetrometer, using a lower pressure may reduce the accuracy of data.

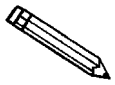


If the filling pressure is higher than any point in the table, an error message is displayed. Delete the pressures lower than the filling pressure or change the filling pressure.

- **Last low pressure point index (column):** Enter the number that corresponds to the point on the pressure table which you wish to be the last low pressure. (Use the index number on the left side of the table, not the actual pressure.) If you use 0, all measurements are made during the high pressure analysis.



You should complete this field after you have finished your pressure table entries. This enables you to be certain that you specify the right index number.



Atmospheric pressure plus the penetrometer's head pressure is the lowest pressure that can be used in the high pressure system (since lower pressures are actually vacuum). If you specify a Last Low Pressure below this number, you will see a warning message. Select a higher pressure.

- **Pressure (column):** Enter the pressure points at which data are to be taken. The table may contain up to 2500 pressure points and must contain at least one point.

  Pressing Ctrl ↓ simultaneously causes pressure points to be added to the table.

  – If you enter two points, then press Ctrl ↓, subsequent points are incremented by the difference between the two previous points.

  – If you enter a single point, then press Ctrl ↓, subsequent points are incremented by the value of the first point.

- **Insert:** Click to insert a pressure point before a selected point. The program suggests the next number based on the existing pressure point sequence. You may change the number, if desired.

- **Delete:** Click to delete a selected point.

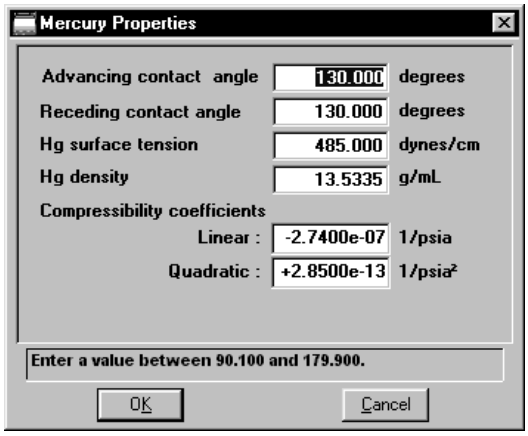
- **Clear:** Click to clear the pressure table.

- **Insert Range:** Click to open the Enter Pressure Range window. This dialog allows you to insert a series of geometrically spaced pressure points.

When you are finished editing the pressure table, click OK to close.

h. **Mercury:**

Click Mercury to open the Mercury Properties window. Mercury properties may change with variations in temperature.



- **Advancing contact angle:** Enter the advancing (intrusion) contact angle.

  Range: 90.100 to 179.900 degrees

- **Receding contact angle:** Enter the receding (extrusion) contact angle.

  Range: 90.100 to 179.900 degrees

- **Hg surface tension:** Enter the surface tension of mercury you are using.

  Range: 400.000 to 600.000 dynes/cm

- **Hg density:** Enter the density of mercury you are using.

  Range: 13.0000 to 14.0000 g/mL

- **Compressibility coefficients:**

  Linear: Enter the linear compressibility coefficient.

  Range: -1.0 to 0.0 l/psia
  -1.4564×10^{2} to 0.0 l/MPa

  Quadratic: Enter the quadratic compressibility coefficient.

  Range: -1.00 to 1.0 $1/psia^2$
  -2.1036×10^{4} to 2.1036×10^{4} $1/MPa^2$

Click OK to close.

Creating Report Options Files

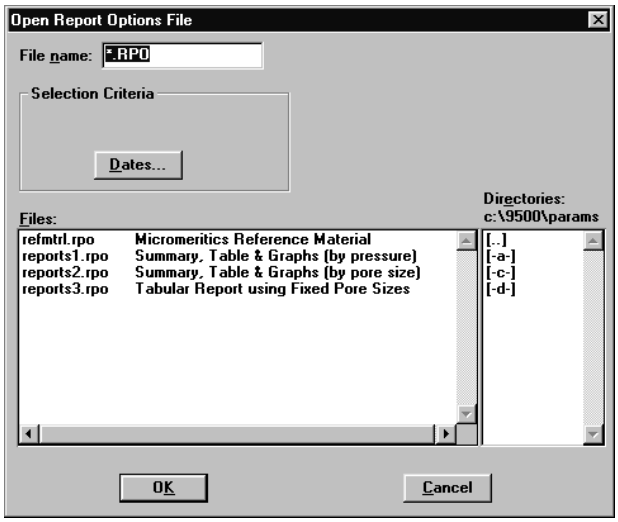
Reports contained in each sample information file are generated automatically when analysis is complete. Report options files specify the types of reports you wish to generate. They also help you customize details of reports, such as axis scale, axis range, and column headings.

You can set up your report options files to accommodate the special requirements of your work flow. For example, you can generate a simple report that lets you determine the basic characteristics of the sample. Then, use that report to make choices about the variables you wish to include in lengthier, more sophisticated reports.

A report options file for each of the standard report sets is included with the AutoPore software. Available reports include a tabular report, an analysis options listing, a summary, and 10 user-configurable graphs.

This section includes instructions for creating or editing a report options file or default file. Instructions for generating reports are found in Chapter 7.

1. From the File menu, select **Open**, **Report options**. The Open Report Options File window is displayed.

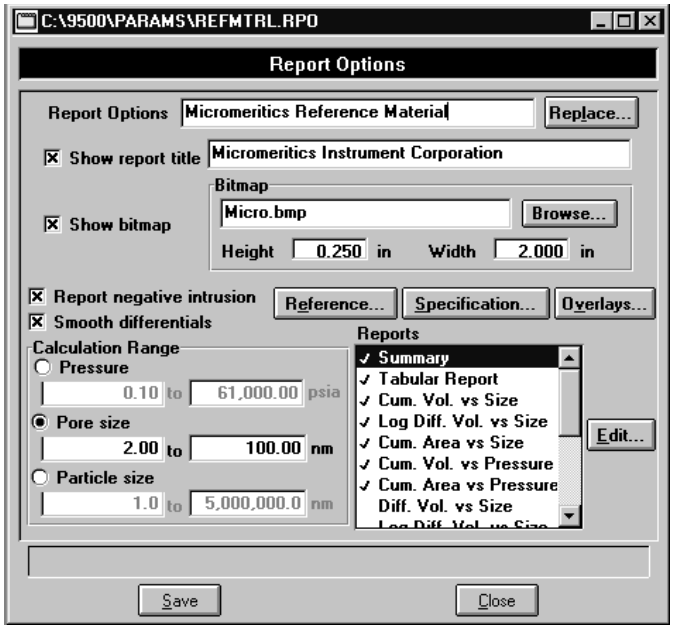


2. Create a name for this file and enter it in the **File name**: field.

3. Click OK. If this is a new file, the following message appears:

   File (file name) does not exist. Do you wish to create it?

4. Click Yes. The Report Options window opens.



5. Make any necessary changes to the variables:

   a. **Report Options (identifier):**

      Enter the name by which the Report Options file will be identified.

      Range: 40 alphanumeric characters, upper or lower case

   b. **Replace**

      Click Replace to copy the parameters of an existing file and use them in the current one.

      Select the desired file, click OK; the values are copied automatically and inserted into the current file.

      You can edit the values in the new file without affecting the values in the file from which they were copied.

   c. **Show report title**

      Select this option to have a title appear on your report, then enter the desired title. If the Show Report title box is checked, the title will be centered at the top of all printed and displayed reports.

      Range: 50 alphanumeric characters, upper or lower case

d. **Show Bitmap**

Check this option to include a bitmap graphic positioned just above the report title. For example, you may wish to include a company logo. Enter the file name or use Browse to select the file, then specify a height and width.

Range: 0.100 to 7.500 inches

e. **Report negative intrusion:**

Select this box to report small incorrect polarities (negative intrusions or positive extrusions) which may indicate the presence of noise, improper blank correction, or instrument malfunction.

f. **Smooth differentials:**

Select this box to apply smoothing to any differentials reported in tables or on graphs. When smoothing is applied, the column headers and graph axes are so labeled.

g. **Calculation Range:**

Select a radio button to indicate whether you wish reports to be limited in range by pore size, pressure, or particle size; then enter the range(s). The "from" value (left field) must be less than the "to" value.

- **Pressure:**

  Range: 0.10 to 61000.000 psia
  0.0007 to 420.5801 MPa

- **Pore size:**

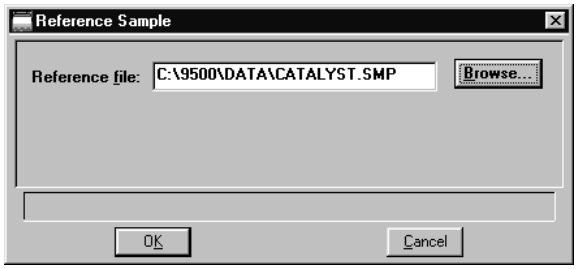
Range:	**Diameter**:	**Radius:**
	0.00050 to 5,000.00000	0.00025 to 2,500.00000 µm
	0.50 to 5,000,000.00	0.25 to 2,500,000.00 nm
	5.0 to 50,000,000.0	2.5 to 25,000,000.0 Å

- **Particle Size:**

Range:	**Diameter**:	**Radius:**
	0.0010 to 5,000.0000	0.0005 to 2,500.0000 µm
	1.0 to 5,000,000.0	0.5 to 2,500,000.0 nm
	10 to 50,000,000	5 to 25,000,000 Å

h. **Reference**

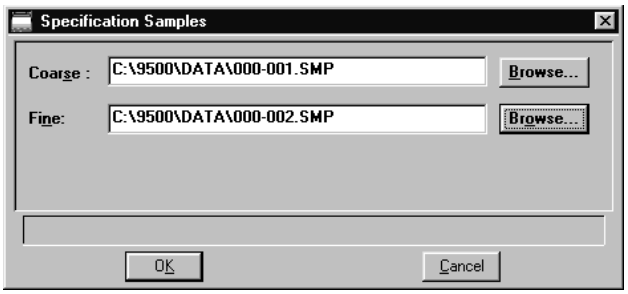
Click Reference to display the Reference Sample dialog so that you can specify a sample file with which to compare analysis results of the current sample.



Click Browse to display the Reference Sample File Selection dialog box. Choose the file you wish to use for the difference in reference calculation, then click OK; the file name is copied to the Reference Sample dialog.

i. **Specification**

Click Specification to display the Specification Samples dialog so that you can specify the sample files to be used for the boundaries of the coarse and fine specifications. Then you can quickly determine if the results of the current sample are within the specified boundaries.

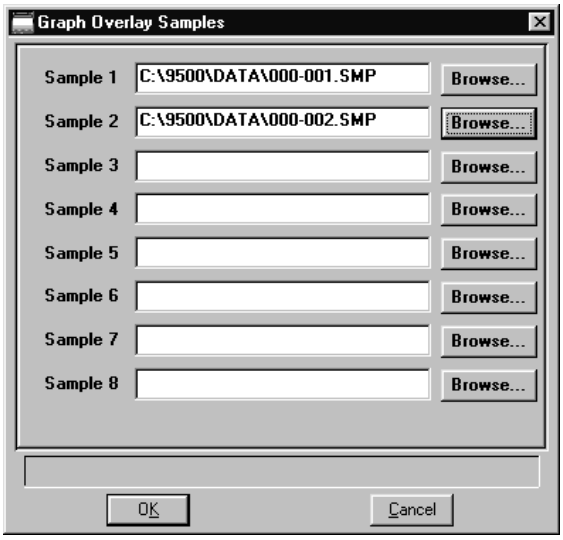


Click Browse to the right of each field to display the Specification Sample File dialog box containing a list of sample files from which to choose.

Choose a file, then click OK; the file name is copied into the Specification Samples dialog box.

j. **Overlays**

Click Overlays... to display the Graph Overlay Samples dialog so that you can choose the sample files containing the data you wish to overlay onto a selected plot.



Click Browse to the right of the sample number field; choose the desired file, then OK.

You can select up to eight files.

k. **Reports to Generate**

Contains a list of available reports.

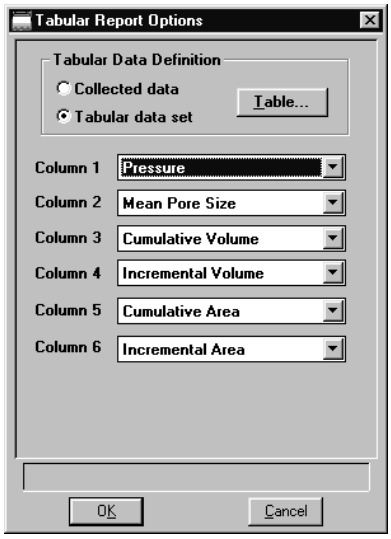
A report is selected when it is preceded with a check mark. Select reports by double-clicking on the desired report(s). You may *deselect* reports in the same manner.

l. **Edit**

Click Edit to edit values of the selected report. Refer to *Editing Tabular Reports, Editing Graphs, Editing the Summary Report, Editing Material Compressibility Options, Editing Throat Ratio Options,* and *Editing Fractal Dimension Options* in this chapter.

Editing Tabular Reports

Highlight the tabular report on the Report Options window; click **Edit** . The Tabular Report Options window is displayed.

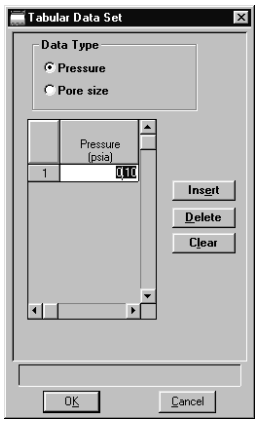


1. In the Tabular Data Definition box, indicate whether you wish to use **Collected data** points or a **Tabular data** set for this report.

   If you choose **Collected data**, your report will use data points collected during analysis. Data are collected at equilibration points on or about the pressure points you specified in the pressure table used for each analysis. These are the points that will be used in your report.

   A **Tabular data** set is a table of specific pressure points you wish to include in tabular reports. This feature enables you to better compare data from various runs, because the system interpolates values for each sample run at the points you specify in the table

   Select the Tabular data set radio button, then click **Table**; the Tabular Data Set window opens.



Adding Tabular Data

Select the **Data Type:** Choose pressure or pore size. The unit of measure is determined by your Data Presentation settings (from the Options menu).

Enter your **Data values**: Enter up to 2500 data values within the following ranges:

- **Pressure**
  Range: 0.100 to 61000.000 psia
  0.001 to 420.580 MPa
- **Diameter**
  Range: 0.0005 to 5000.0000 µm
  5 to 50000000 Å
- **Radius**
  Range: 0.0003 to 2500.0000 µm
  3 to 25000000 Å

The options buttons have the following functions:

Insert — Inserts a data point immediately before the selected point. One method for completing the tabular data set quickly is to enter the highest value in the set, then click on Insert to enter points below that value.

Delete — Deletes the selected data point.

2. Select a variable for up to 6 data columns. Use the individual drop-down lists. Choices include:

   None
   Pressure
   Pore Size
   Mean Pore Size
   Cumulative Volume
   Incremental Volume
   Differential Volume
   Log Differential Volume
   Cumulative Area
   Incremental Area
   Percent of Total Volume
   Percent Incremental Volume
   Particle Size (Mayer-Stowe method)
   Cumulative Volume Finer (Mayer-Stowe method)

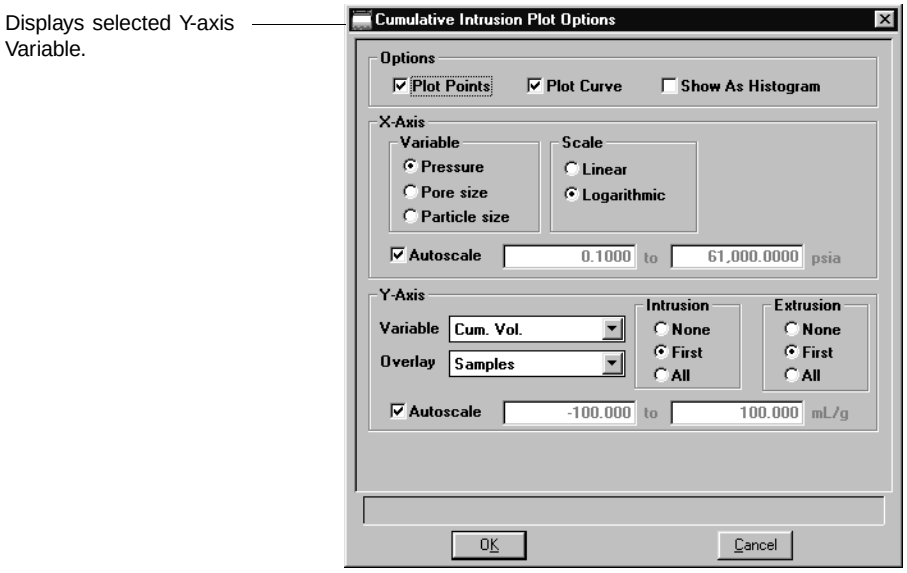
3. When you have completed the Tabular Report Options window, click OK to return to the Report Options window.

Editing Graphs

The AutoPore software gives you tremendous flexibility in creating customized graphs. You may:

- Use 10 different graph types per report options file
- Select x- and y-axis variables
- Overlay up to eight samples' data on a single graph
- Overlay a different variable on the y-axis

1. From the Report Options window, highlight a graph; click Edit. The editing window for this graph appears. The window is named for the Y-axis variable you select.



2. In the Options group box, indicate whether you wish your graph to **Plot Points**, **Plot Curve** (interpolated from data points), both points and curve, or **Show As Histogram**. If you select the latter choice, **Plot Points** and **Plot Curve** are disabled.

3. Make your X-axis choices:

- **Variable**: mark the *Pressure*, *Pore size*, or *Particle size* radio button
- **Scale:** mark the *Linear* or *Logarithmic* radio button
- **Scale range:** mark the *Autoscale* box, and the AutoPore automatically determines the graph's scale based on the data points collected. If you deselect Autoscale, You can specify the range of the graph's scale in the *From* and *To* boxes.

4. Make your Y-axis choices:

   a. **Variable:** Choose the Y-axis variable from the drop-down list.

      Cum. Vol. (Cumulative intrusion volume)
      Range: -100.000 to 100.000 mL/g

      Inc. Vol. (Incremental intrusion volume)
      Range: -100.000 to 100.000 mL/g

      Diff. Vol. (Differential intrusion volume)
      Range: -19999.99 to 200000.00 µm-mL/g (radius and µm)
      -9999.99 to 10000.000 µm-mL/g (diameter and µm)
      -1.999 to 2.000 Å-mL/g (radius and Å)
      -0.999 to 1.000 Å-mL/g (diameter and Å)

      Log Diff. Vol. (Log differential intrusion volume)
      Range: -10000.000 to 10000.000 mL/g

      Cum. Area (Cumulative pore area)
      Range: -10000.000 to 10000.000 m^2/g

      Inc. Area (Incremental pore area)
      Range: -10000.000 to 10000.000 m^2/g

      % Cum. Vol. (Percent Cumulative volume)
      Range: 0.000 to 100.000%

      % Inc. Vol. (Percent Incremental volume)
      Range: 0.000 to 100.000%

      Diff. Ref. % Vol. (Differential reference percent volume)
      Range: 0.000 to 100.000%

      Diff. Ref. Cum. Vol. (Differential reference cumulative volume)
      Range: -100.000 to 100.000 mL/g

      Diff. Ref. Cum. Area (Differential reference cumulative area)
      Range: -10000.000 to 10000.000 m^2/g

      Out Spec. % Vol. (Percent volume out of specification)
      Range: 0.000 to 100.000%

      Out Spec. Cum. Vol. (Cumulative volume out of specification)
      Range: -100.000 to 100.000 mL/g

      Out Spec. Cum. Area (Cumulative area out of specification)
      Range: -10000.000 to 10000.000 m^2/g

      Cum. Vol. Finer (Percent of cumulative volume finer)
      Range: 0.000 to 100.000%

b. **Overlay:** If you wish to overlay data on the Y-axis, choose from the drop down list:

None (no overlay on the Y-axis)

Samples (overlays the samples you select in the Overlays window, described at the end of this chapter)

Cum. Vol. (Cumulative intrusion volume)

Inc. Vol. (Incremental intrusion volume)

Diff. Vol. (Differential intrusion volume)

Log Diff. Vol. (Log differential intrusion volume)

Cum. Area (Cumulative pore area)

Inc. Area (Incremental pore area)

% Cum. Vol. (Percent Cumulative volume)

% Inc. Vol. (Percent Incremental volume)

(The variable chosen for the y-axis is omitted from the Overlay drop-down.)

c. **Intrusion**:

Mark a radio button to indicate which data points to plot:

- **None:** no intrusion data points
- **First:** points from the first intrusion/extrusion cycle
- **All:** all intrusion data points

d. **Extrusion:**

Mark a radio button to indicate which data points to plot:

- **None:** no extrusion data points
- **First:** points from the first intrusion/extrusion cycle
- **All:** all intrusion data points

e. **Scale range:**

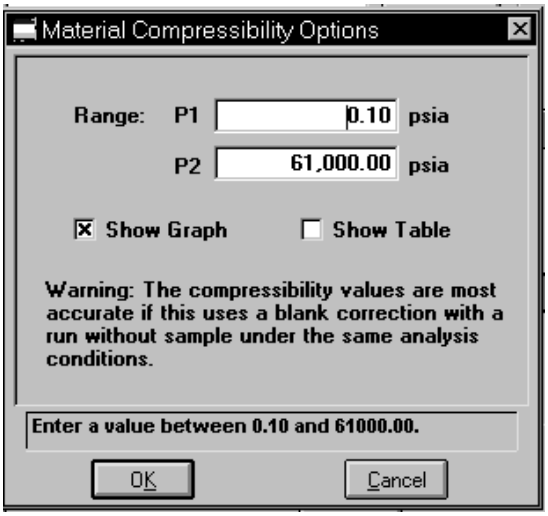
Mark the *Autoscale* box, and the AutoPore IV automatically determines the graph's scale based on the data points collected.

You can specify the range of the graph's scale in the *From* and *To* boxes if you choose not to Autoscale. Use values within the ranges listed above.

5. When you have completed the graph window, click OK to return to the Report Options window.

Editing Material Compressibility Options

Highlight Material Compressibility on the Report Options window; click Edit. The Material Compressibility Options window is displayed.



1. Enter the range of pressure over which the compressibility will be calculated.

   P1 - the beginning pressure
   Range: 0.10 to 61000.00 psia
   0.0007 to 420.5801 MPa

   P2 - the ending pressure
   Range: 0.10 to 61000.00 psia
   0.0007 to 420.5801 MPa

2. You can show the compressibility calculations in a graph, a table, or both. Both the graph and the table have a summary section that contains the linear and quadratic compressibility values and the RMS error to give an indication of the quality of the fit.

   - Select **Show Graph** to show the results in a graph. 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.

   - Select **Show Table** to show the results in a table. The table contains the following columns: Pressure, Volume Compressed, Predicted Volume Compressed, and Error.

3. Click OK to return to the Report Options window.

Editing Throat Ratio Options

Highlight Cavity to Throat Ratio in the Report Options window; click Edit. The Pore throat ratio Options window is displayed.

1. You can show the pore throat ratio in a table, a graph, or both.

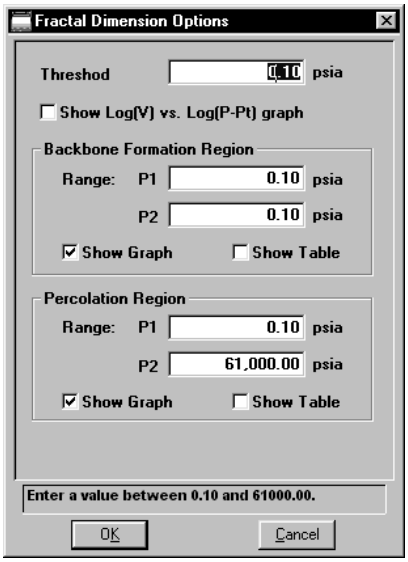
   - Select **Show Graph** to show the results in a graph. The graph plots percent porosity on a linear scale on the X-axis and pore throat ratio on the Y-axis.

   - Select **Show % Porosity Table** to show the results in a table. The table contains the following columns: Percent Porosity and Pore Throat Ratio.

2. Click OK to return to the Report Options window.

Editing Fractal Dimension Options

Highlight Fractal Dim. Perc. Reg. on the Report Options window; click Edit. The Fractal Dimensions Options window is displayed.



1. Enter the pressure at which the calculations are to be performed in the **Threshold** field.

   Range: 0.10 to 61000.00 psia
   0.0007 to 420.5801 MPa

2. Select **Show Log(V) vs. Log(P-P1) graph** if you want to generate an additional graph to help select linear range for calculations (P1,P2).

3. Enter the range of pressure over which the fractal dimension will be calculated for the Percolation Region and the Below Threshold Region.

   P1 - the beginning pressure
   Range: 0.10 to 61000.00 psia
   0.0007 to 420.5801 MPa

   P2 - the ending pressure
   Range: 0.10 to 61000.00 psia
   0.0007 to 420.5801 MPa

4. You can show the fractal dimension in a table, a graph, or both. Both the graph and the table have a summary section that contains the fractal dimension and the RMS error to give an indication of the quality of the fit. For the region below the threshold, the summary also contains the threshold pressure.

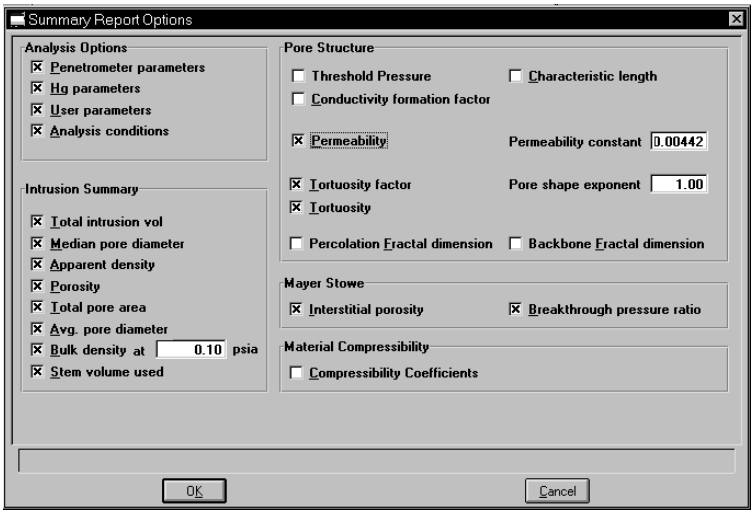
- Select **Show Graph** to show the results in a graph. The graph plots pressure on a log scale on the X-axis and the intrusion volume readings as points on the Y-axis, with the theoretical curve based on the calculated values overlaid.

- Select **Show Table** to show the results in a table. The table contains the following columns: Pressure, Intrusion volume, Predicted Intrusion Volume, and Error.

5. Click OK to return to the Report Options window.

Editing the Summary Report

1. Select the Summary Report on the Report Options window, then click Edit. The Summary Report Options dialog is displayed.



2. Select the options to be included in the report.

   a. **Analysis Options**

   Select the parameters you wish to include in the report.

   b. **Intrusion Summary**

   Select the intrusion options you wish to include in the report.

   If you check **Bulk density**, enter the pressure for the measurement.

   Range: 0.10 to 61000.00 psia
   0.007 to 420.5801 MPa

   If the pressure you enter is below the filling pressure, the filling pressure will be used on the report.

c. **Pore Structure**

Choose the pore structure parameters you wish to report.

If you wish to enter a Permeability constant, enter the value.

Range: 0.00250 to 0.01000

If you check **Tortuosity factor**, enter the **Pore shape exponent**.

Range: 0.00 to 1.00

d. **Mayer Stowe**

If you are reporting Mayer Stowe data, select the options you wish to report.

e. **Material Compressibility**

Select to report Compressibility coefficients if desired.

3. When you have completed the Summary Reports Options dialog, click OK to return to the Report Options window.

CHAPTER 5

PERFORMING AN ANALYSIS

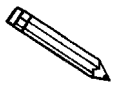
- Selecting Penetrometers
- Preparing the Sample
- Loading the Sample
- Sealing the Penetrometer
- Weighing the Assembled Penetrometer with Sample
- Low Pressure Operation
- High Pressure Operation
- High Pressure Differential Analysis
- Displaying the Instrument Schematic
- Cleaning Penetrometers
- Removing the Penetrometer Nut
- Suggested Sequence for Maximum Throughput (Leapfrogging Procedure)

PERFORMING AN ANALYSIS

A complete low pressure and high pressure analysis sequence follows these steps:

- Select a penetrometer
- Weigh the sample
- Load the sample
- Seal the penetrometer
- Weigh the assembled penetrometer and sample
- Install the penetrometer in the low pressure port
- Perform the low pressure analysis
- Remove the penetrometer from the low pressure port
- Weigh the penetrometer, plus sample, plus mercury (assembly weight)
- Install the penetrometer in the high pressure port
- Perform the high pressure analysis
- Remove the penetrometer from the high pressure port

Each of these steps is described, in order, in this chapter.

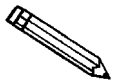


A complete sample file must be created for each sample before beginning analysis on that sample. See Chapter 4 for instructions on creating sample files.

Selecting Penetrometers

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

Penetrometers are available with three sample volumes, with five intrusion capacities, and in configurations appropriate for either solid pieces or powders.Table 5-1 lists parameters and part numbers.



If you plan to generate pressure greater than 30 psia in the low pressure analysis, you must use a penetrometer with threaded closures. Also, make sure that the *Threaded Penetrometers* option (on the Options menu) is selected.

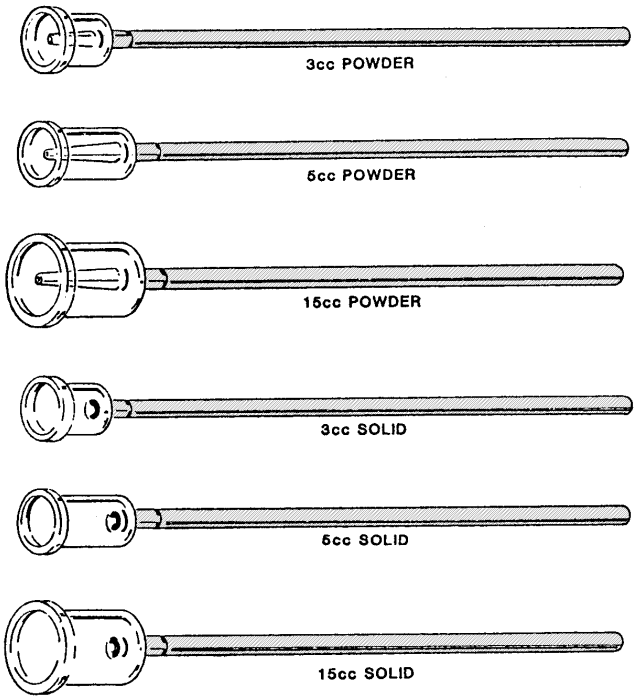


Figure 5-1. Penetrometers

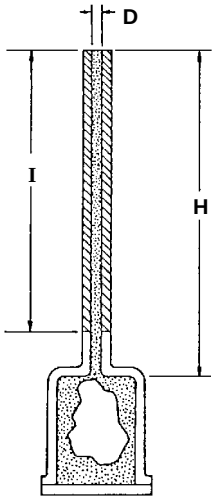
Make sure the sample nearly matches the size of the sample bulb and that the capillary volume is large enough to satisfy intrusion.

Table 5-1. Penetrometer Selection Guide

Bulb Volume (cc)	Sample Type	Maximum Measurable Volume (cc)	Total Stem Volume (cc)	Maximum Head Pressure		Physical Dimensions			Part Number
				(psia)	(kPa)	I (mm)	H (mm)	D (mm)	
3	Solid	0.387	0.412	4.68	32.3	227	242	1.473	950-61713-00
3	Solid	1.116	1.190	4.68	32.3	227	242	2.502	950-61715-00
3	Powder	0.387	0.412	4.68	32.3	227	242	1.473	950-61714-00
3	Powder	1.116	1.190	4.68	32.3	227	242	2.502	950-61716-00
5	Solid	0.366	0.392	4.45	30.7	215	230	1.473	950-61707-00
5	Solid	1.057	1.131	4.45	30.7	215	230	2.502	950-61709-00
5	Solid	1.716	1.836	4.45	30.7	215	230	3.188	950-61711-00
5	Powder	0.366	0.392	4.45	30.7	215	230	1.473	950-61708-00
5	Powder	1.057	1.131	4.45	30.7	215	230	2.502	950-61710-00
5	Powder	1.716	1.836	4.45	30.7	215	230	3.188	950-61712-00
15	Solid	0.366	0.392	4.45	30.7	215	230	1.473	950-61701-00
15	Solid	1.057	1.131	4.45	30.7	215	230	2.502	950-61703-00
15	Solid	1.716	1.836	4.45	30.7	215	230	3.188	950-61705-00
15*	Solid	3.007	(3.263)	4.45	30.7	215	230	4.813	950-61724-00
15*	Solid	3.857	(4.185)	4,45	30.7	215	230	4.813	950-61725-00
15	Powder	0.366	0.392	4.45	30.7	215	230	1.473	950-61702-00
15	Powder	1.057	1.131	4.45	30.7	215	230	2.502	950-61704-00
15	Powder	1.716	1.836	4.45	30.7	215	230	3.188	950-61706-00

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

Maximum Measurable Volume = $[(3.14)(D^2)(I)/4] \times [0.001\ cm^3/mm^3]$
Total Stem (Capillary) volume = $[(3.14)(D^2)(H)/4] \times [0.001\ cm^3/mm^3]$
Maximum Head Pressure (psia) = [H] x [0.01934 psia/mmHg]
Maximum Head Pressure (MPa) = [H] x [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 x 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 Table 5-1, Column 4). 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 you have a sample consisting 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 x 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 Table 5-1 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) x 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%.

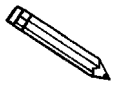
Volume of sample = mass/density = (15 g)/(3.5 g/cc) = 4.29 cc

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 Table 5-1 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 x 15/7.37]. The penetration volume would then be about 0.26 cc [0.13 x 15/7.37] or nearly 66% [0.26/0.39] of the total stem volume.



The calculations above 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 360 μm diameter, whereas a filling pressure of 1.5 psia will fill cavities as small as 120 μm diameter. If some interstitial volume remains unfilled at this point, allowance for this additional volume must be made in choosing the appropriate stem volume.

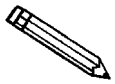
The percentage of the maximum intrusion (stem) volume utilized in each station is displayed on the Status Display as a guide for the operator. A % STEM reading of less than 25% or more than 90% suggests the need for a procedural change. The first instance suggests a larger quantity of sample might give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

Preparing the Sample

To achieve gains in productivity and reduction in instrument maintenance, as well as improved data quality, dry the sample material in a shallow pan at 150°C or higher for one hour. You may use a vacuum oven, 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.

Loading the Sample



It is recommended that you wear latex gloves when handling penetrometers, to avoid transferring skin oils, which may affect results.

To load the sample:

1. Enter the sample file name (or identifier) on a Sample Data Worksheet. A blank Sample Data Worksheet is included in Appendix A.

2. Weigh the sample using an analytical balance. Record the weight on the data sheet as **Sample weight**.

3. Hold the penetrometer with the stem down and carefully pour the sample into the bulb.

   When pouring powders into the bulb, place your finger over the stem opening in the center of the bulb so that powder does not enter the stem. You may find a small funnel useful for loading powders. Large granules or chunks may be loaded with forceps; touching such pieces with the fingers should be avoided as skin oils may be transferred that can slightly alter ultimate results or create evacuation problems.

4. Seal the penetrometer as described in the next section.

Sealing the Penetrometer

It is important that penetrometers are clean and dry. Instructions for cleaning penetrometers are found at the end of this chapter.

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. Apply grease as follows:

Follow these instructions carefully. Neither too much nor too little grease should be used. Too much grease exposes the sample to an unwanted coating and is likely to cause slippage and misalignment of the mating surfaces. Too little grease results in an imperfect seal.

   a. Using your fingertip, apply a light coating of grease to the lip of the bulb.

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

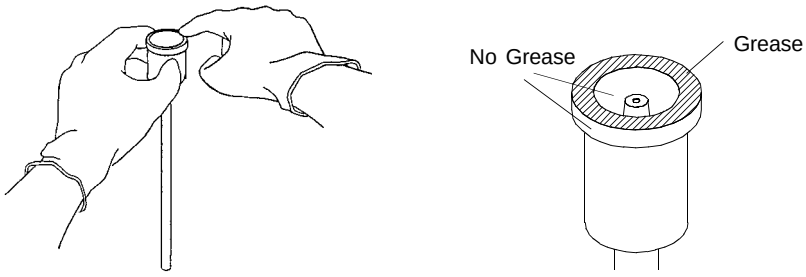


Figure 5-2. Greasing the Penetrometer

   c. Clean off any excess grease from both the inside and outside of the bulb.

2. After removing excess grease from the bulb, hold the penetrometer upright and place a seal on the bulb opening. Then turn the seal one-half turn to seat.

3. Slide the nut over the stem and bulb. Tighten the nut finger-tight.

4. Place the penetrometer tool over the nut.

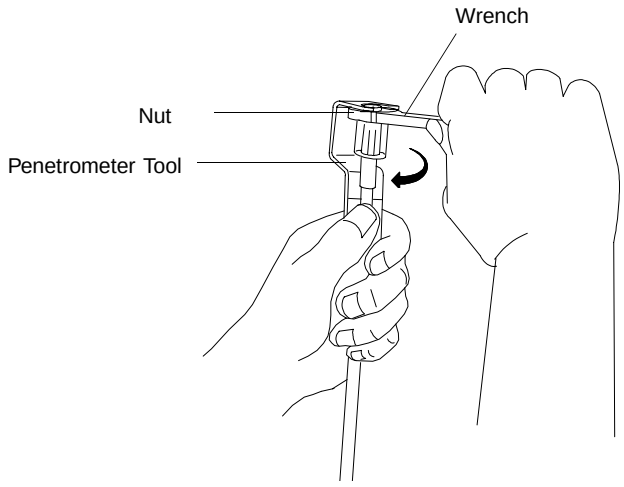


Figure 5-3. Sealing the Penetrometer

5. Position the wrench so the peg on the wrench fits into the notch on the nut.

6. While holding the penetrometer tool, turn the wrench clockwise to tighten the nut.

7. Weigh the penetrometer as described in the next section before installing it in the low pressure port.

Weighing the Assembled Penetrometer With Sample

1. Weigh the assembled penetrometer with sample using an analytical balance. Do not include the spacer when you weigh the penetrometer.

2. Record the weight on the Sample Data Worksheet as **Sample + Penetrometer** weight.

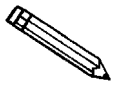
3. Subtract Sample weight from Sample + penetrometer weight; record on the Sample Data Worksheet as **Penetrometer weight**.

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

Low Pressure Operation

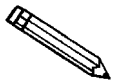
Installing Penetrometers in Low Pressure Ports

The AutoPore IV is designed to perform low pressure analyses on four samples at one time. If fewer than four samples are to be analyzed (or two samples if you are using the AutoPore IV 9510 or 9500), a blank rod must be installed in each unused low pressure port. If a penetrometer or blank rod is not installed in each port, vacuum conditions cannot be achieved and an analysis cannot be performed. The following instructions refer to one penetrometer; the others are installed in the same manner.



These instructions assume installation without prior samples in place. When previous low pressure testing has just been completed, penetrometers must be removed (following the proper procedures) before new ones can be installed. Refer to *Removing Penetrometers From Low Pressure Ports* **later in this chapter.**

1. A vacuum-tight seal must be made at the tip of the penetrometer stem. If you experience difficulty in obtaining a seal, lightly grease the first inch or so of the outside of the stem (using a silicone high vacuum grease). Wipe off any grease that might get on the flat end of the stem, being careful not to get grease inside the stem.



It is not necessary to grease the penetrometer stem before each analysis. Apply grease only if you have difficulty in obtaining a seal.

2. Remove the capacitance detector from the low pressure port by turning it counterclockwise and pulling forward. Place it on top of the AutoPore or stand it on the countertop next to the high pressure chamber.

3. Turn the retaining knob (See Figure 5-4) on the low pressure port counterclockwise until it turns with little resistance.

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

4. Install the two spacers over the penetrometer stem with the largest ends nearest the sample bowl. Insert the penetrometer stem into the port and push it in as far as it will go.

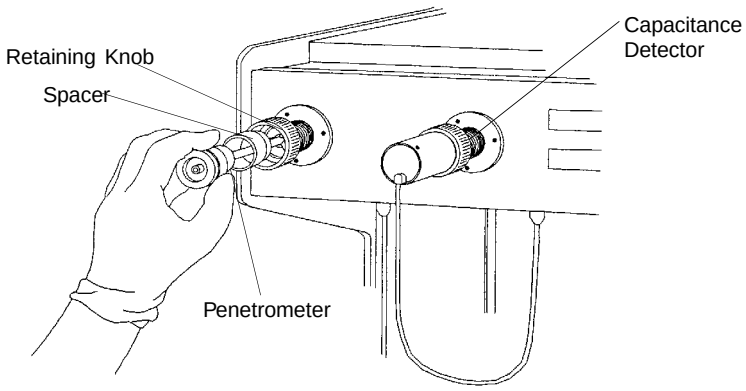


Figure 5-4. Installing Penetrometer in Low Pressure Port

5. Tighten the retaining knob by turning it clockwise until the penetrometer is firmly retained. Because it is lubricated, the penetrometer can be moved slowly back and forth, even after sufficient tightening is achieved. Hence, do not tighten with excessive force.

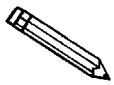
6. Install the capacitance detector over the penetrometer, turning it clockwise to the locked-on position.

Performing a Low Pressure Analysis

The Low Pressure Analysis wizard is comprised of a series of dialog boxes that enable you to:

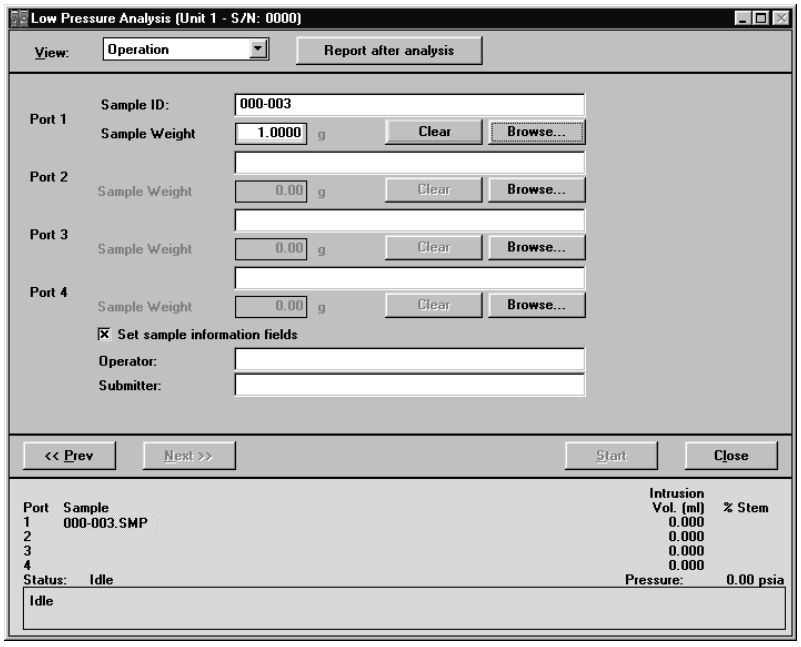
- Select the sample files to be used for analysis on each of the four ports, and, optionally, to change the sample weight.
- Edit the penetrometer properties to be used for analysis.
- Edit the analysis conditions.
- Monitor the analysis as data are being collected.

You may start the analysis from any of these dialog boxes. For example, if you want to use the information in the selected sample information file, without making any changes to it, you can select Start in the first dialog box to begin the analysis.



Before beginning an analysis, make sure 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 **Unit, Low pressure analysis** from the Main Menu; the Low Pressure Analysis dialog is displayed.



2. The **View** drop-down list enables you to select from among the following:

   - **Operation:** Select this option to perform a low pressure analysis. Follow the procedure described below.

   - **Instrument Log**: Select this option to display the instrument log. Refer to *Displaying the Instrument Log* in Chapter 3.

   - **Instrument Schematic**: Select this option to display the instrument schematic. Refer to *Displaying the Instrument Schematic* later in this chapter.

3. Click Browse... to select the sample file for the sample in the first port

   - If the sample file is not found, you will see a warning, and may choose to create the sample file now.

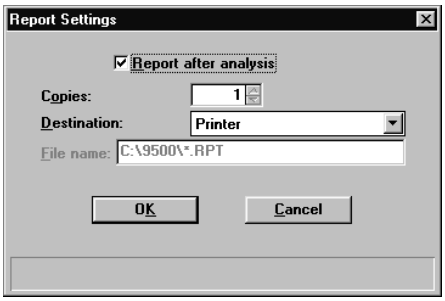
   - Low pressure analysis can only be performed on files whose status is *No Analysis*.

   - If you wish to change the sample weight, enter the weight in grams in the **Sample Weight** field.

4. Select files for the second, third, and fourth sample ports (if using) in the same manner. If the analysis conditions are not the same, you are given the option of choosing the proper ones on a later screen.

5. Select **Set sample information fields** to enter a different **Operator** and/or **Submitter ID**. The identification(s) will overwrite the one(s) shown in the original sample file(s). Do not select this option if you wish to retain the original identification(s).

6. Click Report after Analysis to have the reports in the reports options file automatically generated after analysis. This dialog displays:



Click the **Report after analysis** check box, then select the destination from the drop-down list. The choices are: File, Printer, Printer/Plotter, and Screen.

- If you choose File, enter a file name (or accept the default).

- If you choose Printer or Printer/Plotter, select the number of copies you wish to print (up to four).

7. Choose one of the following:

   - If you want to edit the penetrometer properties or analysis conditions, select Next>> and follow the instructions for *Editing Penetrometer Properties* and *Editing Low Pressure Analysis Conditions* later in this chapter.

   - If you want to start the analysis immediately, click Start. The system starts the analysis and displays a live graph of the analysis as data are being collected. Refer to *Monitoring a Low Pressure Analysis* later in this chapter.

8. If you are using a 9500 or 9505 and your pressure table contains pressures greater than 33,000 psia, the following message is displayed:

   Unit [n] Warning these analysis conditions contain pressures that are too large for the high pressure system of this instrument. Do you want to proceed?

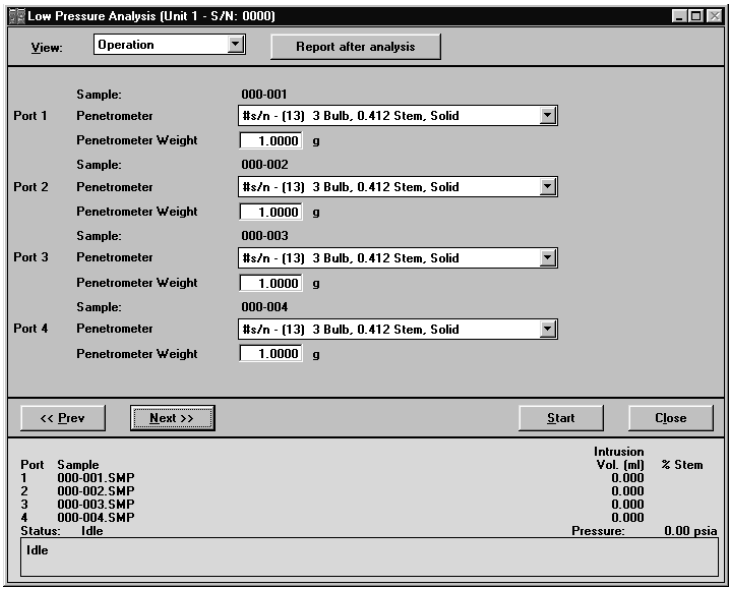
   - If a 9520 or 9510 is available to complete the high pressure portion of the analysis, choose Yes to proceed with the low pressure portion on this instrument. Then perform the high pressure portion on the 9520 or 9510.

   - If a 9520 or 9510 is unavailable, choose No to stop the low pressure analysis. Change the pressures in the pressure table so they will not exceed 33,000 psia and restart the analysis.

Editing Penetrometer Properties

The second dialog box of the Low Pressure Analysis wizard enables you to edit penetrometer properties.

1. When you click Next>> in the first dialog box of the Low Pressure Analysis wizard, this dialog box displays.



2. The penetrometer properties contained in the selected sample file are displayed. You can click the drop-down list arrow in the **Penetrometer** field for any port to select another penetrometer properties file.

   If you want to change the weight, enter the weight (in grams) in the **Penetrometer Weight** field.

3. Choose one of the following:

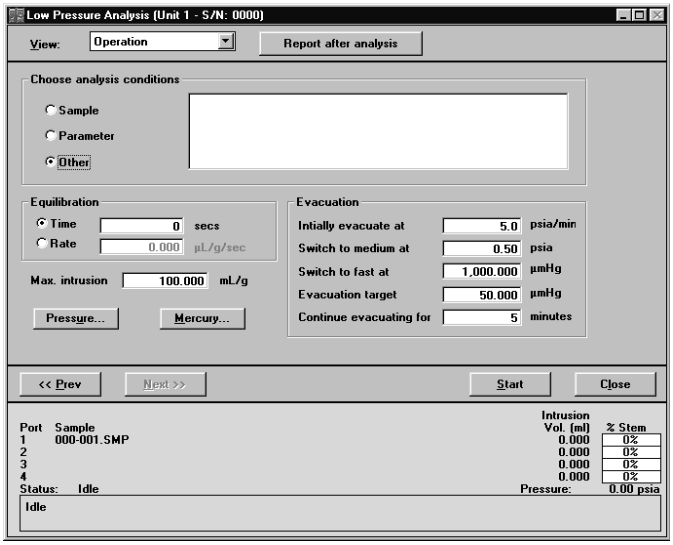
   - If you want to edit the analysis conditions, select Next>> and follow the instructions for *Editing Low Pressure Analysis Conditions* later in this chapter.

   - If you want to start the analysis immediately, click Start. The system starts the analysis and displays a live graph of the analysis as data are being collected. Refer to *Monitoring a Low Pressure Analysis* later in this chapter.

Editing Low Pressure Analysis Conditions

The third dialog box of the Low Pressure Analysis wizard enables you to edit analysis conditions.

1. When you click Next>> in the second dialog box of the Low Pressure Analysis wizard, this dialog box displays.



2. The analysis conditions contained in the selected sample file are displayed. You can select one of the following types of analysis conditions:

   - Click **Sample** to display a list of sample IDs. Select the sample ID of the sample whose analysis conditions you want to use.

   - Click **Parameter** to display a list of analysis conditions files. Select the file you want to use.

   - Click **Other** to enable the fields in the lower portion of the dialog box; enter your changes. (Instructions for completing each field are included in *Creating Analysis Conditions Files* in Chapter 4.)

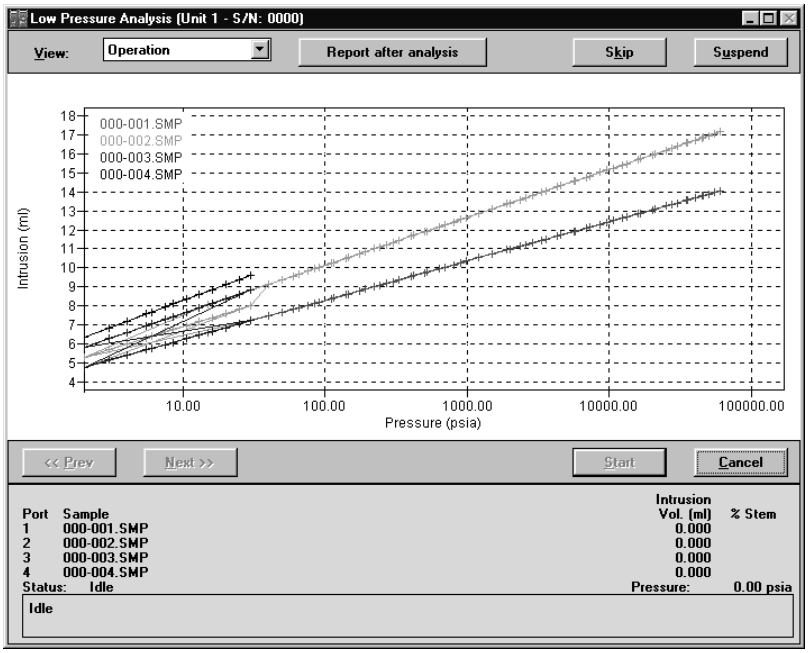
3. Click Pressure... to choose, enter, or edit the pressure table to be used for this analysis. Complete instructions are found in *Creating Analysis Conditions Files* in Chapter 4.

4. Click Mercury... to open the Mercury properties window. Complete instructions for using the Mercury properties window are found in *Creating Analysis Conditions Files* in Chapter 4.

5. Click Start. The system starts the analysis and displays a live graph of the analysis as data are being collected. Refer to *Monitoring a Low Pressure Analysis* below.

Monitoring a Low Pressure Analysis

When you start the analysis, this dialog box displays.



1. This dialog box displays a live graph of the analysis as the data are being collected. It shows intrusion as a function of pressure.

   The lower portion of the dialog box displays the following for each port:

   - The file name/Sequence # of the sample file.
   - The intrusion volume.
   - The percent of the penetrometer stem that is filled with mercury.

   Status: Gives the low pressure system's current status.

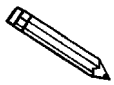
   Pressure: Displays the current pressure in the low pressure system. The pressure is shown in the units selected on the Data Presentation window of the Options menu or in µmHg, when appropriate. The reading shown is from either the vacuum gauge or the 50 psia transducer, depending upon which is currently in range. A simultaneous display of both readings can be viewed in the Transducer Calibration window (select **Unit, Calibration, Transducers** from the Main Menu).

   Skip: Click Skip if you want to skip the next step in the analysis. If you click Skip while a point is being equilibrated, the point will be recorded, but will not be equilibrated for the remaining time.

Suspend/Resume: Click Suspend if you want to suspend the analysis, holding pressure steady. Click Resume to restart the analysis.

Cancel: Click Cancel if you want to cancel the analysis.

2. When the analysis is finished, the results remain displayed and Cancel changes to Close . When you are finished viewing analysis results, click Close , or Next>> to return to the opening screen.



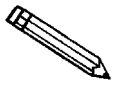
When monitoring a low pressure analysis, you can display the instrument schematic to observe the state of the low pressure system components. Refer to *Displaying the Instrument Schematic* **later in the chapter.**

Removing Penetrometers from Low Pressure Ports

1. Make sure the low pressure ports have returned to near atmospheric pressure.
2. Make sure the Mercury Drained indicator is illuminated.

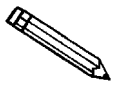
Removing penetrometers when the Mercury Drained indicator is not illuminated may allow mercury to spill from the port. Access Manual Mode and drain the low pressure system. Instructions are found in Chapter 8, *Troubleshooting and Maintenance***.**

3. Hold the retaining knob to prevent rotation, and then turn the capacitance detector counterclockwise to loosen and remove it.
4. Turn the retaining knob counterclockwise, then carefully withdraw the penetrometer assembly. Do not pull on the cap.



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

5. Remove the spacer.
6. 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. Weigh the penetrometer assembly (remove the spacer first) if density calculations are to be made or blank correction by formula is used. Record this weight 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.

High Pressure Operation

The AutoPore IV 9520 and 9505 are designed to perform two high pressure analyses at the same time. (The AutoPore IV 9510 and 9500 perform one high pressure analysis.) If you wish to run only one analysis, the other chamber must be closed tightly and have sufficient high pressure fluid to be drawn into the vent valve. The following instructions refer to one penetrometer; the other is installed in the same manner.

Installing Penetrometers in the High Pressure Chambers

Before opening the high pressure chamber, look at the status display to make sure that the system is not pressurized.

1. Turn the vent valve slowly counterclockwise to release excess pressure.

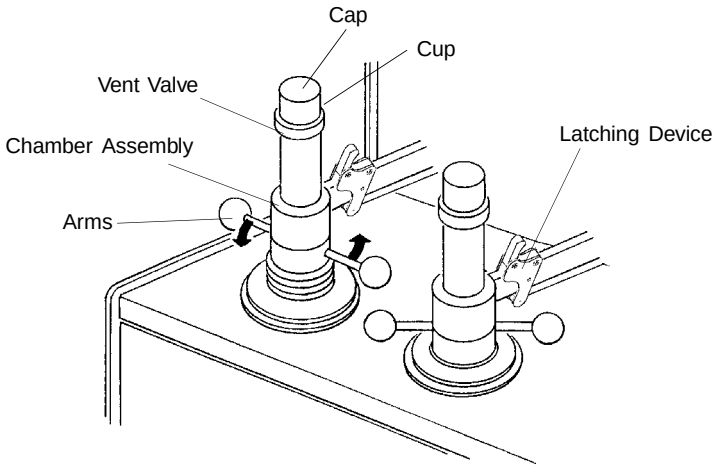


Figure 5-5. Opening the High Pressure Chamber

2. Unscrew the chamber plug assembly by turning the arms counterclockwise. Lift the chamber plug assembly upward 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, part way into the chamber.

4. Guide the penetrometer stem upward into the chamber plug assembly.

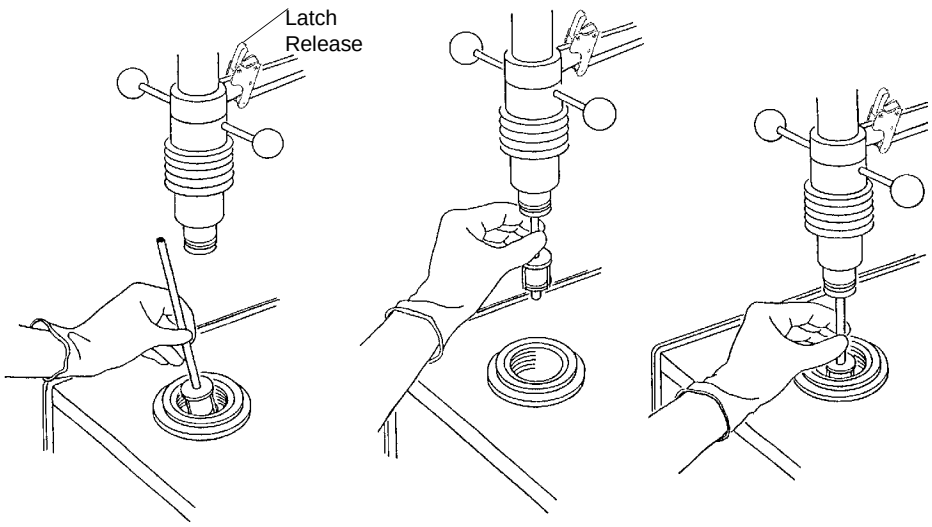


Figure 5-6. Installing the Penetrometer in the High Pressure Chamber

5. Pull the latch release forward to unlock the chamber plug assembly; then lower the assembly until it is two or three inches above the pressure chamber. Now gently slide the penetrometer into the pressure chamber until it makes contact with the banana plug in the bottom of the chamber. Push the penetrometer downward to ensure proper contact, but do not allow the penetrometer stem to leave the chamber plug assembly.

6. Check the high pressure fluid level. It is essential that the high pressure fluid is level with the ledge. If the level is below the visible ledge, add high pressure fluid to bring the level to the ledge. If the level is above the ledge, remove fluid to bring the level to the ledge.

Sealing the High Pressure Chamber

The chamber plug on Micromeritics' porosimeters 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.

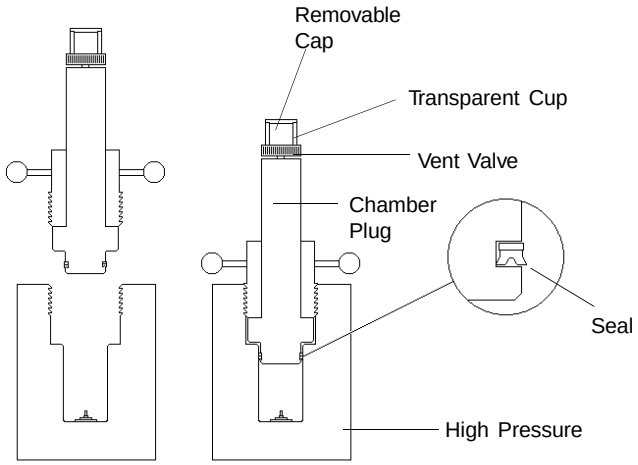
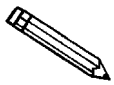


Figure 5-7. Sealing the High Pressure Chamber

Use the following procedure to seal the high pressure chamber:

1. Push the plug into the chamber until you feel it contact the shoulder of the chamber. Several threads of the plug will remain exposed.

2. Make sure the vent valve is partially unscrewed (open). *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 atop the vent valve.

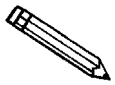


Tightening the plug too quickly may cause unwanted intrusion caused by pressure created when you close the chamber.

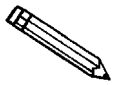
3. The cup should not be completely full. If the cup is too full, slowly open the chamber and recheck the fluid level. It is likely that you need to remove fluid. Repeat steps 1, 2, and 3 to release any trapped air.

4. 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. Some tiny bubbles may remain; they 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 you need to add fluid. Repeat steps 1, 2, and 3 to release any trapped air.



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



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

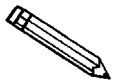
1. **Too much fluid. Remove fluid, clean the seal and try again.**
2. **Damaged seal. Replace the seal, then try again.**
3. **Fluid in the threads. Clean and reseal.**

Performing High Pressure Analyses

The High Pressure Analysis wizard is comprised of a series of dialog boxes that enable you to:

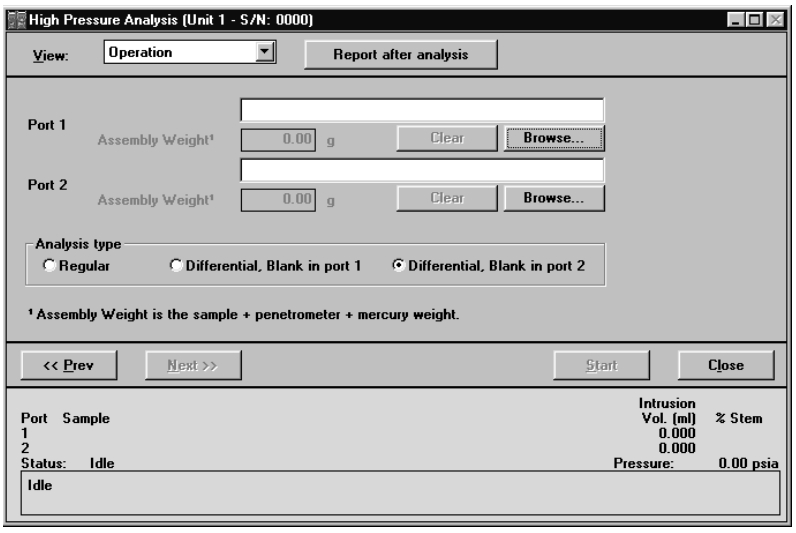
- Select the sample files to be used for analysis on each port, and, optionally, to change the sample weight.
- Edit the analysis conditions.
- Monitor the analysis as the data are being collected.

You may start the analysis from any of these dialog boxes. For example, if you want to use the information in the selected sample information file, without making any changes to it, you can select Start in the first dialog box to begin the analysis.



Each sample's high pressure analysis should be performed on the same analyzer as the low pressure analysis. The AutoPore 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. You may continue the analysis or cancel.

1. Select **Unit**, **High pressure analysis** from the Main Menu. The Start High Pressure Analysis window opens.



2. The **View** drop-down list enables you to select from among the following:

   - **Operation:** Select this option to perform a high pressure analysis. Follow the procedure described below.

   - **Instrument Log**: Select this option to display the instrument log. Refer to *Displaying the Instrument Log* in Chapter 3.

   - **Instrument Schematic**: Select this option to display the instrument schematic. Refer to *Displaying the Instrument Schematic* later in this chapter.

3. Click Browse... to select the sample file for the sample in the first port.

   - If the sample file is not found, you will see a warning, and may choose to create the sample file now.

   - High pressure analysis can only be performed on files whose status is "Low Pressure Complete" or "High Pressure Complete."

   - If you wish to change the assembly weight (sample + penetrometer + mercury weight), enter the weight (in grams) in the **Assembly Weight** field.

   If you are using a 9500 or 9505 to perform this high pressure analysis and the sample file you selected contains pressures which exceed 33,000 psia, the following message is displayed:

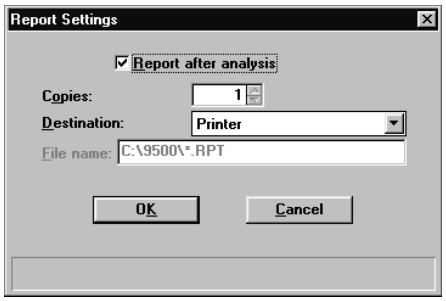
   > **Unit [number] Warning these analysis conditions contain pressures that are too large for the high pressure system of this instrument. Do you want to proceed without the high pressures?**

   - Choose Yes if you wish to proceed with the analysis; only pressures less that 33,000 psia will be used.

   - Choose No to cancel the analysis. Perform the analysis on a 9520 or 9510 if available.

4. If you are using a second sample file in this analysis, select that file's name in the same manner.

5. Click **Report after Analysis** if you wish the reports in the report options file to be generated automatically after analysis. This dialog displays:



Click the **Report after analysis** check box, then select the destination from the drop-down list. The choices are: File, Printer, Printer/Plotter, and Screen.

- If you choose File, enter the file name (or accept the default).

- If you choose, Printer or Printer/Plotter, select the number of copies you wish to print (up to four).

6. Click the radio button for the type of analysis you wish to perform:

   - Click **Regular** to perform a standard analysis.

   - Click **Differential, Blank in port 1** to perform a differential analysis with the blank penetrometer in port 1. Refer to *High Pressure Differential Analysis* later in this chapter.

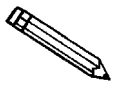
   - Click **Differential, Blank in port 2** to perform a differential analysis with the blank penetrometer in port 2. Refer to *High Pressure Differential Analysis* later in this chapter.

7. Choose one of the following:

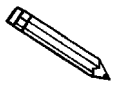
   - If you want to edit the analysis conditions, select Next>> and follow the instructions for *Editing High Pressure Analysis Conditions* later in this chapter.

   - If you want to start the analysis immediately, click Start. The system starts the analysis and displays a live graph of the analysis as data are being collected. Refer to *Monitoring a High Pressure Analysis* later in this chapter.

If you did not request that transducer offset be determined (Main Menu: **Options**, **Atmospheric pressure** window), and if the penetrometer head pressure differs from the atmospheric pressure, the system asks you to verify that the system is at atmosphere. You are also given the option of verifying the transducer offset.



You may start a low pressure run, if desired. Simultaneous low and high pressure runs can be performed.

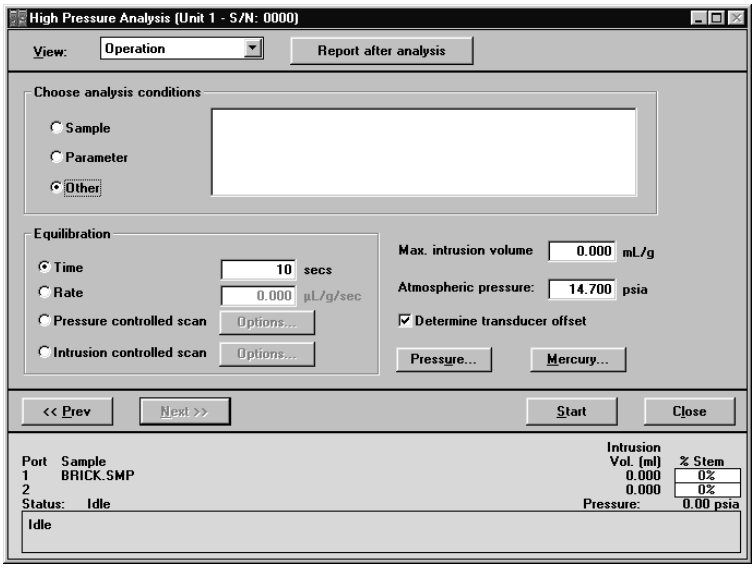


For maintenance purposes, it is possible to perform a high pressure analysis with no penetrometer. Open the High Pressure Analysis window and enter the name of a file with *No Analysis* as its status. You may proceed with the analysis after a warning message.

Editing High Pressure Analysis Conditions

The second dialog box of the High Pressure Analysis wizard enables you to edit analysis conditions.

1. When you click Next>> in the first dialog box of the High Pressure Analysis wizard, this dialog box displays. It enables you to select a single set of conditions for the analysis.



2. The analysis conditions contained in the selected sample file are displayed. You can select one of the following types of analysis conditions:

   - Click **Sample** to display a list of sample IDs. Select the sample ID of the sample whose analysis conditions you want to use.

   - Click **Parameter** to display a list of analysis conditions files. Select the file you want to use.

   - Click **Other** to enable the fields in the lower portion of the dialog box. Enter your changes. (Instructions for completing each field are included in *Creating Analysis Conditions Files* in Chapter 4.)

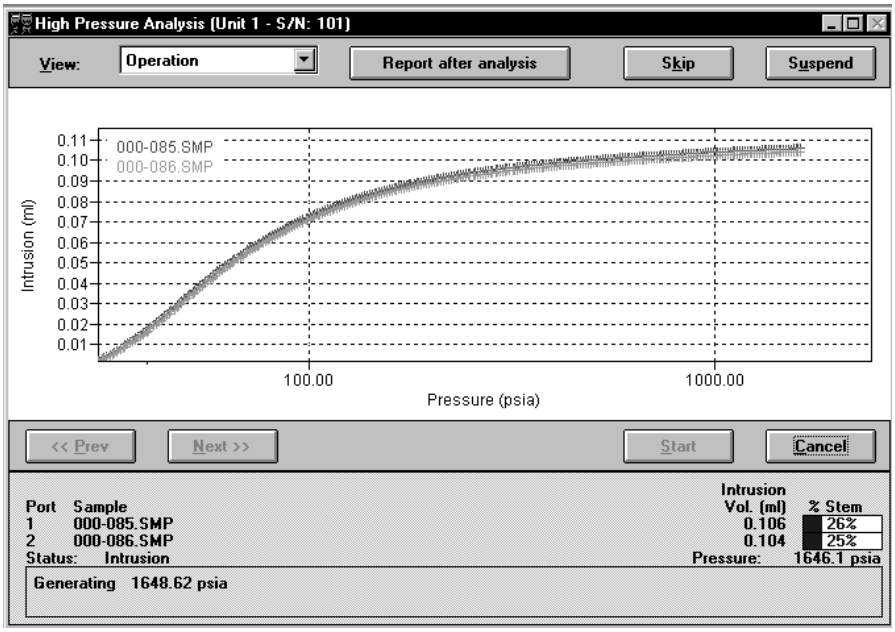
3. Click Pressure... to choose, enter, or edit the pressure table to be used for this analysis. Complete instructions are found in *Creating Analysis Conditions Files* in Chapter 4.

4. Click Mercury... to open the Mercury properties window. Complete instructions for using the Mercury properties window are found in *Creating Analysis Conditions Files* in Chapter 4.

5. Click Start. The system starts the analysis and displays a live graph of the analysis as data are being collected. Refer to *Monitoring a High Pressure Analysis* in this chapter.

Monitoring a High Pressure Analysis

When you start the analysis, this dialog box displays.



1. This dialog box displays a live graph of the analysis as the data are being collected. It shows intrusion as a function of pressure.

   The lower portion of the dialog box displays the following for each port:

   - The file name/Sequence # of the sample file.
   - The intrusion volume.
   - The percent of the penetrometer stem that is filled with mercury.

   Status: Gives the high pressure system's current status.

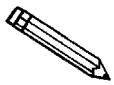
   Pressure: Displays the current pressure in the high pressure system. The pressure is shown in the units selected on the Data Presentation window of the Options menu. The reading shown is from the 60K psia transducer.

   Skip: Click Skip if you want to skip the next step in the analysis. If you click Skip while a point is being equilibrated, the point will be recorded, but will not be equilibrated for the remaining time.

Suspend/Resume: Click Suspend if you want to suspend the analysis, holding pressure steady. Click Resume to restart the analysis.

Cancel: Click Cancel if you want to cancel the analysis.

2. When the analysis is finished, the results remain displayed and Cancel changes to Close . When you are finished viewing analysis results, click Close .



When monitoring a high pressure analysis, you can display the instrument schematic to observe the state of the high pressure system components. Refer to *Displaying the Instrument Schematic* **later in the chapter.**

Removing Penetrometers From High Pressure Chambers

1. When analysis is finished, make sure the high pressure system indicator PRESSURIZED is not illuminated.

2. Loosen the vent valve approximately 1/8 turn (counterclockwise) to make the removal of the plug easier.

3. Unscrew the plug by turning the arms counterclockwise. 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.

4. Remove the penetrometer assembly. Hold it over the chamber for a few moments to allow the high pressure fluid to drain.

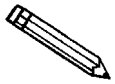
5. Clean the penetrometer as described at the end of this chapter.

High Pressure Differential Analysis

If you are using a blank correction file as your correction method, the correction file must exist before the sample data file can be completed (since you must select the blank correction file when you create the sample file).

Sometimes, however, it is not practical to perform the blank analysis in advance. To make it easier to create sample files when the correction file has not been created in advance, the AutoPore allows you to run a differential analysis of the sample and the corresponding blank. By running the sample and blank together, you can save the time of running first the blank and then the sample. The AutoPore software automatically associates the sample file with the blank correction file after the analysis is completed.

An important 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 gives you the most accurate low porosity sample data possible.



Since differential analyses require two high-pressure ports, they cannot be performed on a 9510 or a 9500.

To run a differential analysis:

1. Follow the instructions in Chapter 4 for creating a sample file for the sample. Also, create a sample file for the blank penetrometer. For each file, use *formula* for the correction method.

2. Run the low pressure analysis on both files.

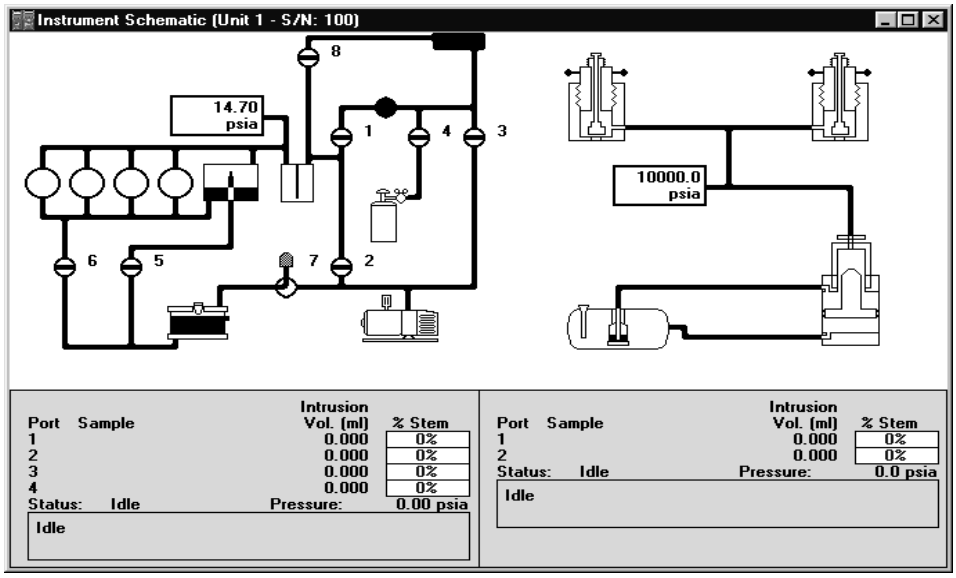
3. When you are ready to begin the high pressure analysis, click the **Differential, Blank in port 1** or **Differential, Blank in port 2** radio button in the High Pressure Analysis dialog. Then follow the instructions for performing a high pressure analysis. Refer to *Performing High Pressure Analyses* earlier in this chapter.

Displaying the Instrument Schematic

The instrument schematic provides a graphical representation of the instrument's low and high pressure systems. During automatic analyses, you can monitor progress by viewing the schematic. You can also use the schematic to operate the AutoPore manually. Manual operation is described in Chapter 6.

To display the instrument schematic, follow this procedure.

1. Select **Unit, Show instrument schematic** from the Main Menu. The schematic displays.

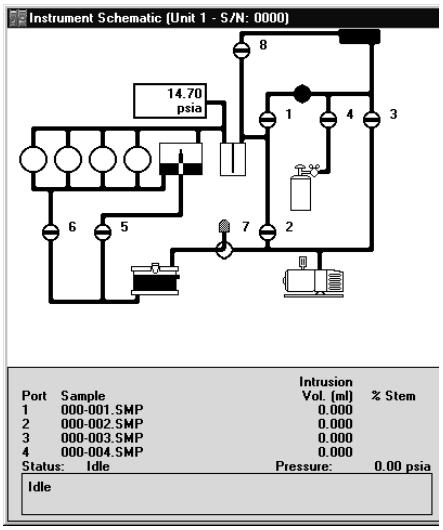


Low Pressure System High Pressure System

The left portion of the window shows the low pressure system (refer to *Low Pressure Schematic* later in this chapter). The right portion shows the high pressure system (refer to *High Pressure Schematic* later in this chapter).

2. To close the schematic window, click X in the upper right-hand corner of the title bar.

Low Pressure Schematic



The lower portion of the Instrument Schematic window shows the status of the system. Refer to *Monitoring a Low Pressure Analysis* in this chapter for a description of the status display.

The symbols in the low pressure schematic are described below.

State of gas and Mercury line valves:

 Open

 Closed

Mercury Degasser: The level of Mercury in the degasser is displayed. The 4 possible states are:

 Drained

 Partially Filled

 Filled

 Overfilled

The alarm state, *Overfilled*, is shown with a red background

.

Mercury Trap:

The state of the mercury trap is indicated on the status display by the following symbols:

 Empty

 Contains more than 6 mm of mercury

The alarm state is shown with a red background.

Mercury Reservoir:

The level of the mercury in the reservoir is shown using the following symbols:

 Low

 OK

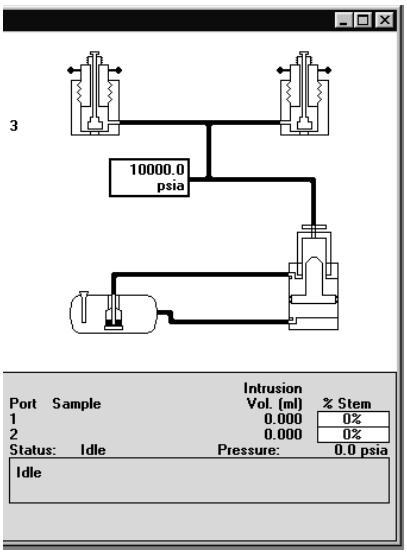
If the level is low, add mercury; instructions are found in Chapter 8.

Servo Valve:

 Open

 Closed

High Pressure Schematic



The lower portion of the Instrument Schematic window shows the status of the system. Refer to *Monitoring a High Pressure Analysis* in this chapter for a description of the status display.

The symbols in the high pressure schematic are described below.

Hydraulic Pump

The following symbol is shown for the pump:

When the pump is operating, the target pressure is displayed below this symbol.

Intensifier

The state of the intensifier limit switches is shown using the following symbols:

 Bottom

 Midway

 Top

Cleaning Penetrometers

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

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

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

Clean penetrometers as follows:

1. Dissolve Alconox® (or other suitable detergent) in water. Make sure 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.

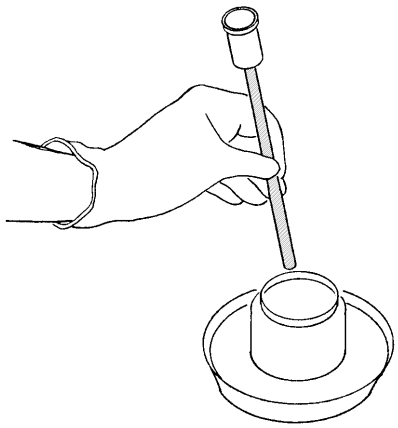


Figure 5-8. Cleaning Penetrometers

4. Remove the cap from the penetrometer. If the cap is difficult to remove, see the instructions for using the penetrometer wrench in the following section.

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 make sure that water runs from the bulb through the stem freely.

8. Rinse the penetrometer with isopropyl alcohol.

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.

Removing the Penetrometer Nut

If the penetrometer nut cannot be easily opened by hand, it may have become sealed too tightly during analysis. You may wish to use a penetrometer wrench to help you open it.

Do not tilt the penetrometer while removing the nut. Hold the penetrometer upright to avoid spilling mercury.

1. Place the penetrometer tool over the nut.
2. Position the wrench so the peg on the wrench fits into the notch on the nut.
3. While holding the penetrometer tool, turn the wrench counterclockwise to loosen and remove the nut.

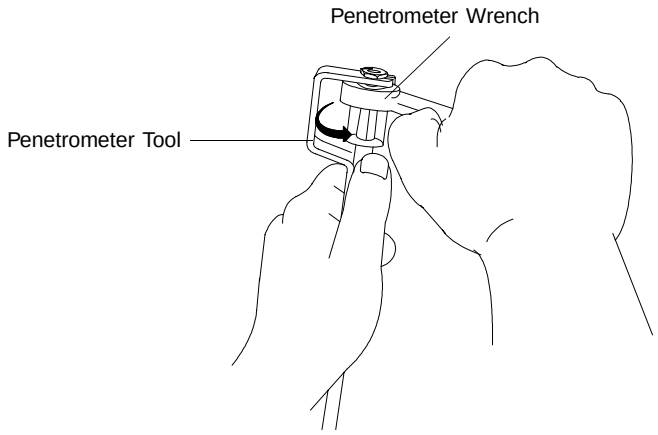
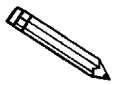


Figure 5-9. Using the Penetrometer Wrench

Suggested Sequence for Maximum Throughput (Leapfrogging Procedure)

Leapfrogging is a procedure for running low and high pressure systems concurrently to maximize throughput. The steps below describe leapfrogging a group of eight samples using the AutoPore 9505 or 9520 system which have four low- and two high-pressure chambers. You can also leapfrog samples on the AutoPore 9500 or 9510 which have two low- and one high-pressure chamber, substituting number of samples accordingly. You may leapfrog groups of samples continuously.



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

1. Prepare and weigh eight samples. Load eight penetrometers with samples; weigh them. Fill in the Sample Data Sheets and install samples 1 through 4 in the low pressure ports.
2. Create sample files for at least the first four samples. (You may wish to create sample files for all samples before beginning the leapfrogging process.)
3. Start the low pressure analysis. (Select **Unit**, **Low pressure analysis** from the Main Menu.)
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. Start the high pressure analysis. (Select **Unit**, **High pressure analysis** from the Main Menu.)
8. While analyses are taking place, you may continue creating sample files or preparing other samples for analysis.
9. When the high pressure run is complete, remove samples 1 and 2 from the high pressure chambers and replace with samples 3 and 4.
10. Start another high pressure run.
11. Automatic reports specified in each sample file will print when that sample's high pressure run ends.

12. 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; weigh them.

13. Install samples 5 and 6 in the high pressure chambers. Start another high pressure run.

14. When the third high pressure run is finished, install samples 7 and 8 in the high pressure chamber and start another high pressure run.

CHAPTER 6

MANUAL OPERATION AND CALIBRATION

- Before You Begin Manual Operation
- System Components
- Manual Control
- Calibration

MANUAL OPERATION AND CALIBRATION

Before You Begin Manual Operation

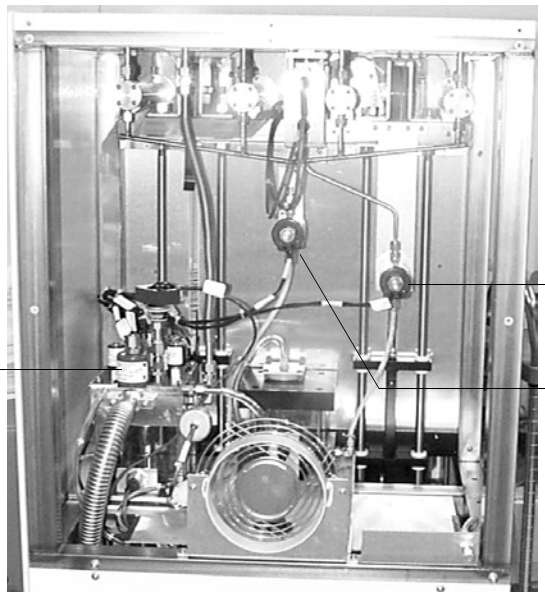
Carefully read the detailed description of the low and high pressure systems before attempting to operate the AutoPore manually. Specific instructions for manual operation follow the system description.

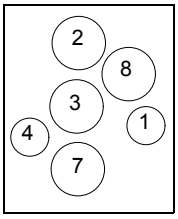
Do not actuate valves during an analysis; doing so could corrupt the data being collected.

During manual control of the system, be very careful not to create damaging or dangerous circumstances.

Do not attempt to operate the AutoPore manually without a thorough understanding of the low pressure and high pressure systems. Improper operation could cause mercury to back up and be discharged from the instrument. (See the Precautions section at the beginning of this manual.)

System Components





Top View of Valve Area

1 Servo Isolation Valve
2 Fast Evacuation Valve
3 Vacuum Valve
4 Gas Inlet Valve
7 Mercury Reservoir Evacuation Valve
8 Evacuation Reservoir Valve

Figure 6-1. System Components (Rear View)

Low Pressure Components

Servo Isolation Valve (1)

The servo isolation valve is used to ensure that 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)

When this valve is open, the sample stations are directly connected to the vacuum system. This valve is used at the end of the evacuation routine to achieve the best vacuum.

Vacuum Valve (3) and Evacuation Reservoir Valve (8)

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.

After 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)

This valve allows pressurization at the back of the servo valve. The servo can then be used to dose pressures from 0.2 psia to 50 psia onto the low pressure stations.

Dry nitrogen or argon is the preferred choice for the gas. Do not use air unless it is dried to remove water vapor.

Mercury Fill (5) and Drain (6) Valves

Mercury flows into the degasser and from there to the low pressure stations and is controlled by the **fill** valve. The **drain** valve allows mercury to flow back into the reservoir.

Mercury Reservoir Evacuation Valve (7)

The mercury reservoir evacuation valve connects the vacuum line to the mercury reservoir.

Low Pressure Ports

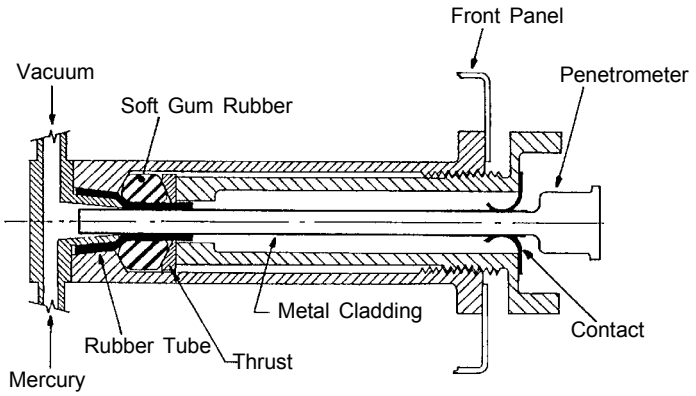


Figure 6-2. Low Pressure Port

A sample encased in a penetrometer is first evacuated, then filled with mercury, and, finally, pressurized to between 15 and 50 psia.

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.

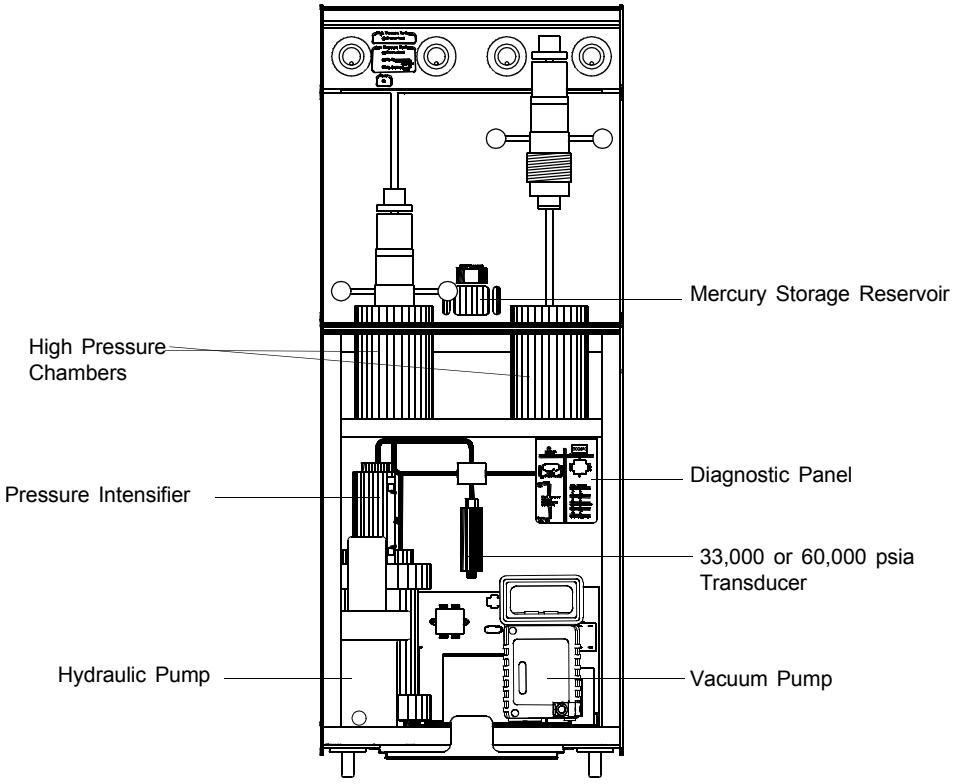


Figure 6-3. System Components (Front View)

Mercury Storage Reservoir

A mercury storage reservoir, which holds 7 to 10 pounds of mercury, 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 computer verifies that the level of mercury in the reservoir is adequate.

Mercury Degasser

On its way to the low pressure stations, mercury from the storage reservoir passes through an orifice from which it is sprayed into a well-evacuated space, called the mercury degasser, to remove trapped gases. From this space, it finally flows into the penetrometer.

When the level of mercury becomes high enough to fill the penetrometers, a sensor completes an electric circuit and flow is stopped by closure of the mercury fill solenoid valve. This sensor causes the HG UP indicator on the upper front panel to illuminate.

A second sensor closes the mercury drain valve when the level of mercury in the degasser is below the level in the low pressure stations. This sensor causes the DRAINED indicator on the upper front panel to illuminate.

A third sensor responds if the level of mercury rises above the normal filling level. The sensor causes both the mercury fill valve and the mercury drain valve to close and causes a buzzer alarm to sound. See **Chapter 8, Troubleshooting and Maintenance**, for information on how to recover from this condition.

Mercury Trap

The mercury trap serves as a large volume reservoir for mercury in the event that operator error or a malfunction allows mercury to travel toward the vacuum system. If not stopped, mercury would destroy the 50 psia transducer, render the vacuum gauge inoperative until cleaned, clog the restrictors in the vacuum valves, and contaminate the vacuum pump. The mercury trap is equipped with a sensor which, in turn, is connected to a high-pitched, continuous buzzer. If more than 6 mm (0.25 in.) of mercury accumulates in this trap, the buzzer will sound. You must correct the fault and remove the mercury before proceeding.

High Pressure Components

Hydraulic Pump

The basic high-pressure generating unit is a hydraulic pump. The pump is integral with a hydraulic fluid reservoir, and its output indicated by a pressure gauge. The maximum pressure of the pump is limited by a relief valve to a maximum of 2800 psia. Pump and drive motor rotation are reversible to allow you to raise or lower the pressure.

Pump speed and direction are controlled by the computer. This prevents overshooting of the desired pressure while minimizing time required to attain a target pressure.

Intensifier

The required 60,000 (33,000 for 9505 and 9500) psia is generated by a double-action, dual-piston intensifier connected to the hydraulic pump.

Two limit switches are located near the upper and lower limits of piston travel. The first of these switches alerts the operating program; the second interrupts the power to the motor.

Transducer

High pressure measurements are made with one pressure transducer. The signal from this transducer is processed by electronic circuitry to yield two pressure output signals. The ranges produced are 0-5000 psia and 0-60,000 psia for the 9520 or 9510; 0-3,000 psia and 0-33,000 psia for a 9505 or 9500.

High-Pressure Chambers

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 countersprung 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.

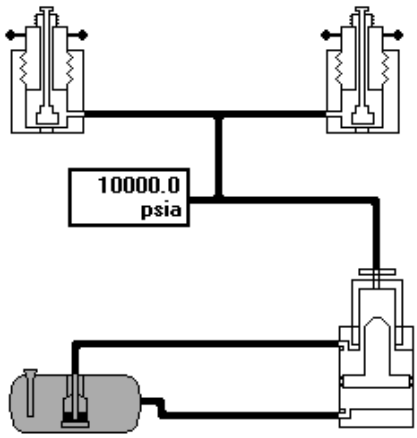
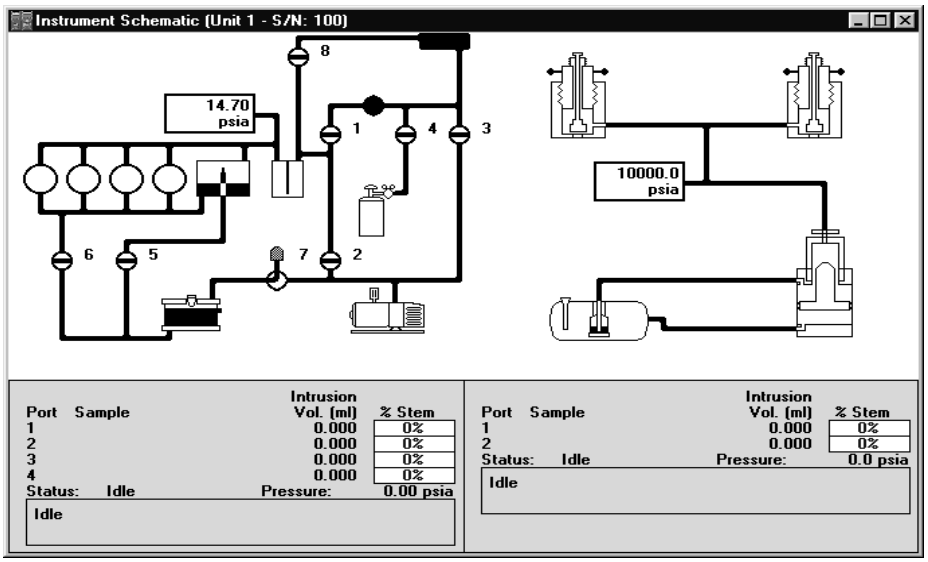


Figure 6-4. Schematic Arrangement of High Pressure System

Manual Control

To switch from automatic to manual control, select **Enable manual control** from the Unit menu. If the instrument schematic is not displayed, select **Show instrument schematic** from the Unit menu.



When you enable manual control, the symbols for valves and the servo pump change color on the screen to indicate manual operation.

- **Valves:** Closed = Yellow Open = Green
- **Servo Pump:** Off = Yellow On = Green

Use one of the following options to move among the system components depicted on the schematic:

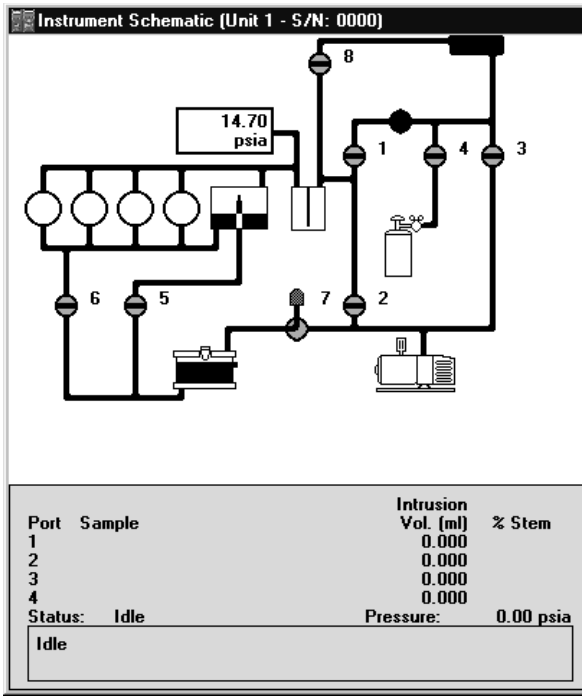
- **Mouse operation**: move the pointer to the desired component and click the left mouse button once.
- **Keyboard operation**: move to the desired component using the arrow keys or Tab.

A component is selected when it is surrounded by a dotted line. Each controllable component has a pop-up menu displaying the operations available for that particular component. These menus may be accessed by right-clicking on the desired component or by using the shortcut keys, Shift F9.

You can open and close valves and turn the servo pump on or off by using one of the following methods:

- access the pop-up menu and select the appropriate action
- double-click on the valve or the servo pump
- select the valve or servo pump symbol and press the spacebar

Manually Controlling a Low Pressure Analysis



Valve No.	Function
1	Servo isolation
2	Fast evacuation
3	Vacuum
4	Gas inlet
5	Mercury fill
6	Mercury drain
7	Mercury reservoir evacuation
8	Evacuation reservoir

Pressure: Displays the current pressure in the system.

- The pressure is shown in the units selected on the Data Presentation window of the Options menu or in μmHg, when appropriate.
- The reading shown is from either the vacuum gauge or the 50 psia transducer, depending upon which is currently in range.
- A simultaneous display of both readings can be viewed on the Transducer Calibration dialog (see *Calibrating Transducers* in this chapter).

Status:

Gives the system's current status. The following are displayed *for each port:*

- **Sample:** file name/sequence number
- **Intrusion volume**
- % of the penetrometer **stem** already used

Mercury Degasser:

The level of mercury in the degasser is displayed. The 4 possible states are:

 Drained

 Partially Filled

 Filled

 Overfilled

The alarm state, *Overfilled*, is shown with a red background.

Mercury Trap:

The state of the mercury trap is indicated on the status display by the following symbols:

 Empty

 Contains more than 6 mm of mercury

The alarm state is shown with a red background.

Mercury Reservoir:

The level of the mercury in the reservoir is shown using the following symbols:

 Low

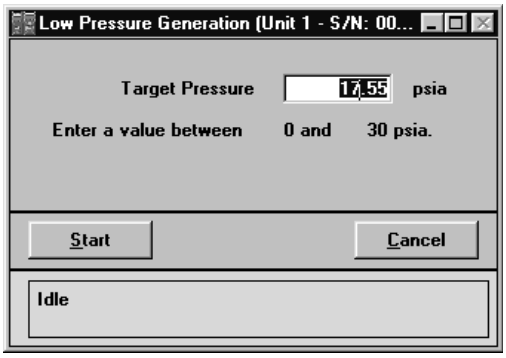
 OK

If the level is low, add mercury; instructions are found in **Maintaining Mercury Level** in Chapter 8.

Generating Low Pressure

You may generate pressure in the low pressure system when a low pressure analysis is not already in progress.

1. Select **Generate low pressure** from the Unit menu. The Low Pressure Generation dialog is displayed.



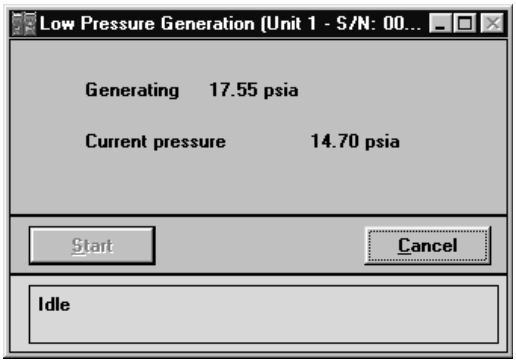
2 Enter the target pressure.

Range: 0.2 to 50.0 psia, threaded penetrometers
0.2 to 30.0 psia, spring closures

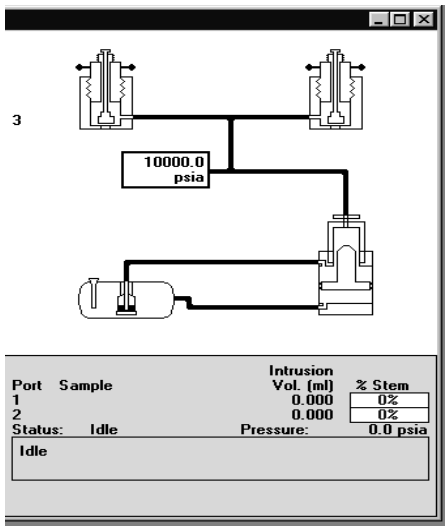
0.0034 to 0.3447 MPa, threaded penetrometers
0.0034 to 0.2068 MPa, spring closures

Threaded penetrometers are required for pressures above 30 psia or 0.2068 MPa. To use threaded penetrometers, select *Threaded penetrometer closures* from the Options menu.

3. Select Start. The current pressure is displayed.



Manually Controlling a High Pressure Analysis



The display features of the status control window may be useful during manual operation.

Pressure: Displays the current pressure in the system.

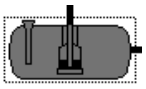
- The pressure is shown in the units selected on the **Data Presentation** window on the **Options** menu.

Status: Displays the system's current status.

The following are displayed *for each port:*

- **Sample:** file name/sequence #
- **Intrusion volume**
- % of the penetrometer **stem** already used

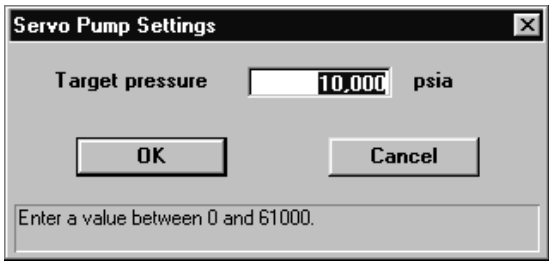
Servo Pump



The current state of the servo pump is shown using the following symbol:

Green = On
Yellow = Off

You may set the target pressure for the servo pump by double-clicking the pump symbol when the pump is disabled or by selecting **Set** from the pop-up menu. The Servo Pump Settings dialog is displayed.



Enter the target pressure, then click OK.

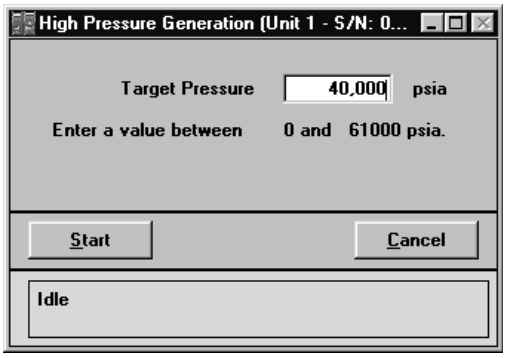
Range: 0 to 61,000 psia
0.0 to 420.58 MPa

The target pressure is displayed beneath the servo pump symbol.

Generating High Pressure

You may generate pressure in the high pressure system when a high pressure analysis is not already in progress.

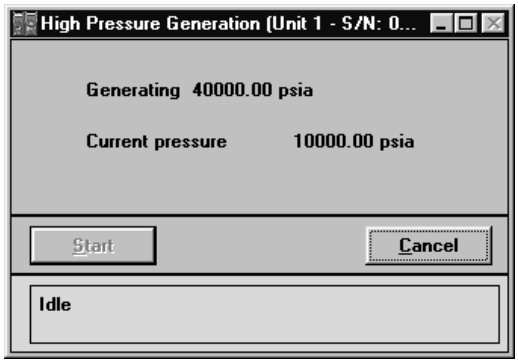
1. Select **Generate high pressure** from the Unit menu. The High Pressure Generation dialog is displayed.



2. Enter the target pressure.

   Range: 0 to 61,000 psia (AutoPores 9510 and 9520)
   0 to 33,000 psia (AutoPores 9500 and 9505)
   0.00 to 420.58 MPa (AutoPores 9510 and 9520)
   0 to 228.00 MPa (AutoPores 9500 and 9505)

3. Select **Start**. The current pressure is displayed.



Calibration

The Calibration screens may be used to observe the operation of the capacitance detectors and other signals. However, making adjustments to the calibration settings of the AutoPore should only be attempted by trained service personnel.

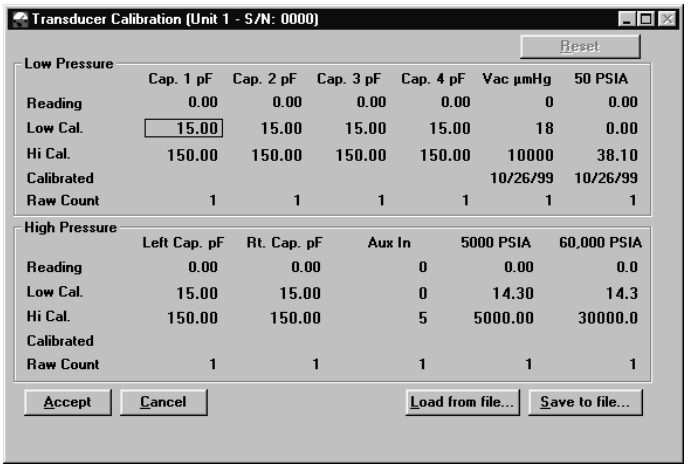
Calibrating Transducers

To calibrate the AutoPore, the capacitance detectors, vacuum gauge, reference gauge, transducer, etc. are read. All the necessary calibration data are displayed on the Transducer Calibration window.

To access the Transducer Calibration window, from the Main Menu, choose **Unit, Calibration, Transducers.**

- If an analysis is in progress, you may view calibration data, but you may not change the data. Data are updated at least once per second.
- The only button that is operational during an analysis is **Cancel** , which you may use to leave the Calibration window.
- You cannot *start* an analysis while the Transducer Calibration window is open.

Calibration data are stored as a database. You may save changes in calibration data to a file, then use those values later by reloading that file.



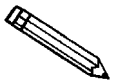
Accept

Saves all changes to the instrument's calibration database and closes the window. The AutoPore protects you from accidentally changing the calibration values by presenting a warning before saving changes:

Accept *(continued)*

Warning! Changing the calibration information will affect the performance of the instrument. Only qualified service personnel should do this. Do you wish to proceed?

Choose **Yes** to save changes to the calibration data. Choose **No** to abandon changes.



No permanent changes are made to the calibration data saved in the database until you select Accept and confirm its operation.

Cancel

Closes the Calibration window without saving any changes.

Save to file...

It is recommended that you save a backup copy of your calibration data; just click **Save to file...**, then designate a destination file. The suggested file name is the instrument serial number plus a **.CAL** extension.

If you make changes to your data, then attempt to exit the calibration window without saving them to a file, you are warned that keeping a backup copy is desirable. The AutoPore then gives you the opportunity to save to a file.

Load from

Allows you to replace current calibration data with stored calibration data. Select the correct file name, then confirm by clicking **OK**. You may also abandon the operation by clicking **Cancel**.

Windows automatically converts previous AutoPore data files. This allows existing parameter files to be used without conversion.

Reset:

Restores the calibration values that were present when you opened this window, discarding any changes.

The following fields and definitions refer to the low pressure system:

Reading (Cap 1-4, pF)
The current values from each of the four capacitance detectors in the low pressure stations. This field is for display only.

Low Cal. (Cap 1-4, pF)
For each of the four capacitance detectors, the capacitance of the reference device with the lower value.

Range: 5 - 50 pF

High Cal. (Cap 1-4, pF)
For each of the four capacitance detectors, the capacitance of the reference device with the higher value.

Range: 100 - 200 pF

Calibrated (Cap 1-4, pF)
The most recent date on which each of the four capacitance detectors was calibrated. Format is MM/DD/YY. This field is for display only and is updated automatically when calibration is performed on a capacitance detector channel.

Raw Count (Cap 1-4, pF)
The digital value produced by the AutoPore electronics for each of the four capacitance detectors. This field is for display only.

Reading (Vac μmHg)
The current value from the vacuum gauge. This field is for display only.

Low Cal. (Vac μmHg)
The pressure value read on a reference vacuum gauge after vacuum is achieved.

Range: 0 - 20 μmHg

High Cal. (Vac μmHg)
The pressure value read on a reference gauge when higher pressure is applied.

Range: 500 - 800,000 μmHg

Calibrated (Vac μmHg)
The most recent date on which the vacuum gauge was calibrated. Format is MM/DD/YY. This field is for display only and is updated automatically when calibration is performed on the vacuum gauge channel.

Raw Count (Vac μmHg)
The digital value produced by the AutoPore electronics for the vacuum gauge. This field is for display only.

Reading (50 PSIA)
The current pressure value from the 50-psia transducer. This field is for display only.

Low Cal. (50 PSIA)
The pressure value read from a reference transducer after vacuum is achieved.

Range: 0 - 1 psia

High Cal. (50 PSIA)
The pressure value read from a reference transducer at the upper end of the 50-psia range.

Range: 30 - 50 psia

Calibrated (50 PSIA)
The most recent date on which the 50-psia transducer was calibrated. Format is MM/DD/YY. This field is for display only and is updated automatically when calibration is performed on the 50-psia channel.

Raw Count (50 PSIA)
The value produced by the AutoPore electronics for the 50-psia transducer; for display only.

The following fields and definitions refer to the high pressure system:

Reading (Left/Rt. Cap, pF)
The current values from the left and right capacitance detectors on the high pressure system. This field is for display only.

Low Cal. (Left/Rt. Cap, pF)
For the left and right high capacitance detectors, the capacitance of the reference device with the lower value.

Range: 5 - 50 pF

High Cal.
(Left/Rt. Cap, pF)

For the left and right high capacitance detectors, the capacitance of the reference device with the higher value.

Range: 100 - 200 pF

Calibrated
(Left/Rt. Cap, pF)

The most recent dates on which the left and right capacitance detectors were calibrated. Format is MM/D/YY. This field is for display only and is updated automatically when calibration is performed on the left or right capacitance detector channel.

Raw Count
(Left/Rt. Cap, pF)

The digital value produced by the AutoPore electronics for the left and right capacitance detectors. This field is for display only.

Reading
(Aux In)

The current reading for the auxilliary input channel. This field is for display only.

Low Cal.
(Aux In)

The value measured on a reference voltmeter when the low end of the auxilliary channel was calibrated.

High Cal.
(Aux In)

The value measured on a reference voltmeter when the high end of the auxilliary channel was calibrated.

Raw Count
(Aux In)

The value produced by the AutoPore electronics for the auxilliary input channel. This field is for display only.

The remaining fields and the information displayed are dependent on the type of high pressure system you have:

9520 or 9510

Reading (5000 PSIA)
The current pressure value from the 5000-psia channel of the 60,000-psia transducer. This field is for display only.

Low Cal. (5000 PSIA)
The pressure value read from a reference gauge at a convenient low pressure, such as atmospheric pressure.

Range: 0 - 100 psia

High Cal. (5000 PSIA)
The pressure value read from a reference gauge in the 4000-to-5000 psia range.

Calibrated (5000 PSIA)
The most recent date on which the 5000-psia channel of the 60,000-psia transducer was calibrated. Format is MM/DD/YY. This field is for display only and is updated automatically when calibration is performed on the 5000-psia channel.

Raw Count (5000 PSIA)
The digital value produced by the AutoPore electronics for the 5000-psia channel of the 60,000-psia transducer. This field is for display only.

Reading (60,000 PSIA)
The current pressure value from the 60,000-psia channel of the 60,000-psia transducer. This field is for display only.

Low Cal. (60,000 PSIA)
The pressure value read from a reference gauge in the 0-to-5000 psia range, such as at atmospheric pressure.

High Cal. (60,000 PSIA)
The pressure read from a reference gauge in the 28,000-to-60,000 psia range.

Calibrated (60,000 PSIA)

The most recent date on which the 60,000-psia channel of the 60,000-psia transducer was calibrated. Format is MM/DD/YY. This field is for display only and is updated automatically when calibration is performed on the 60,000-psia channel.

Raw Count (60,000 PSIA)

The digital value produced by the AutoPore electronics for the 60,000-psia channel of the 60,000-psia transducer. This field is for display only.

9505 or 9500

Reading (3000 PSIA)

The current pressure value from the 3000-psia channel of the 33,000-psia transducer. This field is for display only.

Low Cal. (3000 PSIA)

The pressure value read from a reference gauge at a convenient low pressure, such as atmospheric pressure.

Range: 0 - 100 psia

High Cal. (3000 PSIA)

The pressure value read from a reference gauge in the 2000-to-3000 psia range.

Calibrated (3000 PSIA)

The most recent date on which the 3000-psia channel of the 33,000-psia transducer was calibrated. Format is MM/DD/YY. This field is for display only and is updated automatically when calibration is performed on the 3000-psia channel.

Raw Count (3000 PSIA)

The digital value produced by the AutoPore electronics for the 3000-psia channel of the 33,000-psia transducer. This field is for display only.

Reading (33,000 PSIA)

The current pressure value from the 33,000-psia channel of the 33,000-psia transducer. This field is for display only.

Low Cal. (33,000 PSIA)

The pressure value read from a reference gauge in the 0- to 3000-psia range, such as at atmospheric pressure.

High Cal. (33,000 PSIA)

The pressure read from a reference gauge in the 28,000- to 33,000-psia range.

Calibrated (33,000 PSIA)

The most recent date on which the 33,000-psia channel of the 33,000-psia transducer was calibrated. Format is MM/DD/YY. This field is for display only and is updated automatically when calibration is performed on the 33,000-psia channel.

Raw Count (33,000 PSIA)

The digital value produced by the AutoPore electronics for the 33,000-psia channel of the 33,000-psia transducer. This field is for display only.

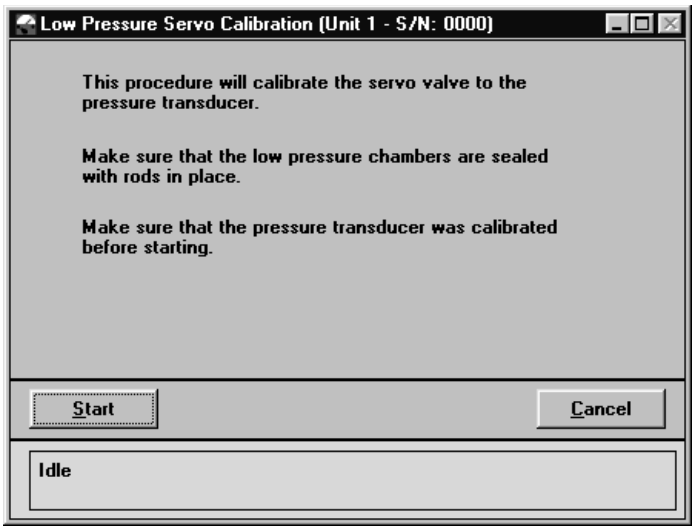
Calibrating the Low Pressure Servo Valve

In order to calibrate the low-pressure valve, you must first calibrate the low-pressure transducer. Refer to *Calibrating Transducers* in this chapter.

Perform the following steps to calibrate the low pressure servo valve to the 50 psi pressure transducer.

1. Make sure the low pressure chambers are sealed with rods in place.

2. From the Main Menu, select **Unit, Calibration**, **Low pressure servo** to display the Low Pressure Servo Calibration window.



3. Select **Start** to begin the calibration. The status portion of the window shows the progress of the calibration process.

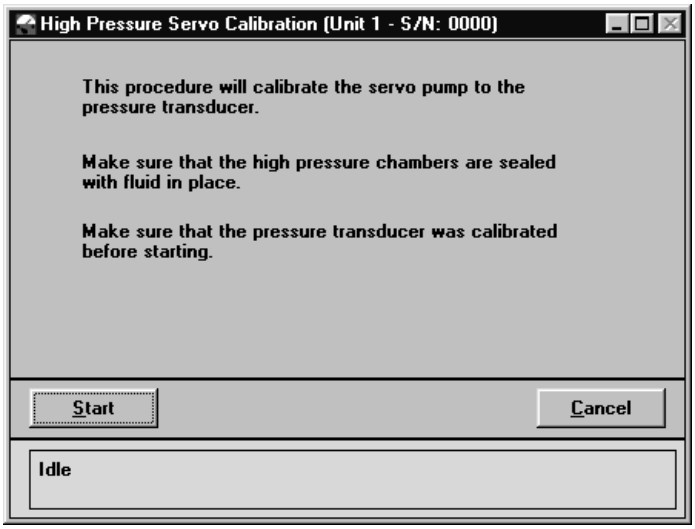
Calibrating the High Pressure Servo Pump

In order to calibrate the high pressure servo pump, you must first calibrate the high pressure transducer. Refer to *Calibrating Transducers* in this chapter.

Perform the following steps to calibrate the high pressure servo pump to the 60,000 psi pressure transducer.

1. Make sure the high pressure chambers are sealed with fluid in place.

2. From the Main Menu, select **Unit, Calibration**, **High pressure servo** to display the High Pressure Servo Calibration window.



3. Select **Start** to begin the calibration. The status portion of the window shows the progress of the calibration process.

CHAPTER 7

GENERATING REPORTS

- Automatically Generated Reports
- Starting Reports
- Selecting SPC Options
- Generating a Regression Report
- Generating a Control Chart
- Generating a PSD History Report
- Printed Reports
- Sample Reports

GENERATING REPORTS

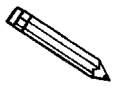
This chapter contains instructions for:

- starting reports
- selecting SPC report options
- generating regression reports, control charts, and PSD history reports

Also contained in this chapter are samples of a few of the reports available using the AutoPore program. Instructions for creating and editing report options files are found in *Creating Report Options File*s in Chapter 4.

Automatically Generated Reports

The reports that you specified in the sample information file are printed automatically at the end of the analysis if specified in the analysis wizard.



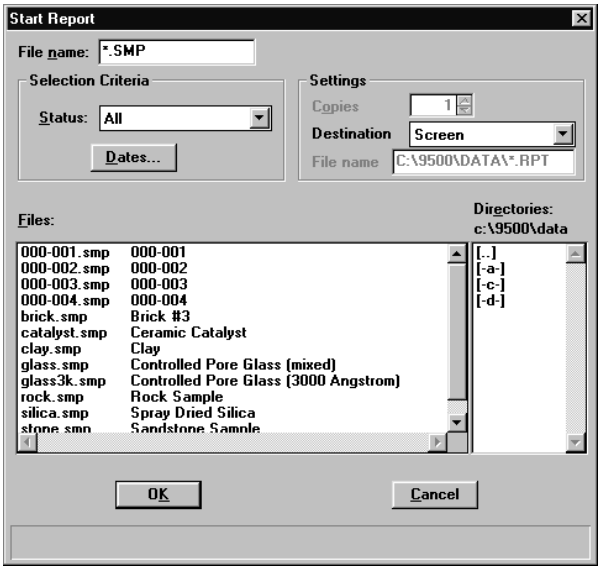
Windows makes it easy for applications to share data. Simply generate your reports to a file, then use Explorer to copy or move the file wherever it's needed. Or, you can use a communications program to transfer the file over a modem or serial line.

Starting Reports

After reviewing the automatically generated reports for a sample, you may wish to print additional reports, or change certain parameters before printing new reports. For example, you may wish to select a different group of samples to overlay, use a different Y-axis variable, or change the range of an axis.

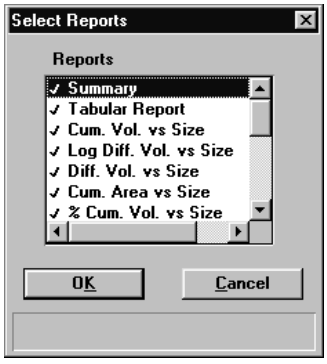
If you simply wish to reprint the reports contained in the sample information file:

1. From the Main Menu, select **Reports**, **Start Report**. The Start Report File selection window is displayed.



2. Select a sample file from the Files list.

3. Indicate the report destination (screen, printer or file); click OK . The Select Reports window opens.



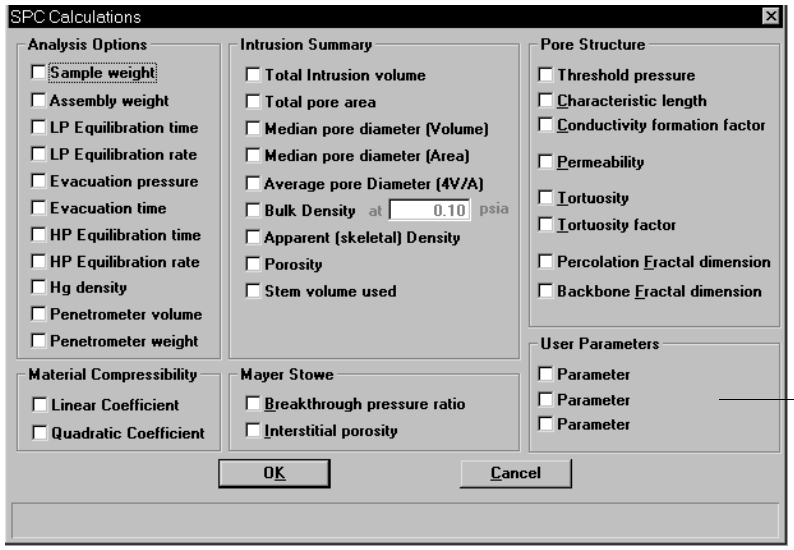
4. From the reports list, choose the report(s) to be generated. Double-click to place a check mark next to the reports you wish to include (double-click again if you wish to exclude a report that is checked). Available reports are those specified in the report options file of the sample you have selected.

5. Click OK.

Selecting SPC Options

SPC Report Options enable you to select the data that are to be reported in Regression and Control Chart reports. For efficiency, it is best to select only the variables you actually intend to use. All variables selected must be computed for each sample file used in an SPC report. These archived values are used in all future reports unless you have selected **Recalculate archived SPC results** on the individual report dialog.

1. From the Reports menu, select **Reports**, **SPC report options**. The SPC Calculations dialog is displayed.



These parameters are specified by selecting Sample defaults from the Options menu.

2. Select each variable you want to include, then click OK.

   If you check **Bulk density**, enter the pressure for the measurement.

   Range: 0.10 to 61000.00 psia
   0.007 to 420.5801 MPa

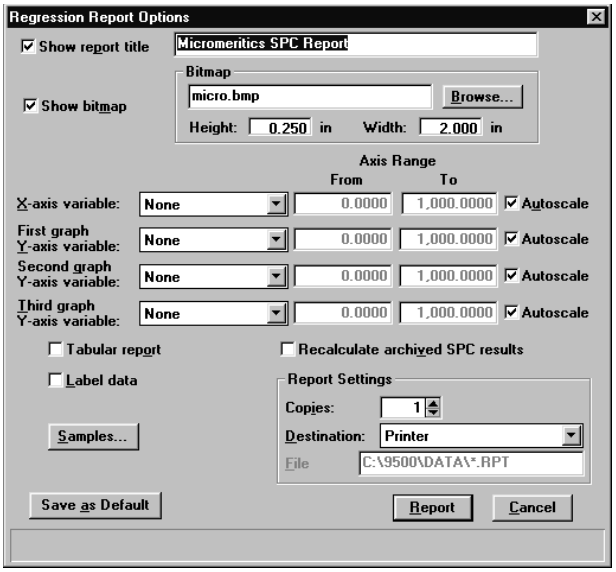
   If the pressure you enter is below the filling pressure, the filling pressure will be used on the report.

3. Click OK to save the selections and close the dialog.

Generating a Regression Report

The Regression Report is used to determine the interdependency between two variables. You may plot the regression of up to three Y-axis (dependent) variables against the X-axis (independent) variable. The degree of correlation between the variables also is reported. The graphs for the regression report are scaled so that all three fit on a single page. If you choose less than three, the graphs are scaled to fill most of the page.

1. From the Main Menu, select **Reports**, **Regression report**. The Regression Report Options dialog is displayed.



2. Enter data in this dialog as described below.

Show report title — Select to have a title display on your report. Accept the default or enter a new title.

Range: 40 characters

Show bitmap — Select to have a graphic (bitmap format) display above the report title. For example, you may wish to display your company logo. Click **Browse** to choose the bitmap, then enter the height and width in the appropriate fields.

X- and Y-Axes Variable fields

Click on the down arrow to choose X- and Y-axes variables. The variables in this list are the ones you specified in **SPC report options**.

With this option, you may plot the regression of up to three Y-axis variables against the X-axis variable. The X-axis specifies the independent variable for the regression, while the Y-axes provide the dependent variables.

Axis Range

Specify the beginning and ending values for the X- and Y-axis ranges. Data collected outside these ranges are not included in the plot. These fields are disabled if you choose *Autoscale*.

Autoscale

Choose this option to have the X- and/or Y-axes scaled automatically. When scaled automatically, both axes begin at zero. The analysis program uses the highest values collected during analysis as the ending points.

Tabular report

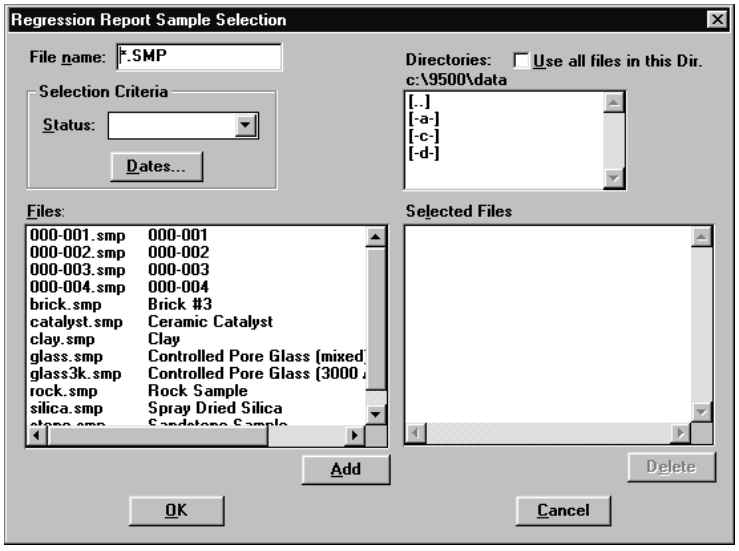
Select to have tabular, as well as graphical, data generated. A tabular report contains the numeric values contributed by each sample.

Label data

Select to have the points on the plot labeled to correspond with the values in the sample files.

Samples

Select to choose the sample files you wish to have reported; the Regression Report Sample Selection dialog is displayed.



Samples *(continued)*

Select **Use all files in this Dir.** to include all files from the selected directory in the Regression report.

Select the file and click **Add** to move a file from the **Files** list to the **Selected Files** list. Alternatively, you can simply double-click on the desired file(s). You can select multiple files by holding down **Ctrl** while making your selections.

You may choose up to 200 sample files.

Use **Delete** to delete a selected file from the **Selected Files** list and move it back to the **Files** list.

Use the **Status** drop-down list and/or **Dates** to limit the files displayed in the **Files** list.

Save as default

Saves the current definition of the report as the default.

Recalculate archived SPC results

Select this option to have archived SPC values recalculated. This ensures that any changes made to the SPC Report Options are included in the new report; however, it will lengthen the time required to generate the report.

Report Settings

Copies

Enabled when the *Printer* or *Printer/Plotter* destination is chosen. This option allows you to print up to four copies of the selected reports.

Destination

Choose a destination for report output.

Choices: File, Printer, Screen, Printer/Plotter

If you choose *File*, enter a name in the **File name** field. Remember, only tabular data can be generated to a file.

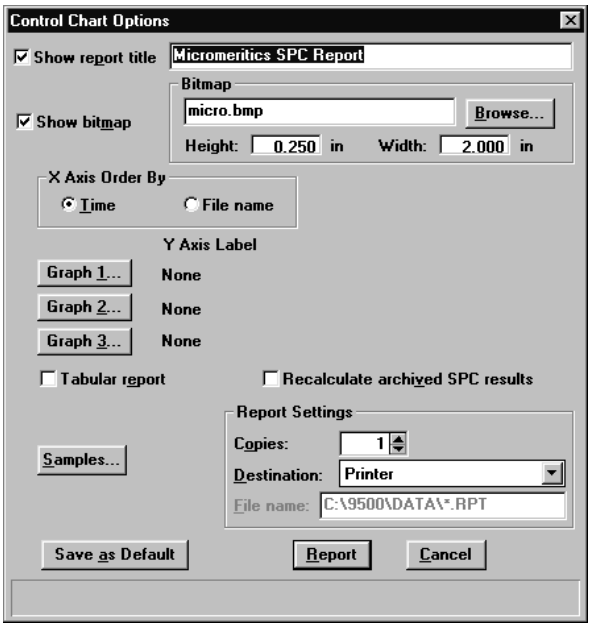
Report

Select to generate the report.

Generating a Control Chart

A control chart report plots the changes in a statistic.

1. From the Main Menu, select **Reports**, **Control chart**. The Control Chart Options dialog is displayed.



2. Enter data in this dialog as described below.

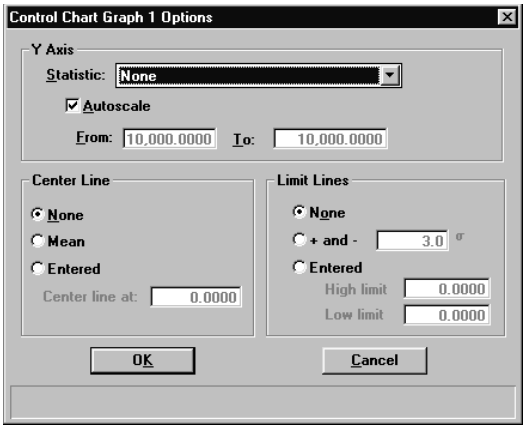
Show report title Select to have a title display on your report. Accept the default or enter a new title.

Range: 40 characters

Show bitmap Select to have a graphic (bitmap format) display above the report title. For example, you may wish to display your company logo. Click Browse to choose the bitmap, then enter the height and width in the appropriate fields.

X-axis Order By Choose whether you wish to have statistics listed by file name or analysis time.

Graph [n] Click to define the Y-axis of the respective graph. The Control Chart Graph [n] Options dialog is displayed.



Statistic Choose a variable; this variable will be plotted against time or file name. This drop-down list displays the SPC variables selected on the SPC Report Options dialog.

Autoscale Select to have the Y-axis scaled automatically. If you wish to specify a range, deselect this option and enter ranges in the **From** and **To** fields.

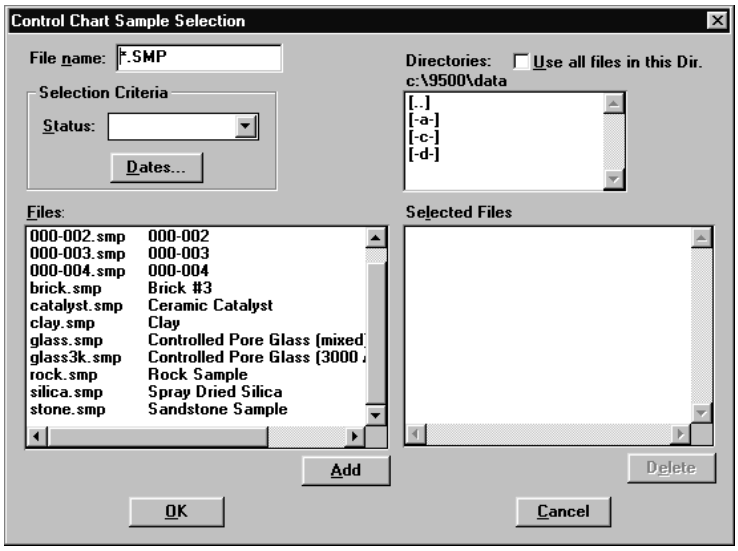
Center Line Choose placement of a line for the variable's optional value (if desired). If you choose *Entered*, specify a value in the **Center line** field.

Limit Lines Choose placement for limiting lines (if desired). You can have the lines placed at some multiple of the standard deviation (σ) or at specified positions (*Entered*).

Tabular report Select to have tabular, as well as graphical, data generated. A tabular report contains the numeric values contributed by each sample.

Recalculate archived SPC results Select this option to have archived SPC values recomputed. This ensures that any changes made to the SPC Report Options are included in the new report. It also lengthens the time required to generate the report.

Samples Select to choose the sample files you wish to have reported. The Control Chart Sample Selection dialog is displayed.



This dialog functions in the same manner as the Regression Report Sample Selection dialog explained previously.

Save as default Saves the current definition of the report as the default.

Report Settings

Copies Enabled when the *Printer* or *Printer/Plotter* destination is chosen. This option allows you to print up to four copies of the selected reports.

Destination Choose a destination for report output.

Choices: File, Printer, Screen, Printer/Plotter

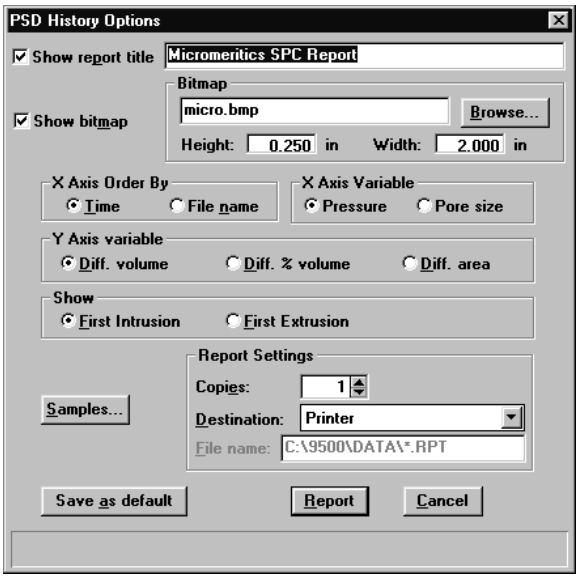
If you choose *File*, enter a name in the **File name** field. Remember, only tabular data can be generated to a file.

Report Click to generate the report.

Generating a PSD History Report

The PSD History Report enables you to generate a sequence of full pore size distribution graphs.

1. From the main menu, select Reports, PSD history. The PSD History Options dialog is displayed.



2. Enter data in this dialog as described below.

Show report title — Select to have a title display on your report. Accept the default or enter a new title.

Range: 40 characters

Show bitmap — Select to have a graphic (bitmap format) display above the report title. For example, you may wish to display your company logo. Click Browse to choose the bitmap, then enter the height and width in the appropriate fields.

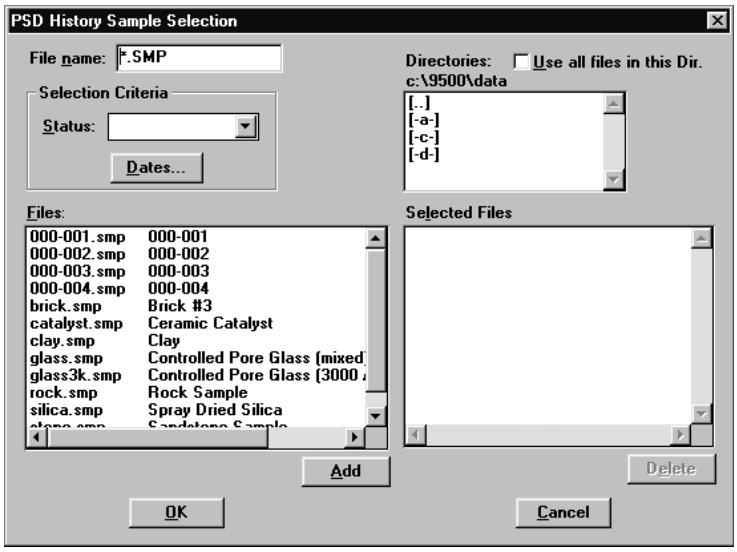
X-axis Order By — Choose whether you wish to have statistics listed by file name or analysis time.

X Axis Variable — Choose whether you wish to have statistics listed by pressure or pore size.

Y Axis Variable — Choose whether you wish to have statistics listed by differential volume, differential % volume, or differential area.

Show Choose whether to show first intrusion or first extrusion.

Samples Select to choose the sample files you wish to have reported. The PSD History Sample Selection dialog is displayed.



This dialog functions in the same manner as the Regression Report Sample Selection dialog explained previously.

Save as default Saves the current definition of the report as the default.

Report Settings

Copies Enabled when the *Printer* or *Printer/Plotter* destination is chosen. This option allows you to print up to four copies of the selected reports.

Destination Choose a destination for report output.

Choices: File, Printer, Screen, Printer/Plotter

If you choose *File*, enter a name in the **File name** field. Remember, only tabular data can be generated to a file.

Report Click to generate the report.

Printed Reports

Header

All printed reports (either to the screen or to a printer) contain a header displaying file statistics.

- Tabular and graphical reports contain sample and instrument statistics such as date and time of analysis, analysis conditions, and so forth.

  The headers for these reports also contain notes of any changes to the sample file which occur after analysis.

- Summary report headers contain the same type of information displayed in tabular and graphical reports with the exception of notes.

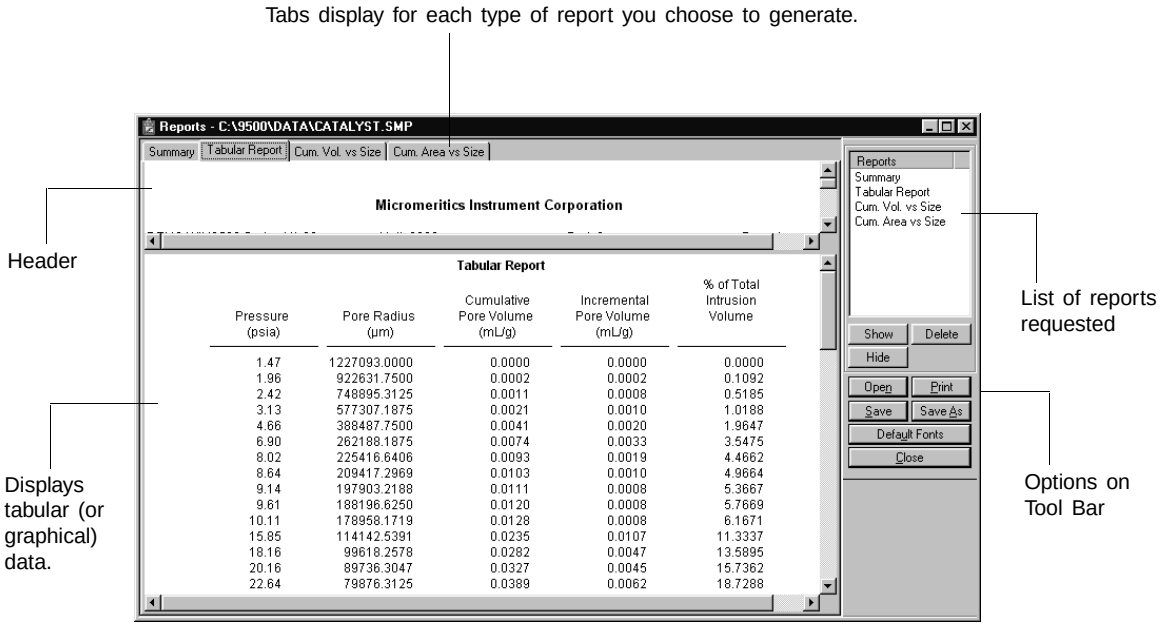
- SPC report headers display the current date as well as the range of dates of the samples you select to have reported.

Onscreen Reports

The report window containing onscreen reports provides many options for customizing and manipulating reports:

- a tool bar
- pop-up menu options
- zoom feature
- axis cross hairs

When reports are printed to the screen, they are printed in a window like the one shown below. Each requested report is listed in the Reports window on the tool bar; they are also indicated by selectable tabs across the top of the report header. To view a specific report, select its tab or select the report in the Reports window and click Show . Horizontal and/or vertical scroll bars are provided on windows containing more information than will fit in the window.



Tool Bar

Reports — Contains a list of all requested reports.

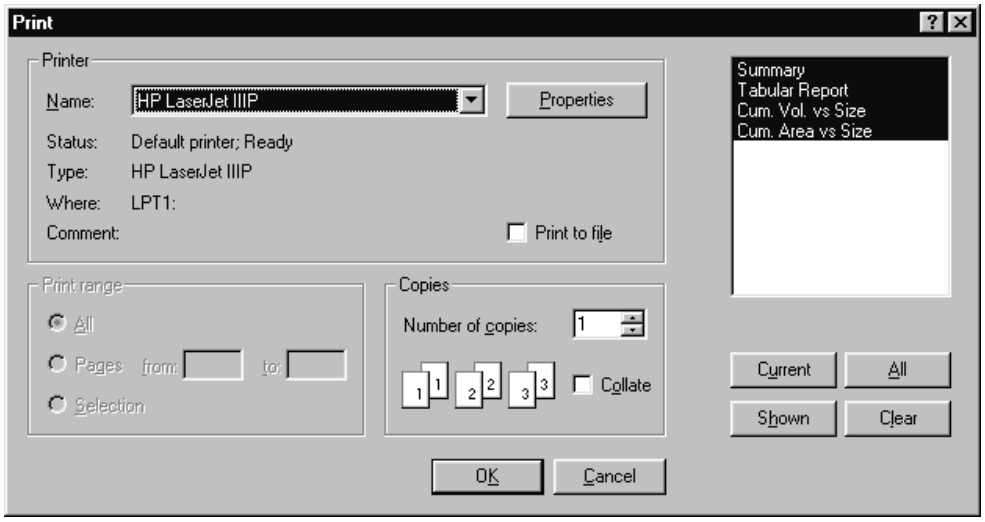
Show — Shows the selected report in the report window. If the report has been hidden, it and its associated tab will become visible.

Delete — Deletes the selected report. A deletion confirmation dialog is displayed since this function cannot be undone. The deleted report(s) will have to be regenerated if deleted in error.

Hide — Hides (removes) the selected report from the report window. The report's associated tab is also removed.

Open — Allows you to open a previously saved report file.

Print Displays a print dialog so that you can choose an appropriate printer for report output. A list of available reports is displayed in the window on the right side of the dialog.



For convenience in selecting reports to print, push buttons are provided beneath the report window. Or, you can make your selection by clicking on the desired reports.

Current selects the report displayed in the report window.

Shown selects only the shown reports; any unlighted reports indicate they are hidden. You can still select hidden reports from this window to print.

All selects all reports, including those that may have been hidden.

Clear clears all selections.

Save Saves all reports of the currently open file in a report format using the same name as the sample file, only with an **rep** extension. If you wish to specify a name and/or specific reports to save, use **Save as** .

Save as

Allows you to save all or specified reports from the currently open file. The push buttons displayed on this dialog perform in the same manner as the print dialog (explained above).

Reports can be saved in three different formats:

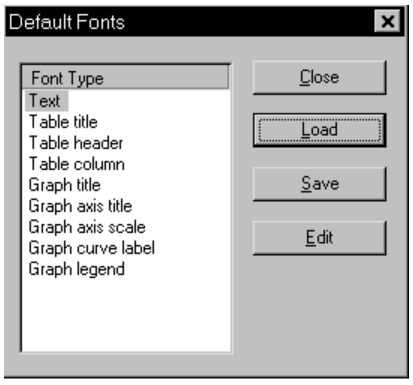
Report system (.rep)*: Saved in a format which allows you to reopen the file using **Open** on the Report window tool bar.

Spreadsheet (.xls)*: Saved in a format which can be imported into most spreadsheet programs.

Ascii Text (.txt)*: Saved in ASCII text which can be imported into programs accepting this type of file.

Default Fonts

Displays the Default Fonts dialog so that you can edit report fonts.



The window on the left lists the items for which the font can be edited.

Click **Edit** to edit the font of a selected item; a font dialog is displayed so that you can specify the desired font and attributes.

Click **Save** to save the changes as the default. If you do not click **Save** , the changes will apply to the current report set only. The next reports will revert to the default font.

Click **Load** to load the default font of a selected item.

Click **Close** to close the dialog.

Pop-up Menus

Pop-up menus are accessed when you right-click on the tabular or graphical portion of a report.

For Tabular Reports

Resize column — Displays a dialog so that you can specify the width of the selected column (in inches).

Rename column — Displays a dialog so that you can edit the name of the selected column. Use Ctrl Enter to insert line feeds.

Move column — Allows you to move the location of the selected column to the left or to the right.

Align column — Enables you to right-align, left-align, or center the data in the selected column.

Hide column — Displays a list of all columns, enabling you to select the one you wish to hide.

Show column — Displays a list of all hidden columns, enabling you to select the one you wish to have shown again.

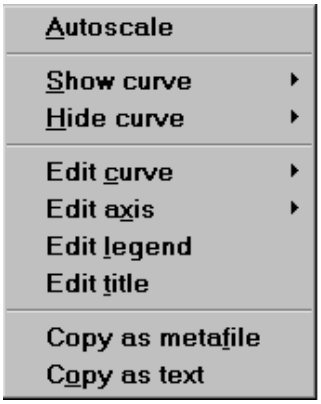
Column font — Displays a Font dialog, allowing you to change font attributes for the tabular data in the current report.

Header font Displays a Font dialog, allowing you to change font attributes for column headers in the current report.

Edit title Allows you to edit the table title and font.

Copy table as text Enables you to copy the entire table (column headers and data) and then insert it into another program. Colums are tab-delimited, allowing easy alignment.

For Graphs

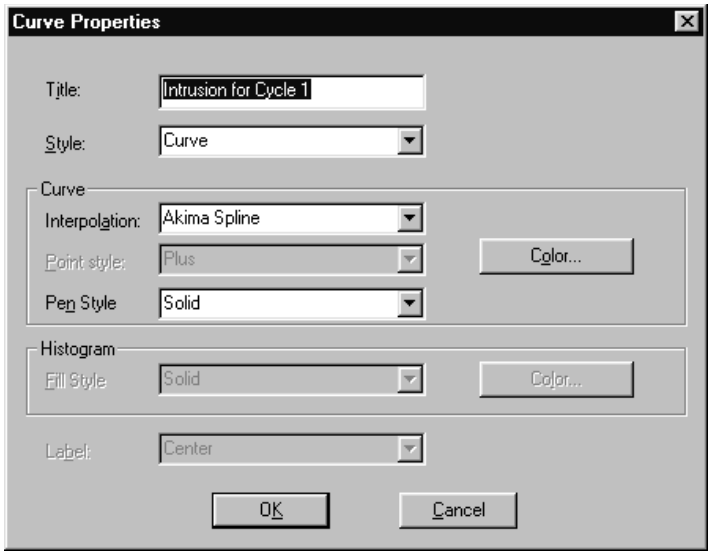


Autoscale Autoscales all axes of the graph. This function is useful for returning to a full view after having zoomed in.

Show curve Allows you to show curves that have been hidden. This option is disabled (greyed) if no curves have been hidden.

Hide curve Allows you to hide (remove) unwanted curves.

Edit curve Displays the Curve Properties dialog, allowing you to edit curve properties.



Title Displays the title of the curve you are editing.

Style Drop-down list containing styles in which collected data can be displayed.

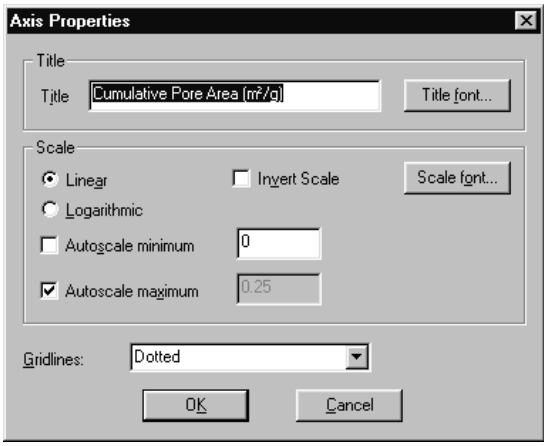
Choices: Curve, Histogram, Points, Curve and Points

Curve group box Contains options for curves and points. You can edit the curve interpolation, the style of curve and/or points, as well as the pen color. The options in this group box are disabled if Histogram is chosen in the **Style** drop-down list.

Histogram group box Allows you to specify the type of fill as well as the color if Histogram is chosen as the style for collected data.

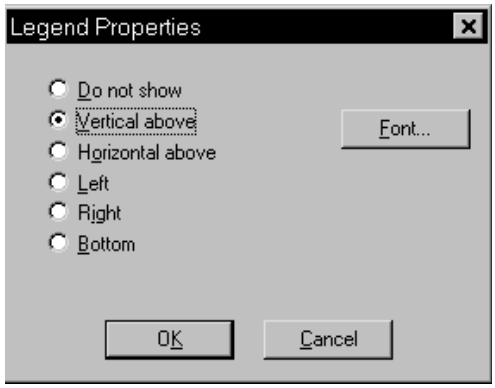
Edit axis

Displays the Axis Properties dialog, allowing you to edit axis properties.



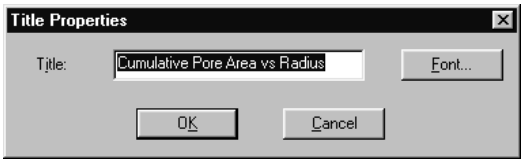
Edit legend

Displays the Legend Properties dialog, allowing you to edit the placement of the legend.



Edit title

Displays the Title Properties dialog, allowing you to edit the current graph's title and font.



Copy as metafile

Copies the graph and places it on the clipboard, allowing you to paste it into other applications accepting Windows metafiles.

Copy as text

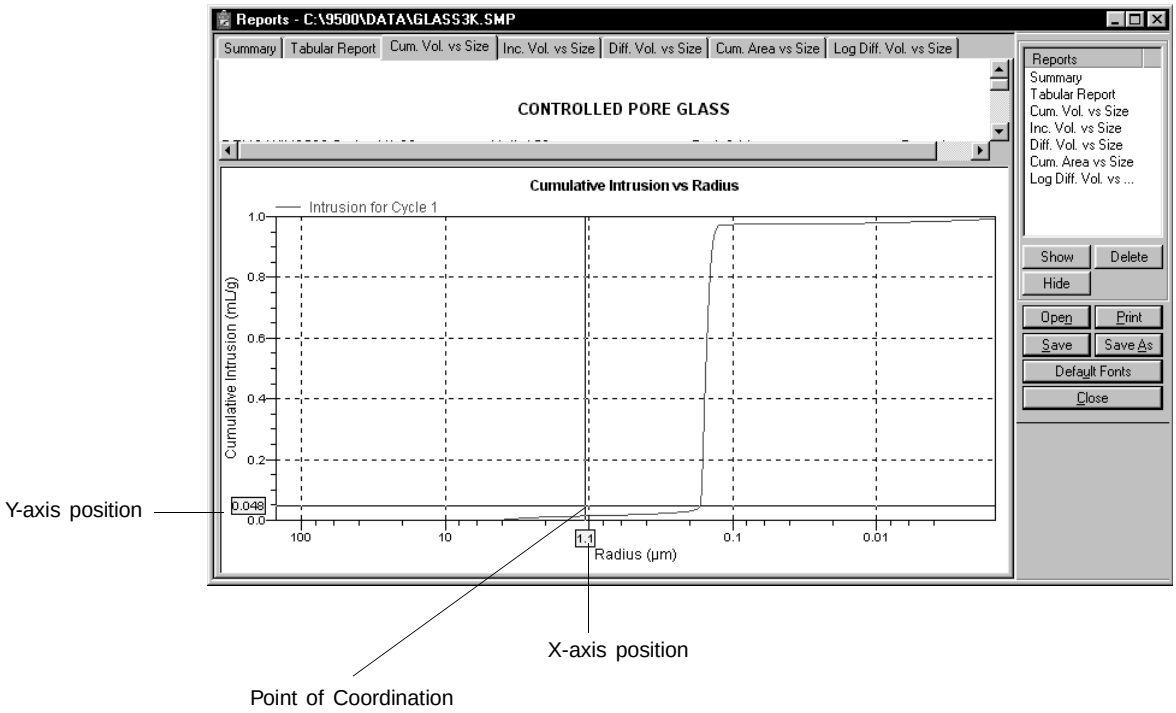
Copies the data used to generate the graph as a series of tab-delimited columns of text.

Zoom Feature

A zoom feature is included with the report system so that you can zoom in to examine fine detail of the distribution. To use this feature, simply hold down the left mouse button and drag the mouse cursor (drawing a box) across the area you wish to view; then release the button. The enlarged area immediately fills the graph area. You can return to the normal view by right-clicking on the graph and selecting **Autoscale**.

Axis Cross Hair

A cross-hair function is available so that you can view axis coordinates. To use this feature, simply left-click in the desired area of the graph.



To remove the cross-hair, click outside the graph area or right-click to display the pop-up menu, then select **Autoscale**.

Sample Reports

This section contains only a small sampling of the reports available with the AutoPore Program.

Graphs

The AutoPore program provides great flexibility for generaing graphs. You can choose to plot the x-axis as pressure, pore size, or particle size. A wide selection of variables is also available for the y-axis. Refer to **Editing Graphs** in Chapter 4 for detailed information on editing graphs.

Cumulative Intrusion vs. Pressure

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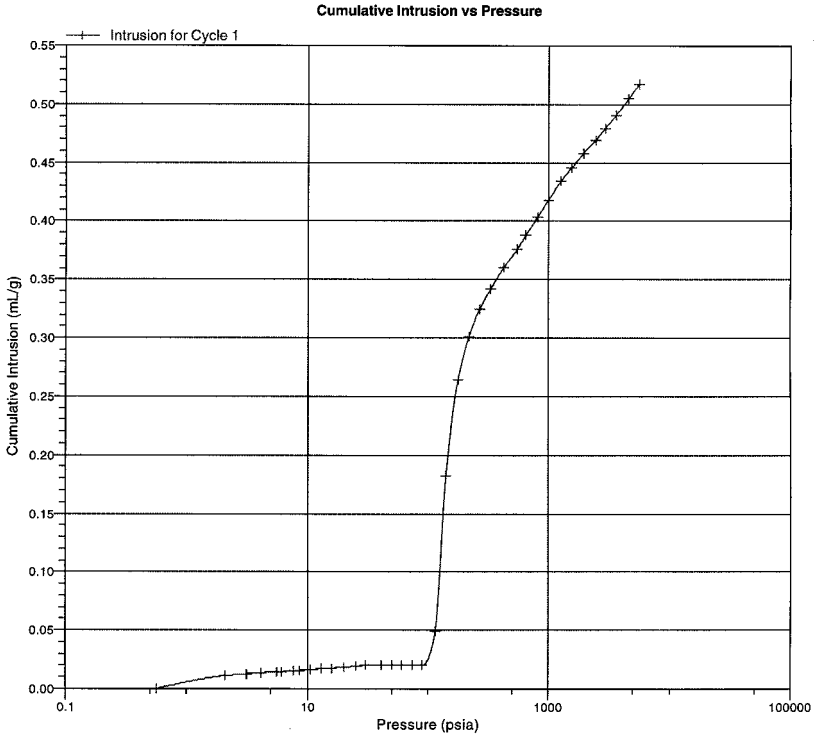
AutoPore IV 9500 V X.XX Serial: 613 Port: 4/2 Page 2

Sample ID: Alumina
Operator: AWT/JK
Submitter: MICROMERITICS
File: A:\6568.SMP

LP Analysis Time:	7/6/1999 5:05:26PM	Sample Weight:	0.5545 g
HP Analysis Time:	7/7/1999 8:51:04AM	Correction Type:	None
Report Time:	9/7/2000 5:32:25PM	Show Neg. Int:	No



Log Differential Intrusion vs. Pore Size

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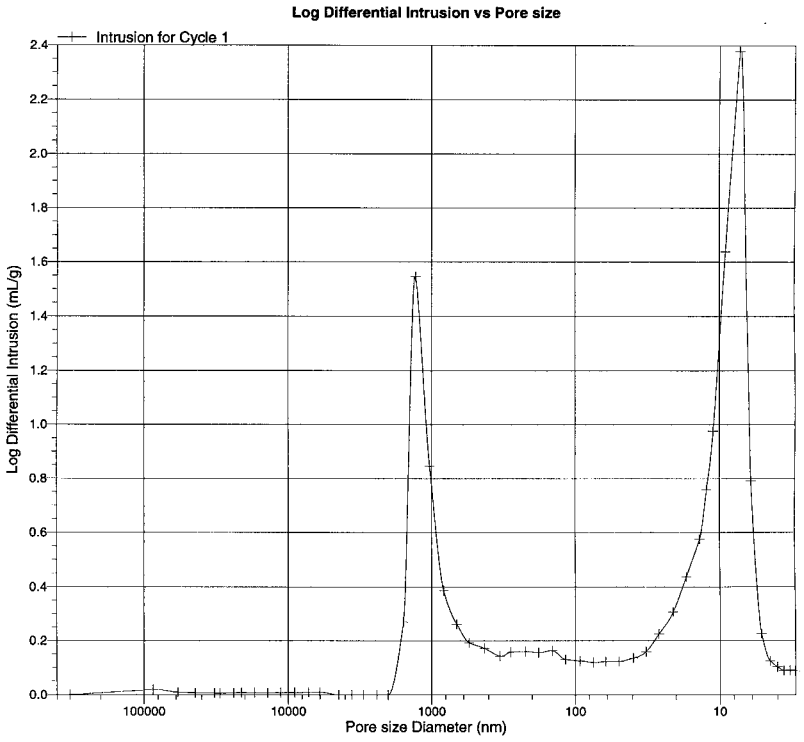
AutoPore IV 9500 VX.XX Serial: 613 Port: 4/2 Page 1

Sample ID: Alumina
Operator: AWT/JK
Submitter: MICROMERITICS
File: C:\AUTOPORE\DATA\CAVITY.SMP

LP Analysis Time:	7/6/99 5:05:26PM	Sample Weight:	0.5545 g
HP Analysis Time:	7/7/99 8:51:04AM	Correction Type:	None
Report Time:	9/12/00 8:45:31AM	Show Neg. Int:	No



Tabular Reports

Tabular reports display the numerical values for data points. You can display up to six columns of data. Refer to **Editing Tabular Reports** in Chapter 4 for editing options.

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AutoPore IV 9500 VX.XX Serial: 379 Port: 2/2

Sample: Rock Sample
Operator: N. KELLY
Submitter: Reseach Lab
File: C:\AUTOPORE\DATA\FRAC.SMP

LP Analysis Time:	5/11/98 10:02:34AM	Sample Weight:	21.6260 g
HP Analysis Time:	5/11/98 11:07:05AM	Correction Type:	Formula
Report Time:	9/12/00 9:00:12AM	Show Neg. Int:	No

Tabular Report

Pressure (psia)	Pore Diameter (nm)	Incremental Pore Volume (mL/g)	Cumulative Pore Volume (mL/g)	Cumulative Pore Area (m^2/g)	Incremental Pore Area (m^2/g)
1.60	113035.5	0.0000	0.0000	0.000	0.000
2.10	86323.9	0.0003	0.0003	0.000	0.000
3.08	58736.9	0.0010	0.0013	0.000	0.000
4.08	44325.3	0.0009	0.0022	0.000	0.000
5.58	32383.9	0.0012	0.0035	0.000	0.000
7.08	25549.9	0.0013	0.0048	0.000	0.000
8.55	21151.9	0.0015	0.0063	0.001	0.000
10.54	17163.3	0.0026	0.0088	0.001	0.001
13.04	13871.5	0.0052	0.0140	0.003	0.001
14.74	12272.4	0.0030	0.0170	0.003	0.001
16.02	11290.5	0.0021	0.0191	0.004	0.001
20.01	9040.0	0.0046	0.0237	0.006	0.002
20.51	8818.7	0.0005	0.0242	0.006	0.000
21.02	8606.2	0.0005	0.0247	0.006	0.000
21.51	8410.0	0.0004	0.0251	0.007	0.000
23.01	7861.9	0.0013	0.0264	0.007	0.001
25.00	7235.0	0.0015	0.0279	0.008	0.001
29.97	6034.1	0.0034	0.0313	0.010	0.002
38.91	4648.0	0.0048	0.0361	0.014	0.004
48.63	3719.6	0.0040	0.0401	0.018	0.004
58.64	3084.3	0.0028	0.0429	0.021	0.003
.	.	.	.	.	.
.	.	.	.	.	.
.	.	.	.	.	.
.	.	.	.	.	.
.	.	.	.	.	.
.	.	.	.	.	.
.	.	.	.	.	.
.	.	.	.	.	.
.	.	.	.	.	.
49480.28	3.7	0.0001	0.0665	3.098	0.126
50246.25	3.6	0.0001	0.0665	3.170	0.072
52952.88	3.4	0.0002	0.0667	3.401	0.231
54445.09	3.3	0.0001	0.0668	3.539	0.138
55971.00	3.2	0.0001	0.0670	3.682	0.143
57907.75	3.1	0.0001	0.0671	3.863	0.182
59938.52	3.0	0.0001	0.0673	4.055	0.192

Cavity to Throat Size Ratio

You can choose to generate a graph and/or tabular data; refer to **Editing Throat Ration Options** in Chapter 4.

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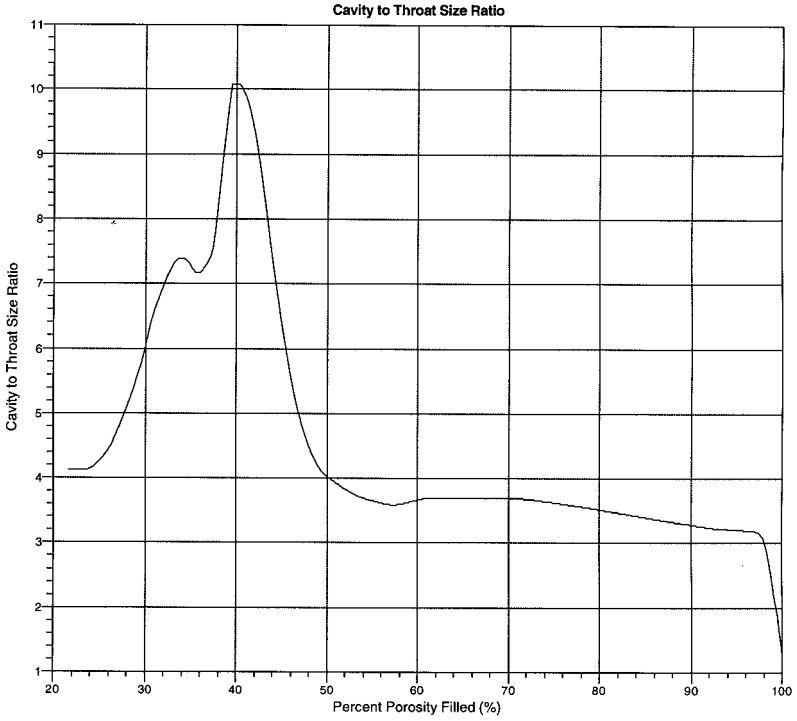
AutoPore IV 9500 VX.XX Serial: 613 Port: 4/2 Page 1

Sample ID: Alumina
Operator: AWT/JK
Submitter: MICROMERITICS
File: A:\6568.SMP

LP Analysis Time:	7/6/1999 5:05:26PM	Sample Weight:	0.5545 g
HP Analysis Time:	7/7/1999 8:51:04AM	Correction Type:	None
Report Time:	9/7/2000 5:51:09PM	Show Neg. Int:	No



Material Compressibility

Refer to **Editing Material Compressibility Options** in Chapter 4 for editing options.

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AutoPore IV 9500 VX.XX Serial: 379 Port: 1/1 Page 3

Sample ID: Clay
Operator: N. KELLY
Submitter: Micromeritics International
File: C:\MIC\DEMO9500\DATA\CLAY.SMP

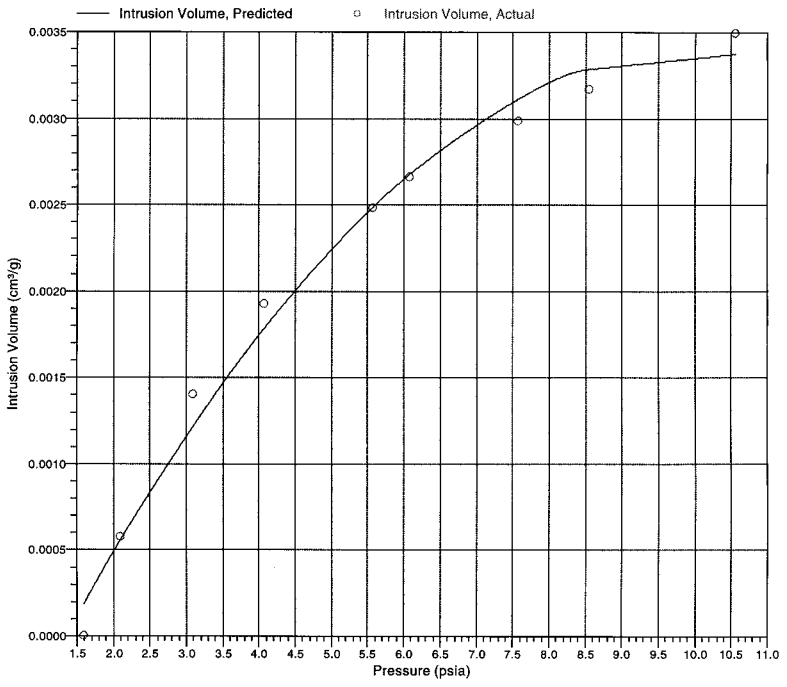
LP Analysis Time: 6/2/1998 3:52:14PM
HP Analysis Time: 6/2/1998 4:52:15PM
Report Time: 9/7/2000 5:40:37PM

Sample Weight: 2.1098 g
Correction Type: Formula
Show Neg. Int: No

Compressibility Summary

Density: 2.7514 g/mL (Calculated)
Linear Compressibility: -2.4748e-03 1/psia
Quadratic Compressibility: 1.2317e-04 1/psia²
RMS Error: 0.0001 mL/g

Material Compressibility



Fractal Dimension

The Fractal Dimension report can be generated to show the backbone formulation and/or the percolation region (shown here). Refer to **Editing Fractal Dimension Options** in Chapter 4 for editing options.

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MICROMERITICS INSTRUMENT CORPORATION

AutoPore IV 9500 VX.XX Serial: 379 Port: 2/2 Page 5

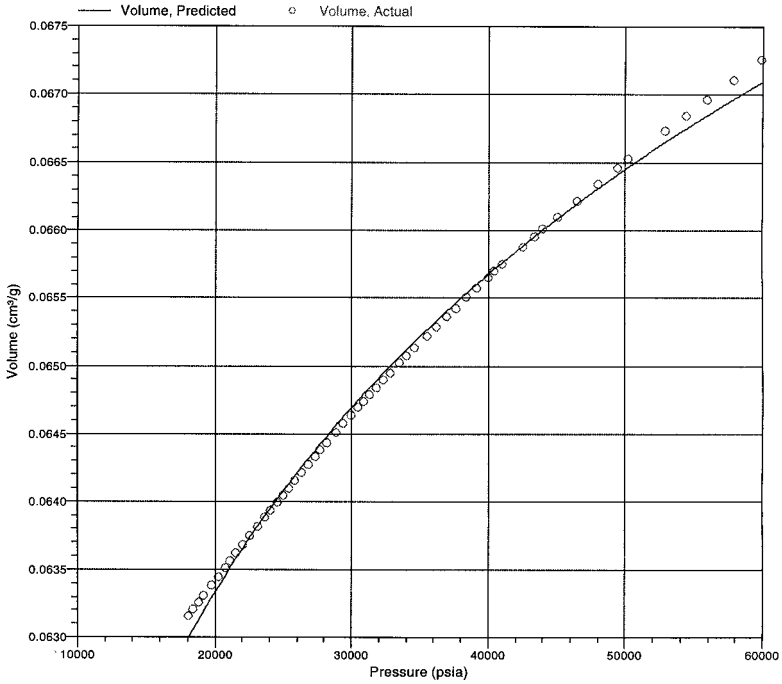
Sample: Rock Sample
Operator: N. KELLY
Submitter: Reseach Lab
File: C:\MIC\DEMO9500\DATA\ROCK.SMP

LP Analysis Time:	5/11/1998 10:02:34AM	Sample Weight:	21.6260 g
HP Analysis Time:	5/11/1998 11:07:05AM	Correction Type:	Formula
Report Time:	9/7/2000 5:48:20PM	Show Neg. Int:	No

Fractal Dimension In Percolation Region Summary

Threshold Pressure: 18.00 psia (Entered)
Pressure Range: 18000.00 - 61000.00 psia
Fractal Dimension : 2.948
RMS Error: 0.0001 mL/g

Fractal Dimension In Percolation Region



Summary

The Summary report provides a condensed listing of data results. The options displayed in the report are chosen by editing the Summary report using the Advanced format. Refer to **Editing the Summary Report** in Chapter 4 for instructions.

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MICROMERITICS INSTRUMENT CORPORATION

AutoPore IV 9500 V**X.XX** Serial: 379 Port: 2/2 Page 1

Sample: Rock Sample
Operator: N. KELLY
Submitter: Reseach Lab
File: C:\MIC\DEMO9500\DATA\ROCK.SMP

LP Analysis Time:	5/11/1998 10:02:34AM	Sample Weight:	21.6260 g
HP Analysis Time:	5/11/1998 11:07:05AM	Correction Type:	Formula
Report Time:	9/7/2000 5:48:19PM	Show Neg. Int:	No

Summary Report

Penetrometer parameters

Penetrometer:	635 - (05) 15 Bulb, 1.836 Stem, Solid		
Pen. Constant:	27.820 µL/pF	Pen. Weight:	62.1860 g
Stem Volume:	1.8360 mL	Max. Head Pressure:	4.4500 psia
Pen. Volume:	16.5382 mL	Assembly Weight:	180.3401 g

Hg Parameters

Adv. Contact Angle:	130.000 degrees	Rec. Contact Angle:	130.000 degrees
Hg Surface Tension:	485.000 dynes/cm	Hg Density:	13.5335 g/mL

User Parameters

Param 1:	0.000	Param 2:	0.000	Param 3:	0.000

Low Pressure:

Evacuation Pressure:	50 µmHg
Evacuation Time:	5 mins
Mercury Filling Pressure:	1.60 psia
Equilibration Time:	10 secs

High Pressure:

Equilibration Time:	10 secs

Blank Correction by Formula

Intrusion Data Summary

Total Intrusion Volume =	0.0673	mL/g
Total Pore Area =	4.055	m²/g
Median Pore Diameter (Volume) =	5317.4	nm
Median Pore Diameter (Area) =	4.9	nm
Average Pore Diameter (4V/A) =	66.3	nm
Bulk Density at 0.10 psia =	2.2993	g/mL
Apparent (skeletal) Density =	2.7198	g/mL
Porosity =	15.4629	%
Stem Volume Used =	74	%

Pore Structure Summary

Threshold Pressure:	18.00	psia (Entered)
Characteristic length =	10048.0	nm
Conductivity formation factor =	0.065	
Permeability constant =	0.00442	
Permeability =	28.8151	mdarcy
BET Surface Area =	200.0000	m²/g
Pore shape exponent =	1.00	
Tortuosity factor =	2.055	
Tortuosity =	6.1496	
Percolation Fractal dimension =	2.948	
Backbone Fractal dimension =	2.405	

CHAPTER 8

TROUBLESHOOTING AND MAINTENANCE

- Troubleshooting
- Preventive Maintenance

TROUBLESHOOTING AND MAINTENANCE

The AutoPore has been designed to provide efficient and continuous service. However, in order to get the best results over the longest period of time, certain maintenance procedures must be followed. Additionally, when operator problems are encountered, the appropriate corrective action must be taken. This section contains both troubleshooting and maintenance procedures.

Troubleshooting

Some common operational problems that are not indicated on the computer screen and their solutions are provided in Table 8-1. Operational problems that are indicated on the screen and their solutions are provided in **Appendix I, Error Messages**.

Table 8-1. Common Operational Problems

What Happened	Why	What To Do
Status display is too large for monitor screen.	Monitor resolution is set below 800 x 600.	Use the Windows Setup function to set your monitor's resolution at or above 800 x 600.
Difficulty attaining adequate vacuum conditions during low pressure analyses.	Vacuum pump oil is low.	Add vacuum pump oil.
	Vacuum hose not properly connected.	Be sure the connection to the vacuum pump is good and that external clamps are tight.
	Sample contains excess moisture adsorbed from atmosphere.	Prepare samples (prior to loading penetrometer) by heat and/or vacuum to remove moisture. **CAUTION:** Never heat a sample that has contacted mercury.

What Happened	Why	What To Do
Difficulty attaining adequate vacuum conditions during low pressure analyses. *(continued)*	Proper sealing not achieved on penetrometers.	Use blank rods to test the ports to eliminate the possibility of having a leaking penetrometer cap/bulb seal. Lightly grease the rods with a high grade vacuum grease, then insert the rods in the low pressure ports. Tighten the retaining knobs on the low pressure ports. If vacuum conditions are satisfactory, check the penetrometer for scratches or chips in the bulb.
	Scratches or other imperfections in either the lip of the penetrometer bulb or the penetrometer cap.	Polish the lip of the bulb and the cap using 600-grit emery paper or crocus cloth. Place the paper or cloth on a flat surface, grit-side up. Clean all grease from the surface to be polished with solvent. 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. Clean the ground surface before regreasing.

What Happened	Why	What To Do
Difficulty attaining adequate vacuum conditions during low pressure analyses. *(continued)*	Leaking valves.	Check the valves for leaks. Refer to *Checking the Valves for Leaks* later in this chapter.
	Moisture has accumulated in the system.	Remove the accumulated moisture. Refer to *Removing Moisture from the System* later in this chapter.
The low pressure analysis is complete and the system has returned to near atmosphere, but the mercury drained indicator is not illuminated.	Indicator is not working properly.	Double-check the state of the mercury degasser by looking at the status display window. If the status display does not agree with the indicator, the indicator may not be working properly. Consult your Micromeritics service representative. If the status display window shows that the mercury has overfilled (the alarm state), follow the instructions for handling mercury overfill (later in this section). If the status display shows that the mercury degasser is either filled or partially filled, follow the steps below.

What Happened	Why	What To Do
The low pressure analysis is complete and the system has returned to near atmosphere, but the mercury drained indicator is not illuminated. *(continued)*	Mercury is not drained. (Status display shows either filled or partially filled.)	In Manual Mode, select **Generate**, **Low pressure** from the **Unit** menu. 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. Then, open the mercury drain valve (#6) until the mercury drains. After the mercury drains, close the mercury drain valve, then the mercury reservoir evacuation valve. To return the system to atmosphere, select **Generate**, **Low pressure** from the **Unit** menu and specify 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), follow the instructions later in this section for handling mercury overfill.

What Happened	Why	What To Do
Mercury warning buzzer sounds in pulses. (Mercury overfill)	Mercury detected by overflow sensor in low pressure port system.	On the Manual Control screen, open the reservoir evacuation valve. Evacuate for twenty seconds. Open the gas inlet valve. Close the gas inlet valve when at least 15 psia or atmosphere is attained. Open the mercury drain valve. Open the front panel of the instrument and locate the Override switch on the small control panel. Press the switch. This allows mercury to drain back into the reservoir. Release the switch as soon as the pulsed buzzing stops. Close the mercury drain valve. Close the reservoir evacuation valve. (If the pulsing tone remains, call the Micromeritics Service Department. You may turn off the AutoPore.)
Mercury warning buzzer sounds with continuous tone.	Mercury has been improperly drawn into the mercury trap.	Drain excess mercury into the reservoir as described above. Remove the plug extending down from the mercury trap. The plug is reached through the front door. Facing the AutoPore, the plug is in the upper, back, right-hand corner. Refer to *Draining Spilled Mercury Dish* later in this chapter. Position a container beneath the trap before removing the plug.

What Happened	Why	What To Do
The high pressure system failed to attain the specified pressure.	Low fluid level in the hydraulic pump.	Add fluid to the hydraulic pump. Refer to *Maintaining Hydraulic Pump Fluid Level* later in this chapter.
High pressure system failed to retain a reasonably constant pressure.	Leakage around the high pressure chamber cap.	Replace the cup-seals and backup rings on the high pressure chamber cap. Refer to *Replacing Chamber Plug Seals* later in this chapter.
Cannot attain low pressure data points above atmospheric pressure.	Gas supply pressure too low.	Verify gas supply regulator is set at 40 to 45 psig (276 to 310 kPa) and that there is sufficient gas in the supply bottle.

Preventive Maintenance

Table 8-2 lists the maintenance procedures which should be performed to keep the AutoPore in proper operating condition. Instructions for each procedure follow the table.

Table 8-2. Preventive Maintenance Schedule

Maintenance Required	Frequency
Maintaining mercury level	Service daily
Draining spilled mercury dish	Check daily, service as needed
Maintaining high pressure fluid level	Check before performing high pressure run, fill as needed
Maintaining vacuum pump fluid level	Check monthly, fill as needed
Greasing low pressure ports	Every 600 samples or 3 months
Replacing chamber plug seals	Every 600 samples or 3 months
Changing the high pressure fluid and cleaning the high pressure chambers	Check every 600 samples or 3 months
Maintaining hydraulic pump fluid level	Check every six months, fill as needed
Changing vacuum pump fluid	Every 1200 samples or 6 months
Replacing vacuum pump exhaust filter	Every 600 samples or 3 months
Checking the valves for leaks	As needed
Cleaning valves	As needed
Removing moisture from the system	As needed
Replacing the banana plug	Check every 3 months or 600 samples, replace as needed

Maintaining Mercury Level

Each analysis may extract from 3 to 15 mL of mercury from the reservoir depending on the penetrometer and sample size used. A message is displayed on the Low Pressure Analysis screen 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.

1. Remove the mercury reservoir cap.
2. Obtain the mercury-filling funnel (provided in your accessories kit), clean with IPA, and insert it into the mercury reservoir.
3. 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.

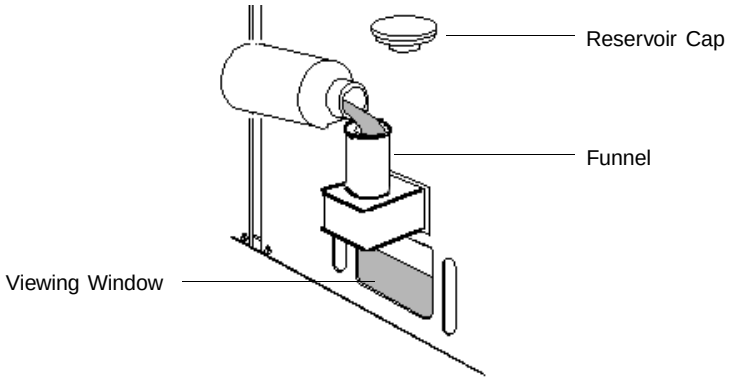


Figure 8-1. Maintaining Mercury Level

Do not overfill. Doing so may interfere with the vacuum system.

4. Remove the funnel, clean with IPA, and store for the next use. Some AutoPore users have found the vacuum pump tray a convenient, safe location for storage.
5. Replace the cap.

Draining Spilled Mercury Dish

A dish for collecting mercury is located just behind the high pressure chambers (refer to Figure 8-2). If mercury accumulates in the dish, remove it by removing the cover and extracting the mercury with the syringe accessory.

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

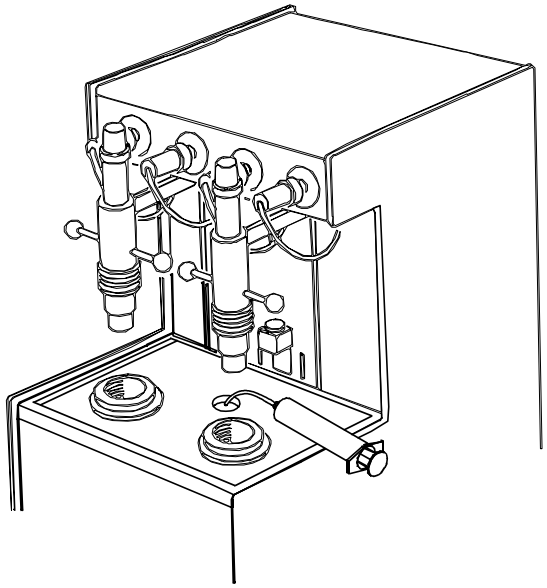


Figure 8-2. Draining Spilled Mercury Dish

Maintaining High Pressure Fluid Level

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

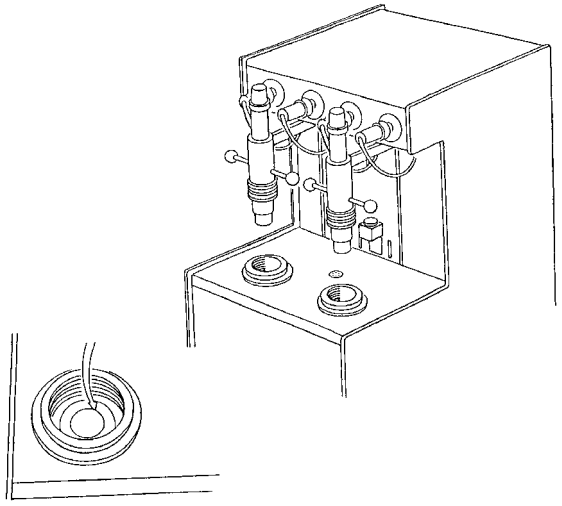


Figure 8-3. Maintaining High Pressure Fluid Level

Maintaining Vacuum Pump Fluid Level

The oil level in the pump should be checked monthly. Refer to the vacuum pump user's manual for instructions.

Greasing Low Pressure Ports

The screw threads visible behind each low pressure port retaining knob should be greased monthly as follows:

1. Remove the capacitance detectors by turning counterclockwise.
2. Unscrew the retaining knobs until the threads disengage.

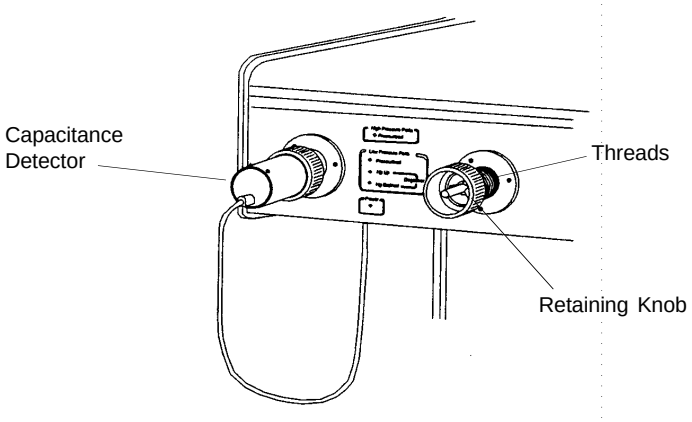


Figure 8-4. Greasing the Low Pressure Ports

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

Replacing Chamber Plug 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 should be replaced when you notice the backup ring extruding, small slivers coming from the cup-seal or backup ring, or pinched areas on the cup-seal. Also, the rings may need replacing when you see oil coming up around the top of the chamber, or when it is difficult to maintain the desired pressure in the high pressure chamber.

1. Remove the cup-seal and backup ring, being careful not to scratch the surrounding surfaces.

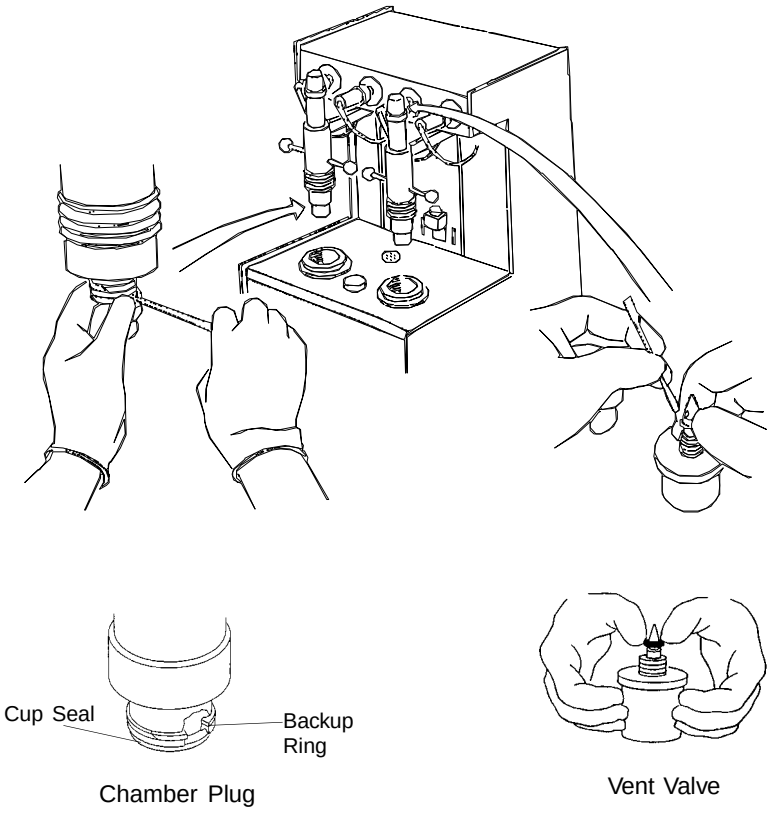


Figure 8-5. Replacing O-Ring, Backup Ring, and Cup-Seal

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 as shown in Figure 8-6.

Changing High Pressure Fluid and Cleaning the High Pressure Chambers

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

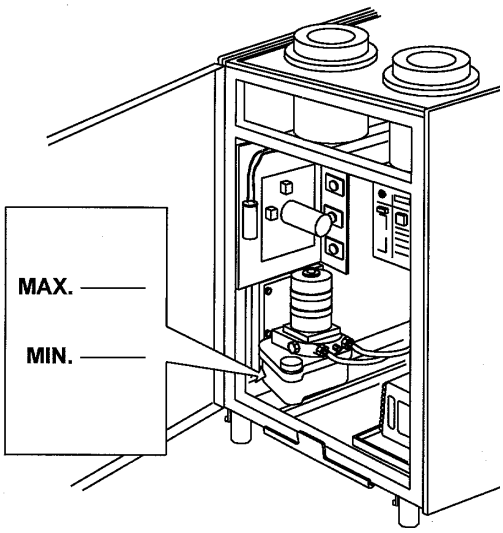
1. Remove the high pressure fluid from the high pressure chambers using the syringe provided.

2. Clean the high pressure chambers using a clean cloth dampened with isopropyl alcohol.

3. When the chambers are completely dry, add new high pressure fluid.

Maintaining Hydraulic Pump Fluid Level

1. View the oil level through the transparent reservoir. Oil should fall between the minimum and maximum marks.



2. Add fluid if necessary.

Changing Vacuum Pump Fluid

The most common cause of loss in efficiency in a vacuum pump is contaminated fluid. Contamination is caused by condensation of vapors and by foreign particles. The pump should be completely drained and filled with fresh fluid when contamination occurs. Refer to the vacuum pump user's manual for instructions on changing the fluid.

Replacing Vacuum Pump Exhaust Filter

The gases used by the instrument are exhausted by the vacuum pump. An exhaust filter is used on the exhaust port of the vacuum pump to minimize the release of oil vapor. Refer to the vacuum pump user's manual for instructions on replacing the filter.

Checking the Valves for Leaks

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

1. Open valves 1, 2, 3, and 8.

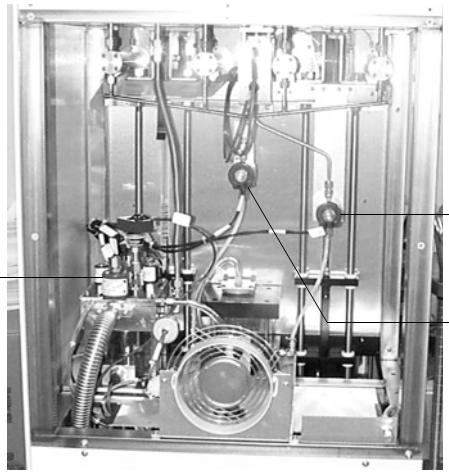
2. If the pressure reaches 100 μm in less than five minutes, the system has a leak. If this occurs, clean the valves as described in the next section.

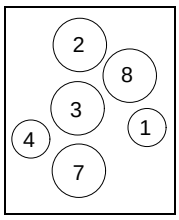
Cleaning Valves

Make sure 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.

Turn off power to the unit 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 unscrewing the retaining screws.
3. Remove the 11/16-in. nut retaining the valve actuating coil from the mercury fill and drain valves. Pull the coils off.





Top View of Valve Area

1 Servo Isolation Valve
2 Fast Evacuation Valve
3 Vacuum Valve
4 Gas Inlet Valve
7 Mercury Reservoir Evacuation Valve
8 Evacuation Reservoir Valve

Figure 8-6. Location of Valves

4. Hold 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 isopropyl alcohol, and expose the valve seat. Use a pipe cleaner to clean the valve seat.

7. Make sure that no lint remains on the sealing surface, then reassemble 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 in the manner described above if cleaning the mercury valves did not solve the vacuum difficulty.

Removing Moisture from the System

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 AutoPore at full rate for several days. If the indicated vacuum continues to decrease slowly over this period, accumulated moisture is the probable cause of the vacuum difficulty.

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

Replacing the Banana Plug

High Pressure Chamber

Replace the banana plug in the high pressure chamber as follows:

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.

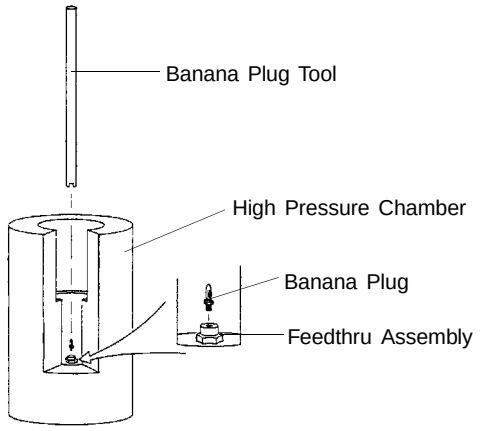


Figure 8-7. Replacing the Banana Plug in the High Pressure Chamber

4. Insert a new banana plug into the banana plug tool, making sure 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

Replace the banana plug in each of the low pressure capacitance detectors as follows:

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.

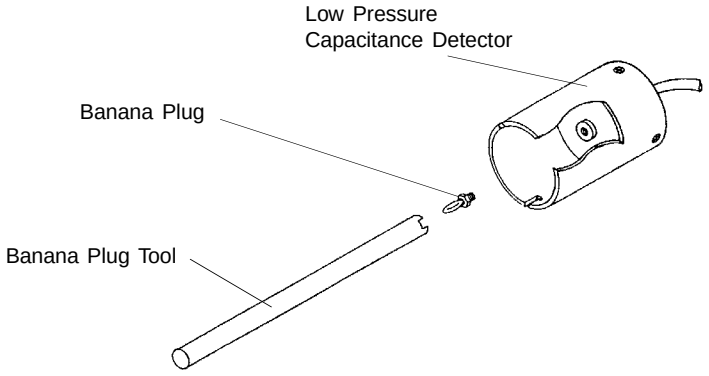


Figure 8-8. Replacing the Banana Plug in the Low Pressure Capacitance Detector

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

CHAPTER 9

ORDERING INFORMATION

ORDERING INFORMATION

Part Number	Item and Description
952-00000-00	AutoPore IV 9520 (100/115/230 VAC, 50/60 Hz) with four low- and two high-pressure ports, for automatic pore structure studies from 360 to 0.003 µm pore opening diameter. Includes computer interface cable and latest software. Requires computer.
951-00000-00	AutoPore IV 9510 (100/115/230 VAC, 50/60 Hz) with two low- and one high-pressure port, for automatic pore structure studies from 360 to 0.003 µm pore opening diameter. Includes computer interface cable and latest software. Requires computer.
951-00002-00	Same as 951-00000-00, no software included.
950-00000-00	AutoPore IV 9500 (100/115/230 VAC, 50/60 Hz) with four low- and two high-pressure ports, for automatic pore structure studies from 360 to 0.005 µm pore opening diameter. Includes computer interface cable and latest software. Requires computer.
950-00002-00	Same as 950-00000-00, no software included.
950-50000-00	AutoPore IV 9505 (100/115/230 VAC, 50/60 Hz) with two low- and one high-pressure port, for automatic pore structure studies from 360 to 0.005 µm pore opening diameter. Includes computer interface cable and latest software. Requires computer.
950-50002-00	Same as 950-50000-00, no software included.
Call for current model and part number	Computer, controls analyses, reduces data, and generates reports; includes color monitor, printer, and cables (100/120 or 230 V, 50/60 Hz).
150-10000-00	Contact Anglometer 1501, for wetting (contact) angle between liquid and flat surface or packed powder bed
950-33001-00	Software and operator's manual, AutoPore IV
950-42801-00	Operator's manual, AutoPore IV
004-25105-00	Front ferrule, Teflon, for 3/8-in. OD tubing
004-25103-00	Front ferrule, Teflon, for 1/4-in. OD tubing
004-25104-00	Rear ferrule, nylon, for 1/4-in. OD tubing
004-25106-00	Rear ferrule, nylon, for 3/8-in. OD tubing

Part Number	Item and Description
950-33600-00	Extended operation supplies for AutoPore IV 9520 and AutoPore IV 9510. Includes O-rings, greases, high-pressure fluid, vacuum pump oil, seals, and valve parts.
942-33021-00	Exhaust Fan Kit; allows you to use a fan as the ventilation method on the AutoPore IV.
004-62200-58	Gas regulator, 2-stage, 100 psia, CGA 580
004-33601-00	Expansion Kit; adds an additional outlet to the gas regulator, includes fittings and instructions
004-16004-01	Mercury, 5 lbs
950-25842-00	Funnel, for pouring mercury into the reservoir
004-16007-00	Apiezon-H vacuum grease, 25 g; operating range is 15 to 250°C
008-16045-00	Dow Corning silicone grease, high vacuum, 150-g tube
004-16011-00	Oil, synthetic, for hydraulic pump
920-16001-00	High-pressure fluid, 1 liter, a proprietary blend of oils
004-16003-01	Vacuum pump oil, 1 liter
942-25823-00	Syringe, 50 cc, used to withdraw high-pressure fluid when cleaning chambers
930-54601-00	Cleaning brush, 0.4-cc stem, used to aid in the cleaning of penetrometers
930-54602-00	Cleaning brush, 1.1-cc stem
930-54603-00	Cleaning brush, 1.8-cc stem
930-17801-00	Penetrometer simulator, 82 pF, used to verify capacitance (intrusion) calibration.
930-17801-01	Penetrometer simulator, 150 pF
930-17801-05	Penetrometer simulator, 15 pF
004-16822-00	Reference material, average pore diameter ≅ 0.01 μm, pore volume ≅ 0.5 mL/g; 15 g
930-25849-00	Banana plug
922-09808-00	Tool, banana plug
004-25023-00	O-ring, size 016, Buna-N, for mercury reservoir cap
004-25466-00	O-ring, size 010, Buna-N, for mercury degasser seals
004-25034-00	O-ring, size 008, Buna-N, for chamber plug relief valve
004-25297-00	Backup ring, for chamber plug seal
922-25890-00	Seal, high pressure chamber plug

Part Number	Item and Description
	Penetrometer Assemblies for Solid Samples
950-61713-00*	Penetrometer, solids; 3-cc sample volume, 0.39-cc intrusion volume
920-61713-01	Glassware only for 950-61713-00
950-61715-00*	Penetrometer, solids; 3-cc sample volume, 1.12-cc intrusion volume
920-61715-01	Glassware only for 950-61715-00
950-61707-00*	Penetrometer, solids; 5-cc sample volume, 0.37-cc intrusion volume
920-61707-01	Glassware only for 950-61707-00
950-61709-00*	Penetrometer, solids; 5-cc sample volume, 1.06-cc intrusion Volume
920-61709-01	Glassware only for 950-61709-00
950-61711-00*	Penetrometer, solids; 5-cc sample volume, 1.72-cc intrusion Volume
920-61711-01	Glassware only for 950-61711-00
950-61701-00*	Penetrometer, solids; 15-cc sample volume, 0.37-cc intrusion volume
920-61701-01	Glassware only for 950-61701-00
950-61703-00*	Penetrometer, solids; 15-cc sample volume, 1.06-cc intrusion Volume
920-61703-01	Glassware only for 950-61703-00
950-61705-00*	Penetrometer, solids; 15-cc sample volume, 1.72-cc intrusion volume
920-61705-01	Glassware only for 950-61705-00
950-61724-00*	Penetrometer, solids; 15-cc sample volume, 3.01-cc intrusion volume
920-61724-01	Glassware only for 950-61724-00
950-61725-00*	Penetrometer, solids; 15-cc Sample Volume, 3.86-cc Intrusion Volume
920-61725-01	Glassware only for 950-61725-00

*Includes glassware

Part Number	Item and Description
	Penetrometer Assemblies for Powder Samples
950-61714-00*	Penetrometer, powder; 3-cc sample volume, 0.39-cc intrusion volume
920-61714-01	Glassware only for 950-61714-00
950-61716-00*	Penetrometer, powder; 3-cc sample volume, 1.12-cc intrusion volume
920-61716-01	Glassware only for 950-61716-00
950-61708-00*	Penetrometer, powder; 5-cc sample volume, 0.37-cc intrusion Volume
920-61708-01	Glassware only for 950-61708-00
950-61710-00*	Penetrometer, powder; 5-cc sample volume, 1.06-cc intrusion Volume
920-61710-01	Glassware only for 950-61710-00
950-61712-00*	Penetrometer, powder; 5-cc sample volume, 1.72-cc intrusion volume
920-61712-01	Glassware only for 950-61712-00
950-61702-00*	Penetrometer, powder; 15-cc sample volume, 0.37-cc intrusion volume
920-61702-01	Glassware only for 950-61702-00
950-61704-00*	Penetrometer, powder; 15-cc sample volume, 1.06-cc intrusion volume
920-61704-01	Glassware only for 950-61704-00
950-61706-00*	Penetrometer, powder; 15-cc sample volume, 1.72-cc intrusion volume
920-61706-01	Glassware only for 950-61706-00

*Includes glassware

Part Number	Item and Description
	Other Penetrometer Parts
950-25831-02	Seal (metal cap) replacement, for 3- and 5-cc sample volume penetrometers
950-25832-02	Seal (metal cap) replacement, for 15-cc sample volume penetrometers
950-25831-01	Nut, 3- and 5-cc penetrometers
950-25832-01	Nut, 15-cc penetrometer
942-25863-00	Spacer for 15-cc penetrometers
950-25831-03	Spacer for 3- and 5-cc penetrometers
950-09803-00	Penetrometer wrench
950-09802-00	Penetrometer holder

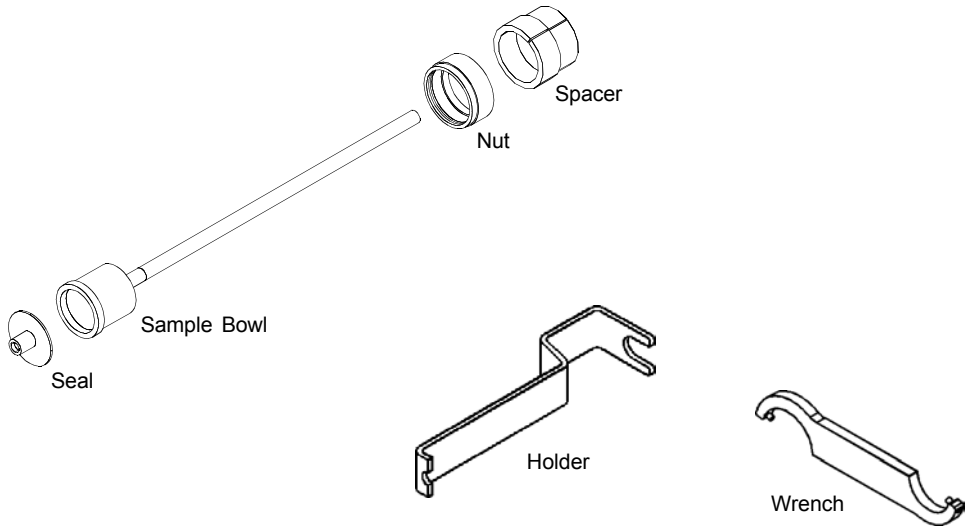


Figure 9-1. Penetrometer Parts

APPENDIX A

FORMS

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Analysis Conditions File Worksheet

Use this worksheet to assemble the data you need to:

- **Basic format:** create analysis conditions files that can be selected from the drop-down lists. From the **File** menu, select **Open**, **Analysis Conditions.** Name the file and select **OK**.
- **Advanced format:** complete the analysis conditions portion of the sample file. With the advanced format sample file open, click on the **Analysis Conditions** tab.

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:	

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Sample Data Worksheet

Use this worksheet to help you gather the sample data needed to create the sample file.To begin creating sample files: From the main menu, select **File**, **Open**, **Sample Information**. Choose the file name for the first sample file, then click **OK**. Enter the data you have collected here. Repeat for all samples to be included in this analysis.

☐ Basic Format Sample Files
☐ Advanced Format Sample Files

	Port 1	Port 2	Port 3	Port 4
Sample file name or identifier: *(optional)*				
Penetrometer number: *(optional; etched on penetrometer)*				
Sample weight: *(0.000 to 500.0000 g)* Enter this value on the Sample Information screen.				
Sample + penetrometer weight: Load the penetrometer with sample and weigh.				
Penetrometer weight: Subtract the **Sample weight** from the **Sample + penetrometer weight**; enter the difference here. -Basic Format: Enter this value on the Sample Information screen. -Advanced Format: Enter this value on the Penetrometer Properties screen.				
High pressure port number: *(optional)* Enter the number of the high pressure port into which this sample is loaded.				
Assembly weight: The weight of the **sample + penetrometer + mercury**. (Weigh the penetrometer after the low pressure analysis.) Enter this value on the Start High Pressure Analysis screen.				

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Penetrometer Properties File Worksheet

Use this worksheet to assemble the data you need to:

- **Basic format:** create penetrometer properties files that can be selected from the drop-down lists. From the **File** menu, select **Open**, **Penetrometer Properties**. Name the file and click **OK**.

- **Advanced format:** complete the penetrometer properties portion of the sample file. With the advanced format sample file open, click on the **Penetrometer Properties** tab.

Penetrometer number: (etched on penetrometer)	
File name:	
Weight: Weigh the loaded penetrometer. Subtract the weight of the sample. Enter the difference here.	
Volume:	
Constant:	
Stem Volume:	
Max. head pressure:	
Correction Method: ☐ Blank ☐ Formula ☐ None	If Blank, give file name:

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Penetrometer Volume Calibration

Penetrometer Number: _____ Date: _____

By: _____

First Calibration of Penetrometer Volume:

1. Weight of penetrometer filled with mercury . . . . . _____ g
2. Weight of sealed, empty penetrometer . . . . . _____ g
3. Weight of mercury (No. 1 minus No. 2) . . . . . _____ g
   Room temperature = _____°C
4. Volume of penetrometer (No. 3 divided by Density of Mercury*) . . . . . _____ cc

Second Calibration of Penetrometer volume:

1. Weight of penetrometer filled with mercury . . . . . _____ g
2. Weight of sealed, empty penetrometer . . . . . _____ g
3. Weight of mercury (No. 1 minus No. 2) . . . . . _____ g
   Room temperature = _____°C
4. Volume of penetrometer (No. 3 divided by Density of Mercury*) . . . . . _____ cc

Third Calibration of Penetrometer Volume:

1. Weight of penetrometer filled with mercury . . . . . _____ g
2. Weight of sealed, empty penetrometer . . . . . _____ g
3. Weight of mercury (No. 1 minus No. 2) . . . . . _____ g
   Room temperature = _____°C
4. Volume of penetrometer (No. 3 divided by Density of Mercury*) . . . . . _____ cc

Average Volume of Penetrometer ($\overline{V}$) . . . . . _____ cc

*Density of Mercury, refer to the following table.

°C	g/cc	°C	g/cc	°C	g/cc	°C	g/cc
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

Comments: _____

_____

_____

_____

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AutoPore IV Pressure Table and Tabular Data Worksheet

- **Basic Format**: The pressure table is accessed by clicking [Pressure] on the Sample Information screen.
- **Advanced Format**: The pressure table is accessed by clicking [Pressure], which is located on the Analysis conditions portion of the Sample Information screen.
- **Tabular Data Set**: When you are editing a tabular report, you can open the Tabular Data Set window by clicking on the Tabular Report Options screen.

For pressure table, enter:

Filling pressure: _____
Last low pressure point index: _____

For tabular data:

☐ Pressure
☐ Pore size

Entry	Entry	Entry	Entry

Entry	Entry	Entry	Entry

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Report Options File Worksheet

Report options files are created for use in Advanced format. Use this worksheet to assemble the data you need to complete the report options portion of the sample file.

- To create an independent report options file that can be used on subsequent sample files: from the main menu, select **File**, **Open**, **Report Options.** Name the file and click **OK**.

- To complete the report options portion of this sample file only: click on the **Report Options** tab of the open advanced format sample file. (If you use this option, it is not possible to copy this group of data automatically onto subsequent sample files using the **Replace** button.)

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:

APPENDIX B

THEORY

THEORY

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

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

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 "ink-well" structures. These phenomena give rise to hysteresis phenomena, i.e., distinct intrusion and extrusion P-V curves. Refer to Appendix H for a discussion of surface area calculations.

APPENDIX C

PROPER HANDLING OF MERCURY

PROPER HANDLING OF MERCURY

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

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

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

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

Proper clean-up of mercury spills and disposal of mercury-contaminated articles will limit exposure. In a poorly ventilated, closed area, where mercury spills have not been properly and thoroughly cleaned, mercury concentration in air can become significantly elevated above the TLV of 0.05 mg per cubic meter of air. Figure C-1 shows that the equilibrium concentration of mercury at a room temperature of 25°C reaches a level of 20 mg per cubic meter of air. This is 400

times the TLV, resulting in a dangerous work environment. Surveys in labs where mercury is routinely used reveal the presence of mercury in porous surfaces, in pools under cabinets or floors, and inside drawers and lab equipment. This accumulation can be attributed to the lack of an effective clean-up procedure for both large and small spills.

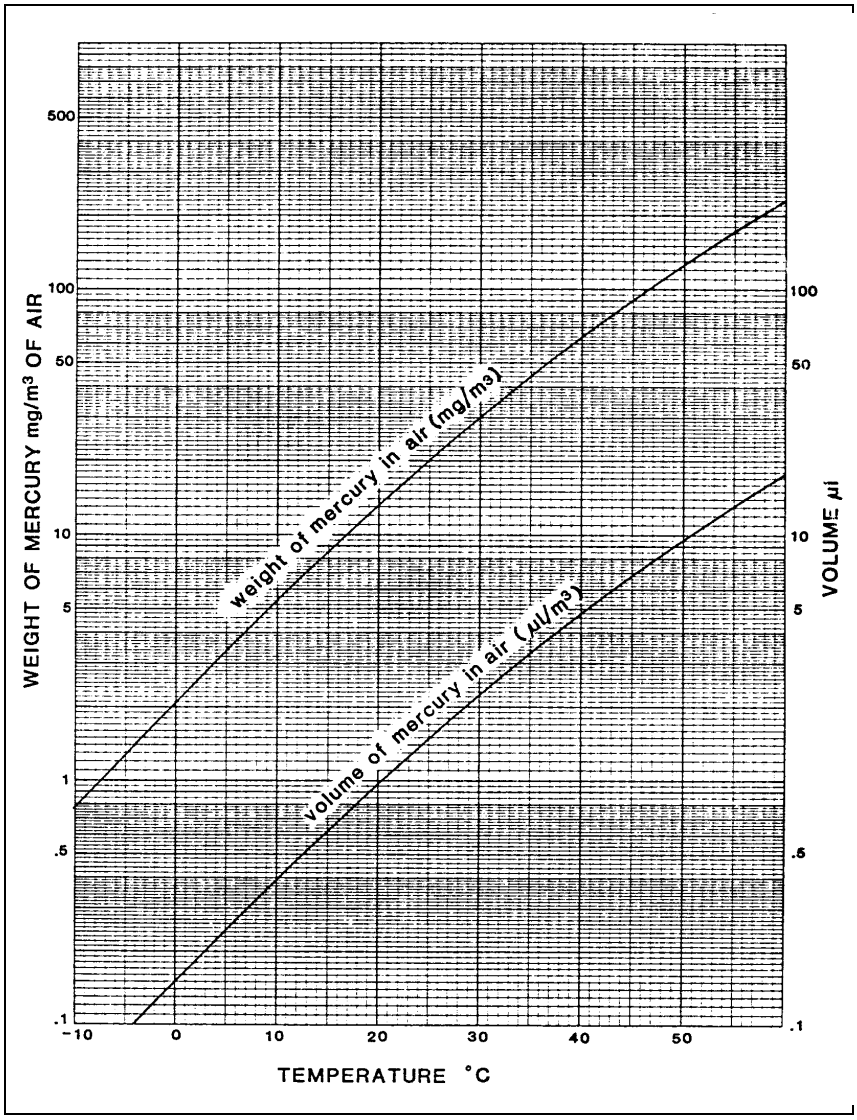


Figure C-1. Equilibrium Concentration of Mercury

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 decontaminant* and allowed to dry.

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

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.

* 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.

Proper Use of Mercury as a Tool in Pore Structure Analyses

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

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

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

APPENDIX D

DATA REDUCTION

DATA REDUCTION

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

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

$$\text{WASHCON=Washburn constant} = \frac{10^4\ \mu m/cm}{68947.6\ \text{dynes}/cm^2\text{–psia}} = 0.145038$$

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

Diameter for the i^{th} point is:

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

Radius for the i^{th} point is:

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

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

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

Mean diameter for the i^{th} point is:

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

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

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

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

Incremental specific pore area for the i^{th} point is:

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

Cumulative specific pore area for the i^{th} point is:

$$A_i = Ai_i + Ai_{i-1} + \ldots + Ai_1$$

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

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

1. Interpolation of cumulative specific intrusion volume vs. log diameter is made to get cumulative specific intrusion volume corresponding to evenly spaced log diameters.

2. The uniform cumulative specific intrusion volume data are then subjected to a 1st derivative calculation, using a 9-point smoothing method. This gives the desired differential data in terms of uniform intervals of collected data.

3. Log differential data are normalized by dividing by the log diameter interval between points. Since the points are evenly log spaced, this interval is the same for all points. Differential data are normalized by dividing by the diameter interval between points. Since the points are evenly log spaced, this interval is larger for larger diameters.

4. Interpolation of the differential or log differential data vs. log diameter is made to get data corresponding to collected data points.

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

Differential specific intrusion volume by diameter for the i^{th} point is:

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

Log differential specific intrusion volume by diameter is:

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

Differential specific intrusion volume by radius for the i^{th} point is:

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

Log differential specific intrusion volume by radius is:

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

Total intrusion volume is:

$$V_{tot} = V_j$$

where the j^{th} point is the first such that:

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

Total specific intrusion volume is:

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

Percent of total specific intrusion volume for the i^{th} point is:

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

Total specific pore area is:

$$A_{tot} = A_j$$

for point j as defined above.

Median diameter by volume is:

$$D_{mv} = D_k$$

where

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

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

Median diameter by area is:

$$D_{ma} = D_k$$

where

$$A_k = \frac{A_{tot}}{2}$$

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

Average diameter is:

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

Blank Correction by Formula:

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

For equilibration time < 6 seconds: X = 0.0

$$A_1 = [1.23 \times 10^{-7} + 2.67 \times 10^{-7}X] - V_p [1.78 \times 10^{-7} + 1.0 \times 10^{-8}X] + V_m [1.64 \times 10^{-7} + 2.4 \times 10^{-8} X]$$

$$A_2 = -2.78 \times 10^{-12} X$$

For intrusion,

$$B = A_1P_i + A_2P_i^{\,2}$$

For extrusion points ≥ 1000 psia,

$$B = A_1P_i + A_2P_i^{\,2} + 8.85 \times 10^{-3} \left(1\frac{P_i}{60000}\right)$$

For extrusion points < 1000 psia,

$$B = A_1P_i + A_2P_i^{\,2} + 8.7 \times 10^{-6} P_i$$

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

$$V_i = Vr_i - B$$

where

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

Blank correction by file is described in Chapter 3.

Bulk volume is:

$$V_b = V_p - V_m$$

Bulk density is:

$$Y_b = \frac{W_s}{V_{bfp} - V_{bup}}$$

Skeletal volume is:

$$V_s = V_b - V_{tot}$$

Skeletal density is:

$$Y_s = \frac{W_s}{V_s}$$

Porosity % is:

$$P_{pc} = \frac{100 \text{ x } V_{tot}}{V_b}$$

Percent capillary used is:

$$V_{pc} = \frac{100 \text{ x } V_{tot}}{V_c}$$

Computation Algorithm for Volumetric Pressure Coefficients of Compressibility by AutoPore 9500

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

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

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

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

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

$$V(Pn) = V_o + B*Pn + C*Pn^2 .$$

where

V_o = the exact volume of the sample material computed as the ratio of the sample weight and the sample density supplied by the user or, alternatively, supplied as the pre-measured sample volume by the user;

B = the linear pressure coefficient of volumetric compressibility (must be a negative real number to avoid violation of fundamental physical laws); and

C = the quadratic pressure coefficient of volumetric compressibility.

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

$$\sum_{n=1}^{n=Nmax} \left[V(Pn) - (Vo + B*Pn + C*Pn^2) \right]^2 = \text{minimum}$$

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

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

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

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

Fractal Dimensions

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

According to percolation theory (see reference to Katz and Thompson in Permeability seciton of this appendix), at some threshold pressure P_T, the invading fluid first apans the entire sample, that is, the fluid percolates for the first time. This then produces a geometrical configuraiton of fluid known as the percolation backgone and pressures from the point of percolation to completion of the backgone are in the "backbone formation" region. At greater pressures, filling of pore cavities behind smaller pore throats continues but wihtout the sudden influx of fluid as observed at the threshold pressure. The backbone is a fractal with fractal dimension D_H, but at higher pressures, the geometry of the fluid cluster changes rapidly to another fractal with fractal dimentsion D_V ($>D_H$) of the supporting media.

MIP Data Reduction

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

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

The equation that defines fractal dimension is as follows:

$$I = \alpha \, (P - P_{thresh})^{(3-D)}$$

* R.F. Angulo, V. Alvarado, and H. Gonzalez, "Fractal Dimensions from Mercury Instrusion Capillary Tests," II LAPEC, Caracas, March 1992.

where

I	=	specific intrusion volume
P	=	pressure
P_{thresh}	=	threshold pressure
D	=	the fractal dimension
α	=	proportionality constant

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

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

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

Material Permeability

Background

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

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

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

Basis of Data Reduction Method to be Used

1. A.J. Katz and A.H.Thompson, Quantitative prediction of permeability in porous rock: Physical Review, Series B, Vol. 34, pp. 8179-8191 (1986).

2. A.J. Katz and A.H. Thompson, "Prediction of Rock Electrical Conductivity From Mercury Injection Measurements," *Journal of Geophysical Researc*h, *Vol. 92*, No. B1, pp. 599-607, (1987).

3. E.J. Garboczi, "Mercury Porosimetry and Effective Networks for Permeability Calculations in Porous Materials," NIST.

4. Kelli Murbach, "Permeability in Cement Impedance Spectroscopy," Case Western Reserve University.

5. P.J. Tumidajski and B. Lin, "On the Validity of the Katz-Thompson Equation for Permeabilities in Concrete", pp. 643-647.

6. A.H. Thompson, A.J. Katz, and C.E. Krohn, "The microgeometry and transport properties of sedimentary rock," *Advances in Physics*, *Vol*. 36, No. 5, pp. 625-694 (1987).

7. A.H.Thompson, S.W. Sinton, S.L. Huff, A.J. Katz, R.A. Raschke, and G.A.Gist, "Deuterium magnetic resonance and permeability in porous media," *Journal of Applied Physics*, *Vol.* 65, pp. 3259-3263 (1989).

Theory

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

$$k = cl_c^2 \, \sigma/\sigma_o$$

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

The Katz-Thompson Methof of Data Reduction Using Mercury Porosimetry

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

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

$$\text{Perm} = C\, L^2_{char}\, \sigma/\sigma_o$$

C = user-entered permeability constant

σ/σ_o = user-entered conductivity formation factor

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

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

$$\text{Perm} = \frac{1}{89} L^2_{max} \frac{L_{max}}{L_{char}} \cdot I_{tot} \cdot Y_b \cdot S_{Lmax}$$

$$\frac{\sigma}{\sigma_o} = \frac{\text{Perm}}{CL^2_{max}}$$

where

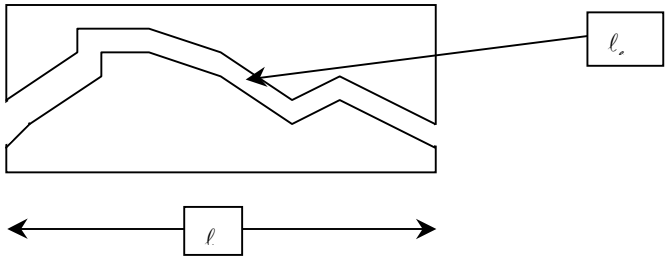
C = user-entered permeability constant

Y_b = bulk density, either calculated (if chosen) or user-entered on the Material Properties dialog

Tortuosity

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

$$\xi = \text{tortuosity} = \frac{\text{actual distance traveled}}{\text{shortest distance}} = \frac{l_e}{L} \quad (1)$$



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

ρ = density (mass/volume) – from pycnometry

V_{tot} = total pore volume (volume/mass)

K = permeability (area)

Differential intrusion from Hg Porosimetry

The tortuosity can be calculated from the following expression:

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

where

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

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

$$D_{avg}^2 = Y_s\left[\frac{1}{2} I_1 O_1^2 + \sum I_i D_i^2 + \frac{1}{2} I_n O_n^2\right]$$

where

Y_S = skeletal or true density, either calculated (if chosen) or user-entered on the Material Properties dialog

D_i = pore diameter for the i^{th} point

I_i = specific intrusion volume for the i^{th} point

Given this value, the tortuosity is calculated as follows:

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

where

$Perm$ = $C\, L^2_{char}\, \sigma/\sigma_o$
I_{tot} = total specific intrusion volume

Calculating Tortuosity Factor

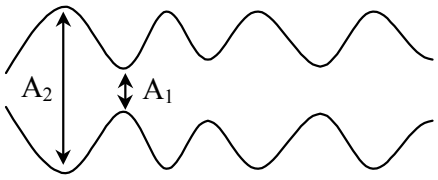
Tortuosity factor is commonly used in the area of heterogeneous catalysis and is the ratio of tortuosity to constriction.

$$\tau = \frac{D_{eff}}{D\theta_c} \quad (4)$$

where

D = diffusivity
D_{eff} = effective diffusivity

$$\tau = \frac{\xi}{\sigma} \quad (5)$$



$\beta = \frac{A_2}{A_1}$, area ratio

σ = f(beta) , constriction factor

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

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

For nonintersecting cylindrical pores the following simple correlation may be used:

$$\tau = 2.23 - 1.13\ V_{tot}\rho_b \quad (6)$$

where

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

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

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

$$\tau = (2.23 - 1.13\ V_{tot}\rho_b)\left(0.92\left(\frac{4}{S}\sum\frac{\Delta V_i}{d_i}\right)^{1+\varepsilon}\right) \quad (7)$$

where

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

The tortuosity factor is calculated as follows:

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

where

ΔI_i = difference in specific intrusion volume for two adjacent points $I_i - I_{i-1}$

$\overline{D}$ = average pore size for the interval between adjacent points $0.5\ (D_i + D_{i-1})$

S = user-entered BET surface area

I_i = specific intrusion volume for the i^{th} point

D_i = pore diameter for the i^{th} point

I_{tot} = total specific intrusion volume

Y_b = envelope density, either calculated (if chosen) or user-entered on the Material Properties dialog

APPENDIX E

EXPORTED DATA FORMAT

EXPORTED DATA FORMAT

The collected data are exported as a two-column table with commas between the entries on a row of the table. No blank correction is applied.

If an ASCII version of a reduced report is desired, it may be obtained by printing the report with a destination of “File”.

The exact export format is as follows:

<sample status>(2=Analyzing, 4=Entered, 8=No Analysis,

16=LP Complete, 32=HP Complete)

<sample id>(quoted string)

<sample weight (g)>

<blank line>

<hg advancing contact angle (degrees)>

<hg receding contact angle (degrees)>

<hg surface tension (dynes/cm)>

<hg density (g/ml)>

<blank line>

<pressure 1 (psia)>,<intrusion volume 1 (mL)>

<pressure 2 (psia)>,<intrusion volume 2 (mL)>

<pressure 3 (psia)>,<intrusion volume 3 (mL)>

.

.

.

<pressure N (psia)>,<intrusion volume N (mL)>

APPENDIX F

USE OF THE MAXIMUM INTRUSION VOLUME OPTION

USE OF THE MAXIMUM INTRUSION VOLUME OPTION

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

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

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

APPENDIX G

BLANK AND SAMPLE COMPRESSION CORRECTIONS FOR MERCURY POROSIMETRY

BLANK AND SAMPLE COMPRESSION CORRECTIONS FOR MERCURY POROSIMETRY

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

Baseline Errors

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

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

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

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

Approaches to 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 weight 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.

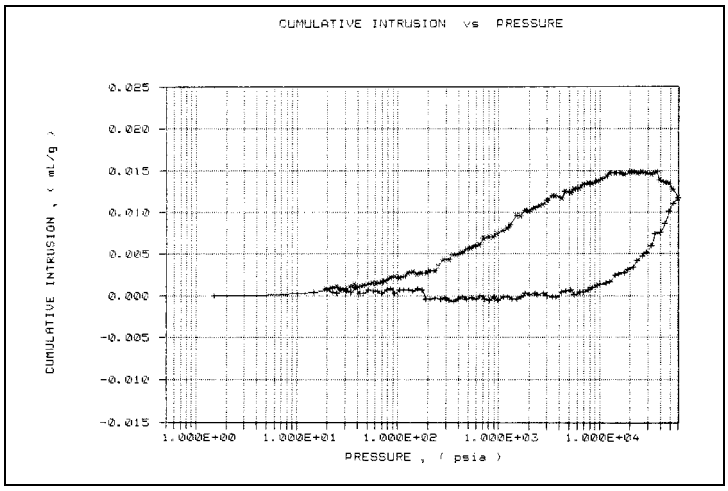


Figure 1

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 receded in the stem, giving the appearance of positive intrusion during depressurization.

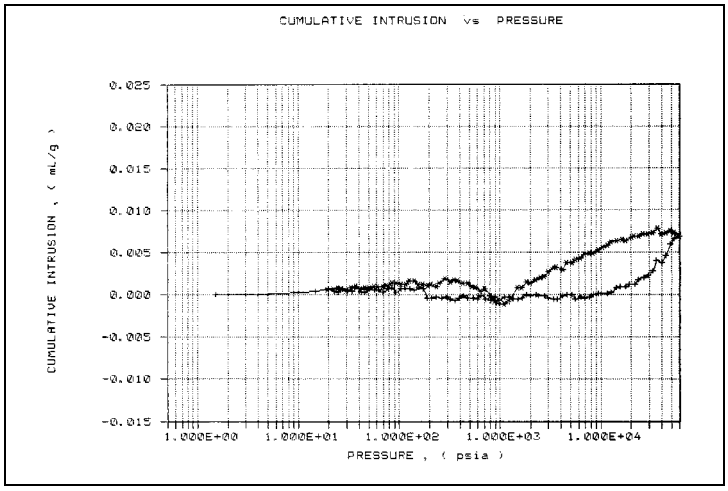


Figure 2

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.

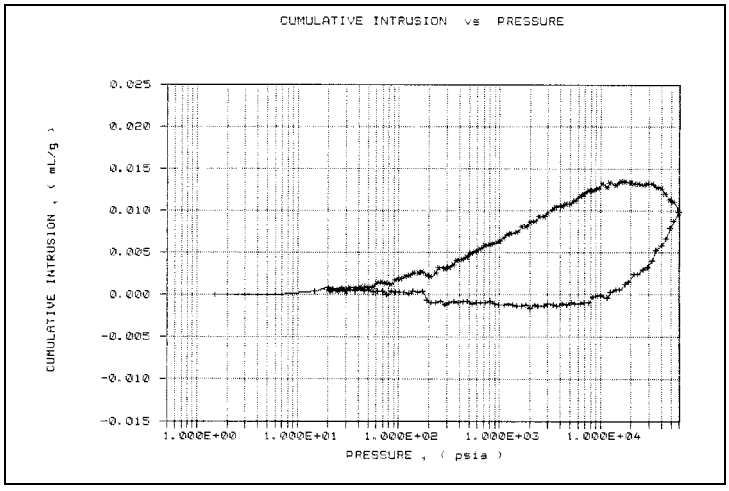


Figure 3

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

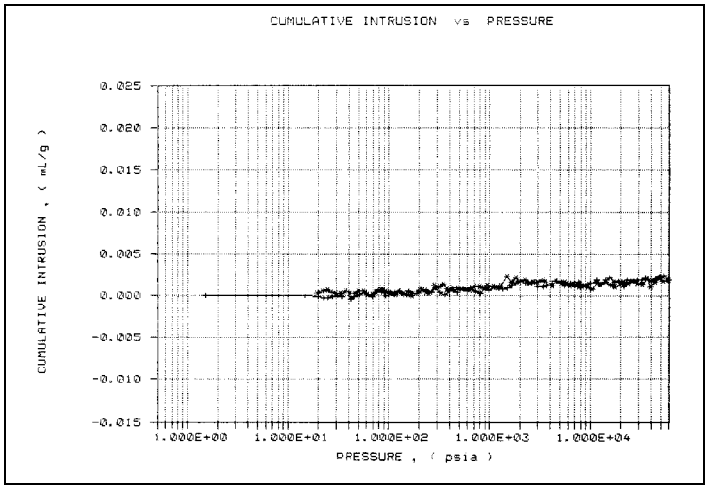


Figure 4

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

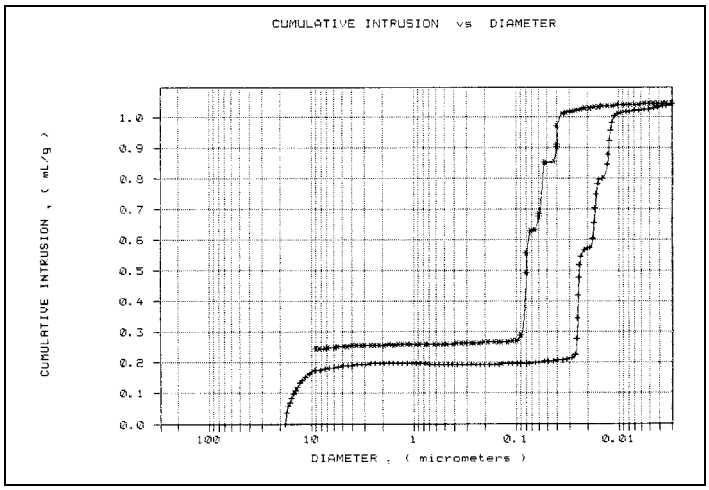


Figure 5

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

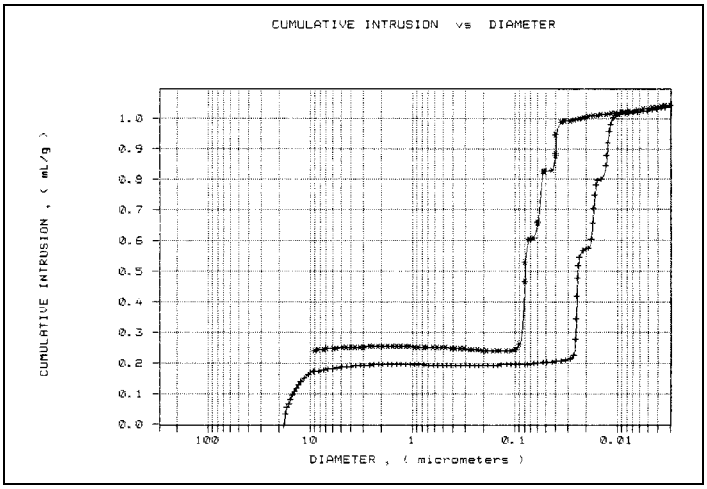


Figure 6

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

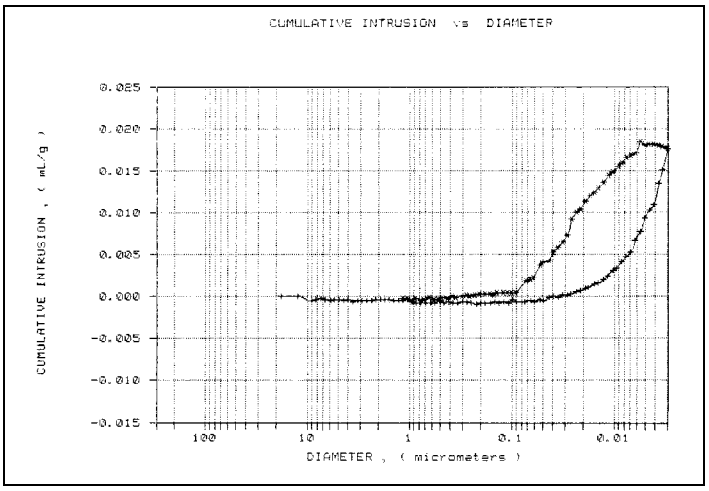


Figure 7

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

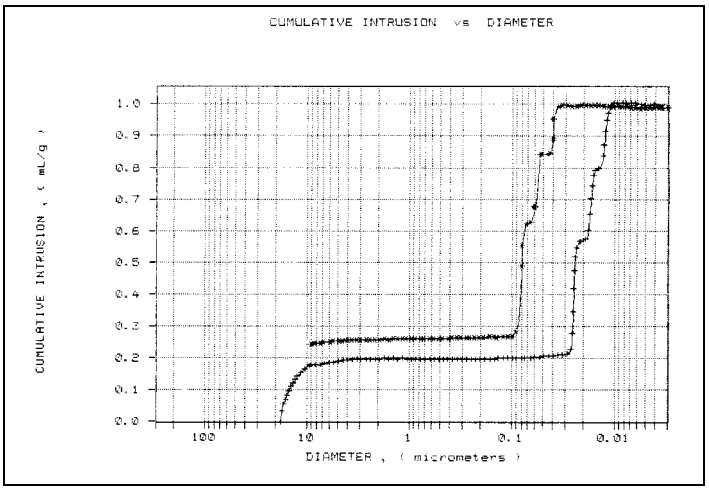


Figure 8

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

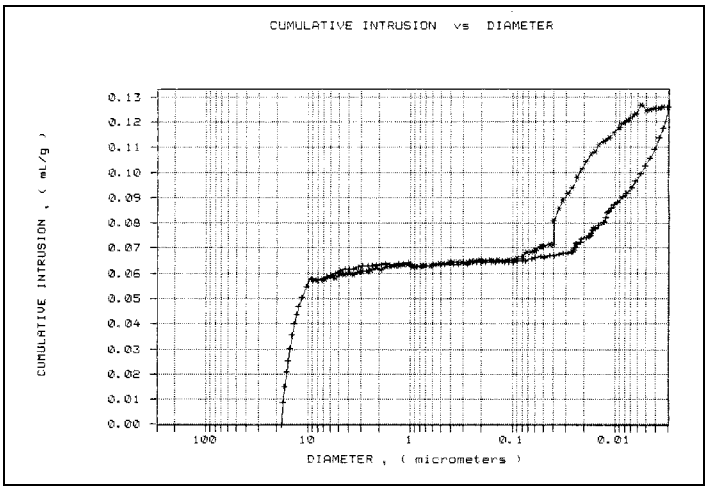


Figure 9

Uncorrected data from an essentially nonporous sample of the same type of glass shown in Figure 5. The weight of sample used was approximately equal to the weight 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.

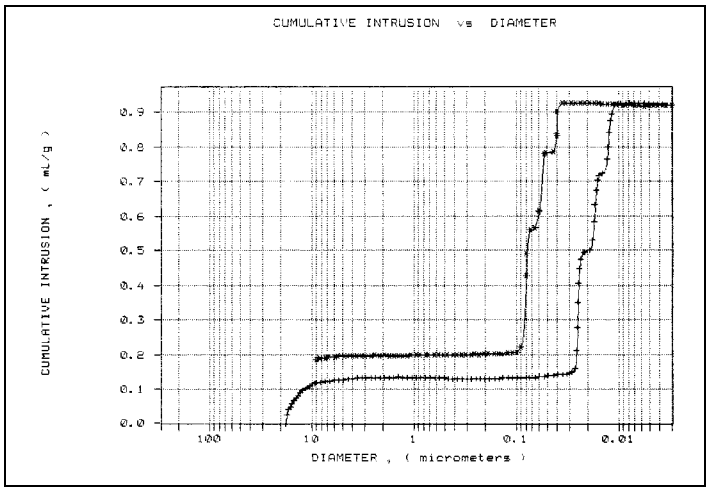


Figure 10

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.

APPENDIX H

PORE SURFACE AREA COMPUTATION

PORE SURFACE AREA COMPUTATION

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

Work

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

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

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

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

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

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

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

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

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

Cylindrical Geometry

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

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

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

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

Combining equations 5 and 6, yields

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

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

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

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

APPENDIX I

ERROR MESSAGES

ERROR MESSAGES

This appendix contains a numerical listing of the error messages associated with the AutoPore operating program.

2430- Error accessing file (file name), error code = (number).

Cause A: Diskette may be damaged.

Action A: Clean the diskette drive. If this does not eliminate the problem, attempt operation using a backup copy of the file.

Cause B: Hard disk may be damaged.

Action B: Contact your service representative.

Cause C: A software error occurred when the file was accessed.

Action C: Contact your service representative.

Cause D: The name specified contains one or more invalid characters.

Action D: Enter a valid filename; do not use characters such as * or ?.

2431- Error writing file (file name), error code = (number).

Cause: The hard disk does not have enough space left to perform the operation.

Action: Copy files not used regularly from the hard disk to diskette, delete them from the hard disk, and then try the operation again.

2432- Invalid response from MMI 'FILE_READ' request.

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

Action: Contact your service representative if you continue to receive this error message.

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

Cause: Several sample or parameter files have been added to this directory by some function other than the AutoPore program.

Action: Click **Yes** to update the directory information with data from each new file. This operation may take a minute.

Click **No** if you do not want to spend the time updating the directory information. This option may be feasible if a large number of files have been copied into the directory and you know the name of the file you wish to access.

2434- File (file name) - Subset # (number) wrote wrong amount of data.

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

Action: Contact your service representative if you continue to receive this error message.

2436- Path specification (path name) is invalid.

Cause: You entered an invalid path name and/or extension.

Action: Type a valid path name (including the proper extension) and press **Enter**.

2437- File (file name) does not exist.

Cause: You entered an invalid file name.

Action: Enter the name of an existing file or select a file name from the list box.

2438- Disk drive (letter): is inaccessible.

Cause: You selected a disk drive that is not presenly accessible.

Action: Ensure that the drive is not write protected.

2439- Could not register file.
2440- Subset not found.
2441- Seek within file failed.
2442- Had header in subset file.
2443- Subset owner denied access.
2444- Not a valid file format.
2445- Subset wrote the wrong amount of data.
2446- Error reading data.
2447- Error writing data.

Cause: An unexpected error occurred when you tried to access a data file.

Action: Contact your service representative.

2448- File directory (path name) is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid. The directory may have been deleted or moved to a different location.

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

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

Cause: You entered an invalid file name.

Action: See the description of file naming conventions in your DOS or Windows manual and reenter the name.

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

Cause: You are attempting to exit the application while the analyzer is performing a critical operation. This operation must be completed before the application can be exited.

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

2459- An instrument is busy. Continue with program Exit? (Yes, No)

Cause: You are attempting to exit the application while an analysis is in progress. While this is possible, the data collected while the application is inactive will not be permanently recorded until the application is restarted. A power failure to the instrument could cause some data to be lost.

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

2460- Fatal Communications error on (Unit n - S/N: nnnn)

Cause: There was a fatal error in the serial communications between the application and the analyzer. All displays for that analyzer will be closed.

Action: Check that the analyzer is connected to the computer on the communications port configured in the Setup program. Exit the analysis program and then restart it. If this error persists, contact your service representative.

2461- No active instruments. Application will stop.

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

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

Action B: Check the cable connection between the analyzer and the computer. Verify that the instrument has the power switch in the ON position and that the light on the front panel is illuminated. If the application continues to fail in its attempts to initialize the instrument, contact your service representative.

2468- The instrument contains an unknown software version. Do you want to reset it?

Cause: The application has discovered a different version of software operating in the instrument.

Action: If there is no chance that an instrument other than the AutoPore is connected to the computer, click **Yes** to reset the instrument and download the proper software. Otherwise, click **No** to leave the instrument unchanged.

2469- The instrument software did not initialize properly on Unit n - S/N: nnnn.

Cause: The analysis software failed to execute properly.

Action: Reinstall the software, then restart it. If the problem persists, contact your service representative.

2470- Unable to read instrument software file for Unit n - S/N: nnnn.

Cause: The application tried to read the instrument software file to download it to the instrument. It was unable to do so.

Action: Reinstall the instrument software, then restart it.

2477- Unit n - S/N: nnnn did not properly initialize.

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

Action: Correct the problem as described above, then restart the application.

2486- Could not construct (name) report type. Program will terminate.

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

Action: Contact your service representative if you continue to receive this error message.

2487- Could not start report generator. Error code (number). Program will terminate.

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

Action: Contact your service representative if you continue to receive this error message.

2488- File (file name) cannot be opened for editing. It is already in use.

Cause: The file you specified is already open for editing.

Action: Check the Windows list to locate the other edit session.

2489- File (file name) cannot be opened for writing. It is already in use.

Cause: The file you specified in a **Save As** operation is already open for edit.

Action: Select a different file for the **Save As** operation.

2490- No '.INI' file present. Application will terminate.

Cause: The ASCII file containing initialization information and system options information used during program startup does not exist.

Action: Run the Setup program (located on the applications CD), select **Change the number of units** and enter the pertinent information.

2492- This field's entry is INVALID.

Cause: The highlighted field contains an invalid entry.

Action: Check the entry and correct the error.

2493- An entry is REQUIRED for this field.

Cause: This field requires a valid entry for you to proceed.

Action: Enter or select an appropriate value.

2494- Value is out of the valid range.

Cause: The value you entered in the highlighted field is outside the valid range of values.

Action: Check the entry and enter or select an appropriate value.

2495- Value is out of the valid range. Enter a value between (value) and (value).

Cause: The value you entered in the highlighted field is outside the valid range of values.

Action: Check the entry and enter or select a value within the indicated range.

2496- Invalid number.

Cause: The number you entered in the highlighted field is invalid.

Action: Check the entry and enter or select a valid number.

2497- This field contains an invalid character.

Cause: You entered an invalid character in the highlighted field.

Action: Check the entry and enter valid characters.

2498- The requested change to the Sample's status is invalid at this time.

Cause: A request to change the file's status (for example, from automatically collected to manually entered) could not be done.

Action: Contact your service representative if you continue to receive this error message. Record the name of the sample file in which the problem occurred.

2499- Sequence number must contain at least 3 digits.

Cause: You tried to enter a sequence number that did not contain at least three digits.

Action: Enter a sequence number that contains at least three digits.

2500- All sample file names that can be created using the sequence number pattern already exist. You may want to modify the next sequence number.

Cause: No more sample information files can be created using the currently specified file sequence number.

Action: Select **Options**, **Sample defaults** from the Main menu and enter a new sequence number.

2501- System resources have reached a dangerously low level. Please close some windows to avoid the loss of data.

Cause: A large number of windows are open and consuming the system resources available to all applications.

Action: Close one or more windows on the screen. Contact your service representative if you continue to receive this error message.

2502- Error writing to file (name) during print. Error code: (number).

Cause: An error occurred in the file being written to during a print operation.

Action: Ensure that there is sufficient space on the drive containing the file.

2505- Error Logger cannot be initialized!! Error code (number). Program will exit.

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

Action: Contact your service representative.

2506- (sample file) Output device (name) is not installed. Printing cannot be accomplished.

Cause: The selected output device is not installed.

Action: Select a different output device in the System Configuration dialog box. Install the device using the Control Panel, Printers operation.

2507- Error opening file (name) for printing. Error code: (number).

Cause: An error occurred in the selected file for print output.

Action: Ensure that sufficient space is available on the drive containing the file.

2508- (sample file) Overlay file (name) was not found. It will not be included in the reports.

Cause: The specified overlay file could not be found.

Action: Ensure that the file specified as an overlay does exist.

2509- (sample file) Error opening file (name): (error). Reports cannot be produced.

Cause: An error occurred while the program was opening a file necessary to the report operation.

Action: Use the name given in the error message to investigate. Contact your service representative if you continue to receive this error message.

2510- (sample file) Error parsing reports from file (name). Reports cannot be produced.

Cause A: One or more data entry fields in the sample file may contain an invalid character (such as a single quote or double quotes).

Action A: Review the data entry fields (for example, the Sample field) and remove the invalid character.

Cause B: The system was unable to create the usual temporary files during the report, possibly due to insufficient disk space.

Action B: Check the space available on the hard disk.

Cause C: An internal processing error occurred.

Action C: Contact your service representative.

2511- Print job (name) has been canceled due to insufficient disk space. Delete unnecessary files and restart the report.

Cause: The disk drive does not have enough space for the temporary file required by the Windows Print Manager. Therefore, printing of the requested report has been canceled.

Action: Delete unnecessary files from the disk. You will require at least five megabytes of free space for normal operation.

2512- Print job (name) has been canceled.

Cause: The requested print job was canceled at your request.

Action: None required.

2521- Unable to program controller.

Cause: A hardware malfunction has occurred.

Action: Contact your local Micromeritics representative.

2522- Invalid controller application file.

Cause: The application’s control file has been corrupted or deleted.

Action: Reinstall the AutoPore analysis program.

2523- Programming controller failed.
2524- CRC check failed on programming controller.
2525- Unknown error programming controller.

Cause: A hardware malfunction has occurred.

Action: Contact your local Micromeritics representative.

2526- Controller download was not successful.

Cause: A communications problem between the computer and the analyzer has occurred.

Action A: Check the cable connection between the computer and the analyzer.

Action B: Access the Unit configuration dialog from the Unit menu and verify that the TCP/IP configuration for the computer and the analyzer are correct.

Action C: Exit the AutoPore application, and turn off the analyzer. Then turn on the analyze and restart the application. If the problem persists, contact your local Micomeritics representative.

2527- Controller CRC error on boot block.
2528- Controller DRAM error.
2529- Controller Com1: error.
2530- Controller Com2: error.
2531- Controller debug port error.

Cause: A hardware malfunction has occurred.

Action: Contact your local Micromeritics representative.

2532- The instrument contains an unknown software version. Do you want to reset it?

Cause: A different version of software is already operating on the instrument.

Action: Click **Yes** to reset the instrument and download the proper sofware. Click **No** to retain the current software version and cancel initialization.

2533- Mass initialization failed.

Cause: A hardware malfunction has occurred.

Action: Contact your local Micromeritics representative.

6000- An instrument is performing a critical operation. Wait a few moments before exiting the application.

Cause: You attempted to exit the AutoPore application while the instrument is performing a critical operation. This operation must be completed before the application can be stopped.

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

6001- An instrument is busy. Continue with program Exit? (yes, no)

Cause: You attempted to exit the AutoPore application while an analysis or calibration is in progress. While this is possible, the data collected while the AutoPore application is inactive will not be permanently recorded until the application is re-started. A power failure to the instrument could cause some data to be lost.

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

6002- No active instruments. Application will stop.

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

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

Action B: Check that the instrument is attached to the computer on the communications port configured with the Setup program. If the AutoPore continues to fail in its attempts to initialize the instrument, contact your Micromeritics Service Representative.

6003- Error accessing the sample information file <file name>. (Code <number>).

Cause: An error occurred while accessing the indicated file.

Action: Record the "code" reported in the message. Record the conditions under which this message was encountered and report the problem to your Micromeritics Service Representative.

6004- File <file name> cannot be analyzed. It is currently being edited.

Cause: You selected a file for analysis which is currently open for editing.

Action: Complete the editing of the named file. Save any changes and close the editing dialog. Repeat your attempt to start the analysis.

6006- An analysis cannot be performed on <file name>. It is open for editing and contains errors.

Cause: You selected a file that is currently being edited. Editing cannot be finished because the sample contains entry fields with errors.

Action: Correct the errors and finish editing the sample file. Then save, close the editing window, and try opening the file again.

6007- The edit session for <file name> must be saved before the analysis. Save changes and continue with the analysis?

Cause: You selected a file for analysis that is currently being edited. The changes to this file must be saved before it can be used in an analysis.

Action: Click **Yes** to save the changes and proceed with the analysis. Click **No** to review the changes before you save them.

6008- File cannot be opened for writing. It is already in use.

Cause: You attempted a **Save As** operation to a file which is already in use. The save could not be completed

Action: Wait until the selected file is no longer being used or select a new target name for the Save As operation.

6009- The instrument on unit <number> is not calibrated.

Cause: The AutoPore application is in the process of initializing the instrument and has noticed that no calibration information is available for that unit.

Action: Click **OK**. When the instrument has initialized, open the calibration screen and calibrate the instrument.

6010- Blank correction information not loaded from <file name>. An error occurred while accessing it.

Cause: You specified **Blank Sample** as the error correction method on the Penetrometer Properties dialog of a sample file. The file which you selected as containing the blank correction data can not be accessed.

Action: Verify that the named file is a valid AutoPore data file by opening it for editing. If the file is accessible and contains valid data, report this error to your Micromeritics Service Representative.

6011- Blank correction information not loaded from <file name>. It contains no valid data.

Cause: You specified **Blank Sample** as the error correction method on the Penetrometer Properties dialog of a sample file. The file which you selected as containing the blank correction data is empty.

Action: A sample file must contain valid data to be used for blank correction. Examine the reports of the indicated file and verify that it contains valid data.

6012- Negative intrusion/extrusion data were excluded from the stored blank correction data.

Cause: You specified **Blank Sample** as the error correction method on the Penetrometer Properties dialog of a sample file. The file which you selected as containing the blank correction data contains negative intrusion or extrusion data. This message informs you that this negative data will not be used for blank correction.

Action: If the negative data should be excluded (the usual case), no action is required. If you desire the negative data to be used for blank correction, open the 'blank' sample file for editing. Check the **Report negative intrusion** option in the Report Options dialog of the Advanced presentation.

6013- The pressure table for this sample was closed with a "Last low pressure index" of zero. Please note that, when analyzed, no Low Pressure data will be collected.

Cause: The Last Low Pressure index on the Pressure Table dialog indicates which pressures in the table are to be used for the low pressure analysis. If this index is zero, the penetrometer will only be filled with mercury, and no low pressure data collected.

Action: If you failed to enter a Last Low Pressure index by mistake, open the pressure table dialog and correct the value; otherwise, the message may be ignored.

6014- The pressure table for this sample has a "Last Low Pressure" of <pressure>. This may be less than atmosphere plus the maximum head pressure.

Cause: The pressure selected by the Last low pressure index on the Pressure Table window is less than 20.0 psia. This may be below the combined pressure of the atmosphere and the mercury in the stem when the penetrometer is mounted in a high pressure port. It may not be possible to match the low pressure analysis to the high pressure analysis.

Action: If no high pressure analysis is to be done on the sample, this advisory may be ignored. Otherwise, change the Last low pressure index on the Pressure Table window to select a pressure of 20.0 psia or greater. Pressures as high as 50.0 psia may be included in the low pressure analysis if threaded penetrometer closures are being used.

6015- Invalid configuration. Proceeding with the available ports.

Cause: The hardware configuration for this instrument is neither a 9520 nor a 9510. This will occur if one or more of the analysis ports are functioning improperly.

Action: Make sure that all the capacitance detector connections are plugged into the instrument, and restart the AutoPore application. If the message persists, the instrument may be operated without all of the analysis ports. Contact your Micromeritics Service Representative to arrange for the repair of the non-functioning ports.

6016- Unit <number>: Low pressure analysis canceled. Mercury has overfilled.

Cause: During the execution of the low pressure analysis, the mercury reservoir overfilled. The analysis cannot continue and was canceled. The data already collected are stored in the sample file.

Action: Click **OK** to acknowledge the message. Consult your Micromeritics Service Representative to correct the overfill condition.

6017- Unit <number>: Low pressure analysis canceled. Mercury failed to drain.

Cause: During the execution of the low pressure analysis, the mercury did not drain. The analysis cannot continue and was canceled. The data already collected are stored in the sample file.

Action: Click **OK** to acknowledge the message. Consult your Micromeritics Service Representative.

6018- Unit <number>: High pressure analysis canceled. The intensifier has reached its upper limit.

Cause: During the course of a high pressure analysis, the intensifier reached its upper limit switch without achieving the desired pressure. The analysis cannot continue and was canceled. All data collected prior to this problem are stored in the sample file.

Action: Usually this error only occurs if the vent valve on the top of the pressure chamber was left open or did not seal properly. If this is not the case, it may indicate a problem with the equipment. Click **OK** to acknowledge the message.

6019- Unit <number>: High pressure analysis canceled. The intensifier has reached its lower limit.

Cause: During the course of a high pressure analysis, the intensifier reached its lower limit switch without achieving the desired pressure. The analysis cannot continue and was canceled. All data collected prior to this problem are stored in the data file.

Action: Usually this error occurs if very low pressures (below 30 psia) are requested during extrusion. Because of the heat generated during pressurization, it may not be possible to de-pressurize the sample below 30 psia. If the problem occurs at reasonable extrusion pressures, the instrument may require calibration. Alternatively, the vent valve on the pressure vessel may not have been opened to atmosphere when the analysis was started. Click **OK** to acknowledge the message.

6020- Unit <number>: High pressure analysis canceled. An invalid target pressure (<number> psia) was requested.

Cause: There is an invalid pressure in the pressure table for the analysis. The analysis was canceled. A pressure is invalid for a low pressure analysis if it exceeds 30 psia and the **Threaded Penetrometers** option is not checked on the Options menu. A pressure is invalid for a high pressure analysis if it exceeds 61000 psia. Under normal operation you will be warned that a pressure is invalid during the entry of the pressure table or during analysis startup.

Action: Click **OK** to acknowledge the message. Edit the pressure table to remove the invalid pressure, and restart the analysis.

6021- Unit <number>: Low/High pressure analysis can not be suspended. It is performing a critical operation.

Cause: You attempted to suspend a low/high pressure analysis while the instrument is in a potentially dangerous condition. The request has been ignored.

Action: Wait a short time for the critical operation to be completed and then try again.

6023- Unit <number>: Pump command IGNORED. The pump is ON. It must be turned OFF for five seconds before changing its direction.

Cause: You attempted to change the direction of the high pressure pump while it was operating. The pump must be OFF for five seconds before attempting to change its direction.

Action: Turn the pump off and wait five seconds. Repeat the command to change the pump's direction.

6024- Unit <number>: Pump command IGNORED. The pump has not been OFF for five seconds before changing its direction.

Cause: You attempted to change the direction of the high pressure pump before it had come to a complete stop. The pump must be off for five seconds before attempting to change its direction.

Action: Wait five seconds and repeat the command to change the pump's direction.

6025- Unit <number>: The pump's voltage can not be increased while it is ON and the system is de-pressurizing above <number> psia.

Cause: An attempt was made to manually increase the pump voltage while the high pressure system was de-pressurizing above the given pressure.

Action: Allow the system to reach a pressure below the one shown, then increase the voltage.

6026- Unit <number>: The pump cannot be turned ON while the pump direction is down, the pressure is above 32,000 psia, and the voltage is above 10 Volts.

Cause: You tried to turn the pump on manually while its voltage was above 10 volts.

Action: First decrease the voltage below 10 volts, then turn the pump on.

6027- Unit <number>: Verify that vent valves are CLOSED.

Cause: The high pressure analysis on unit <number> is about to begin pressurizing the sample. The vent valves must be closed.

Action: Close the vent valves and choose **OK**. If for any reason you do not wish the high pressure analysis to proceed, click **Cancel** to abort the analysis.

6028- Unit <number>: OPEN the vent valves so the transducer offset can be re-calculated.

Cause: The high pressure analysis is at a stage where it requires the high pressure system to be at atmospheric pressure.

Action: Open the vent valves, and expose them to atmospheric pressure, then click **OK**. If something is wrong, click **Cancel** to abort the analysis.

6029- Unit <number>: Analysis canceled. The mercury reservoir is low.

Cause: During the execution of the low pressure analysis, the level of mercury in the reservoir dropped below the acceptable level. The analysis cannot continue and was canceled. Any data already collected are stored in the sample file.

Action: Click **OK** to acknowledge the message. Consult the relevant section of the manual for instructions on refilling the mercury reservoir.

6030- Unit <number>: Received calibration information while a low pressure analysis was in progress.

Cause: You tried to send calibration information to the interface controller while a low pressure run was in progress.

Action: Wait until the low pressure analysis has finished, then try sending it again.

6031- Unit <number>: Received calibration information while a high pressure analysis was in progress

Cause: You tried to send calibration information to the analyzer while a high pressure run was in progress.

Action: Wait until the high pressure analysis has finished, then try sending it again.

6032- <Unit n> The pressure is too high for this instrument.

Cause: During a manual control operation, you requested the instrument to perform an operation that would exceed the highest recommended pressure. For safety concerns, the pump automatically turned off, resulting in cancellation of any analysis which might have been in progress.

Action: Do not attempt to overpressurize the system.

6033- <Unit n> Unable to reach pressure.

Cause: A hardware failure has occurred.

Action: Contact your Micromeritics Service Representative.

Cause: A power failure has occurred.

Action: Wait until the power is restored and try the operation again.

6034- Conversion has not finished, close anyway?

Cause: You tried to close the conversion window, but there are still conversions remaining to be completed.

Action: Click **Yes** to abort those conversions and close the window, or **No** to leave the window alone.

6035- Cannot read source file “<file name>”.

Cause: The file selected for conversion could not be read.

Action: Select a valid file, and try the conversion again.

6036- Destination file (“<file name>”) already exists.

Cause: The destination file for the conversion already exists, and will not be overwritten.

Action: Select an nonexisting destination, and try the conversion again.

6037- Unable to create destination file (“<file name>”).

Cause: The conversion routine was unable to write to the converted file.

Action: Select a valid destination file name and try again.

6038- Cannot load pressure table for file <file name>.

Cause: An error occurred in converting a sample file from the old (non-Windows) format to the Windows format. The pressure table file required to convert the specified file was not found.

Action: Place the required pressure table in the system directory specified on the conversion dialog and try the conversion again.

6039- Cannot find blank correction file for (file name).

Cause: An error occurred in converting a sample file from the old (non-Windows) format to the Windows format. The blank correction file for the given sample was not found during a conversion from the old format to the Windows file format.

Action: Place the blank file in either in the source file’s directory or in the system directory and try again.

6040- Cannot read system defaults for (file name).

Cause: An error occurred in converting a sample file from the old (non-Windows) format to the Windows format. The 9520 System Defaults file is required to convert a sample file to the Windows file format. This file could not be found.

Action: Move the SYSPRM.DAT file to the directory specified as the System Directory on the conversion dialog and try again.

6041- Cannot read Report Table for (file name).

Cause: An error occurred in converting a sample file from the old (non-Windows) format to the Windows format. A tabular data set file is required to convert the named sample file to the windows file format. This data set could not be found.

Action: Make sure that the required tabular data file is in the System Directory selected on the file conversions dialog and try again.

6043- Error opening file <file name> for report preparation.

Cause: An error occurred while attempting to open the specified file for report generation.

Action: Ensure that the specified file is a valid AutoPore sample file by opening it for editing. If the file may be edited and contains valid collected data, report this problem to your Micromeritics Service Representative.

6044- Error preparing data for Tabular Data Set tabular report.

Cause: A tabular report was requested with the Tabular Data Set option enabled. An internal error occurred while generating the report.

Action: Open the sample file being reported. Edit the Tabular Report Options and select Collected Data as the reporting mode. Report this error to your Micromeritics Service Representative.

6045- Error preparing data for Tabular Data Set tabular report. Tabular Data Set may be empty.

Cause: A tabular report was requested with the Tabular Data Set option enabled. No pressure/pore size entries, however, were specified in the table.

Action: Open the sample file being reported. Edit the Tabular Data Set dialog under the Tabular Report Options and insert a list of the pressures or pores sizes which you would like reported.

6046- A Tabular Data Set was specified for the tabular report. None of the collected data is within the bounds of the table.

Cause: A tabular report was requested. The Tabular Data Set option was selected and a table of pressures or pore sizes to be reported was entered. None of the collected data in the sample file, however, falls within the range of pressures or pore sizes specified.

Action: Either correct the tabular data set to match the collected data or select the Collected Data option on the Tabular Report Options dialog.

6060- Instrument [unit no.] is not calibrated.

Cause: The AutoPore application is in the process of initializing the instrument and has noticed that no calibration information is available for that unit.

Action: Click **OK**. When the instrument has initialized, open the calibration screen and calibrate the instrument.

6061- Low pressure servo calibration failed.

Cause A: The maximum time was exceeded before the target pressure point was reached. The nitrogen regulator may be set too low or turned off.

Action A: Set the analysis gas regulator to 10 psig (0.7 bar), then resume the analysis.

Cause B: The analysis gas bottle is empty.

Action B: Connect a new analysis gas bottle, then resume the analysis.

6064- High pressure system is overpressurized.

Cause: The high pressure system was instructed to increase the pressure when it is already at too great a pressure.

Action: Reduce the pressure in the high pressure system. If this error was part of an automatic analysis, the pressure will be reduced automatically by returning the intensifier to the lower limit switch.

6065- Intensifier reached upper limit switch.

Cause: During the course of a high pressure analysis, the intensifier reached its upper limit switch without achieving the desired pressure. The analysis cannot continue and was canceled. All data collected prior to this problem are stored in the sample file.

Action: Usually this error only occurs if the vent valve on the top of the pressure chamber was left open or did not seal properly. If this is not the case, it may indicate a problem with the equipment. Click **OK** to acknowledge the message.

6066- Intensifier reached lower limit switch.

Cause: During the course of a high pressure analysis, the intensifier reached its lower limit switch without achieving the desired pressure. The analysis cannot continue and was canceled. All data collected prior to this problem are stored in the data file.

Action: Usually this error occurs if very low pressures (below 30 psia) are requested during extrusion. Because of the heat generated during pressurization, it may not be possible to de-pressurize the sample below 30 psia. If the problem occurs at reasonable extrusion pressures, the instrument may require calibration. Alternatively, the vent valve on the pressure vessel may not have been opened to atmosphere when the analysis was started. Click **OK** to acknowledge the message.

6067- High pressure system pump has overheated.

Cause: The high pressure pump has been on too long at a high power, possibly due to extensive cycling at very high pressures.

Action: Allow the pump to cool and return it to the lower limit switch. Avoid allowing the pump to operate for extended periods at high power.

6068- Low pressure analysis canceled. Mercury has overfilled.

Cause: During the execution of the low pressure analysis, the mercury reservoir overfilled. The analysis cannot continue and was canceled. The data already collected is stored in the sample file.

Action: Click **OK** to acknowledge the message. Consult your Micromeritics Service Representative to correct the overfill condition.

6069- Low pressure analysis canceled. Mercury failed to drain.

Cause: During the execution of the low pressure analysis, the mercury did not drain. The analysis cannot continue and was canceled. The data already collected are stored in the sample file.

Action: Click **OK** to acknowledge the message. Consult your Micromeritics Service Representative.

6070- Low pressure analysis canceled. No mercury available.

Cause: You attempted to start a low-pressure analysis, but there was not enough mercury to complete it.

Action: Add mercury to the system and try again.

6071- Warning, the instrument is not calibrated.

Cause: The AutoPore application is in the process of initializing the instrument and has noticed that no calibration information is available for that unit.

Action: Click **OK**. When the instrument has initialized, open the calibration screen and calibrate the instrument.

6072- Warning, not all samples have compatible analysis conditions.

Cause: You attempted to start multiple analyses, but not all analysis conditions are compatible.

Action: Edit analysis conditions so that they are compatible.

6073- You must select sample files for both ports to do a differential analysis.

Cause: You specified a differential high pressure analysis, but chose only one sample file.

Action: A differential analysis requires a sample file for each port; choose another sample file for the other port. Or change the current analysis to a standard one.

6074- (Unit n) Warning these analysis conditions contain pressures that are too large for the high pressure system of this instrument. Do you want to proceed without the high pressures?

Cause: You have a 9500 or 9505 installed and have selected a sample file for a high pressure analysis. This sample file contains pressures which exceed 33,000 psia, the maximum pressure for this instrument.

Action A: Click **Yes** to proceed with the analysis; only pressures below 33,000 will be used.

Action B: Click **No** to cancel the analysis on this instrument and restart it using the 9520 or 9510 which can handle pressures exceeding 33,000 psia. Or return to the analysis conditions screen and edit analysis conditions.

6075- Instrument (Unit n): The calibration is for a diffrent type of instrument and has been reset.

Cause: The calibration stored with this instrument is for a different type of instrument.

Action A: Make sure the correct instrument is installed.

Action B: Properly calibrate the instrument.

6076- Low pressure analysis canceled. Evacuation timeout.

Cause: The pressure did not drop to 7 psia in a reasonable amount of time during a low pressure evacuation.

Action: Ensure that the penetrometers are installed properly and that there is no leak in the system; then try again.

6077- File (filename) contains no data.

Cause A: You requested a report on a sample file that is currently being used with an analysis (*Analyzing* status) and has not collected any data.

Action A: Wait until some data are collected and request the report again.

Cause B: You requested a report on a sample file that has not been used with an analysis (*No analysis* status).

Action B: Perform an analysis using the sample file, wait until data are collected, and request the report again.

APPENDIX J

CONVERTING NONWINDOWS SAMPLE FILES TO WINDOWS-COMPATIBLE FILES

CONVERTING NONWINDOWS SAMPLE FILES TO WINDOWS-COMPATIBLE FILES

You can convert sample files created with the nonWindows version of the AutoPore to sample files compatible with Windows. Each sample file requires three system files from the system directory, the:

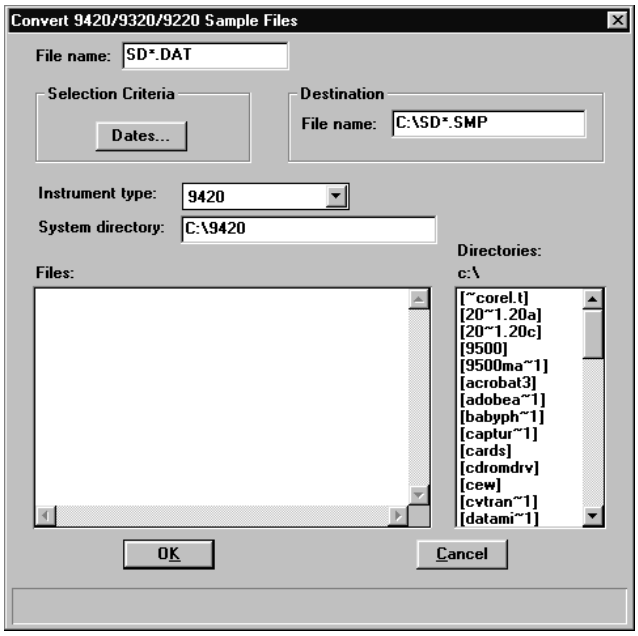
- system data file, **sysprm.dat**
- pressure point data file, **pt**<table number>**.dat**
- tabular report data file, **td**<table number>**.dat** (if requested in the sample file)

If you are converting files using the same computer on which the files were created, all required files will be located automatically during conversion.

If you plan to use a different computer for converting the files, it is *imperative* that all the required files for each sample file be copied. If you are converting files one at a time on an as-needed basis, it is not necessary to copy the system data file (sysprm.dat) but once. It is best to zip the entire directory from the source computer and then unzip on the destination computer, especially if you have numerous files to convert. This way, you are ensured that all required files accompany the sample file.

Perform the following steps to convert files:

1. From the Main Menu, select **File**, **Convert...** ; the Convert 9420/9320/9220 Sample Files dialog box is displayed.



2. Navigate to the appropriate directory and select the file you wish to convert.

3. Select the type of instrument on which the file was created.

4. Select the system directory where the files of the previous instrument were located; then **OK**.

After the file conversion process, a list is displayed showing the status of the file conversion. This display tells you whether the conversion was successful or not. If not, possible reasons are displayed.

Because files have been restructured for the Windows interface, the conversion process does not always convert files as they are structured for other instruments.

It may be helpful to know that for sample files using blank correction, the *data* stored in the blank correction file is copied to the sample file (not just the file name). This way, if either the sample file or the correction file is moved, the original blank correction data will be available in the sample file. This provides for repeatable results for reprinted reports, comparisons with samples run later, or overlays. If, however, you wish to change the blank correction file associated with a sample file, you may still do so by changing the blank correction file name, then choosing the new name in the sample file.

If the pressure table and/or tabular data set are needed for the file conversion, the data from the old files are read into the correct locations in the new sample file; the files themselves are not converted.

APPENDIX K

CHOOSING THE PROPER PUMP-DOWN RATES FOR UNFAMILIAR SAMPLE MATERIALS

CHOOSING THE PROPER PUMP-DOWN RATES FOR UNFAMILIAR SAMPLE MATERIALS

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

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

- a fine dust is raised upon stirring or shaking and the sample shows little sign of quickly settling
- the sample is a finely powdered organic material
- the sample is known to be microporous or mesoporous with pores of less than 100 Angstrom width
- the sample is known or suspected to be a carbon
- the sample is known or suspected to be a zeolite
- the sample is known or suspected to be a fluid cracking catalyst
- the sample has significant fine particle content below 10 μm
- 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 μmHg 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 μmHg 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 μmHg.

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 pumpdown rates and pressures of path changeovers are reasonable trial increases.

Monitor closely during all portions of the pumpdown 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.

APPENDIX L

COMPUTING VOLUMETRIC COMPRESSIBILITY OF A SAMPLE MATERIAL

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

The sample weight and sample density must be known and available to a resolution and accuracy at least three significant digits (preferably better) to permit accurate computation of the initial volume of the sample material. Alternatively, an accurate geometric volume of a material such as one containing closed pores (such as plastic foam) may be supplied.

Before data reduction can be performed, you must have available a “blank run” file consisting (at least ideally) of a run made with the same penetrometer and accessory hardware that is to be used in the compressibility test and (again ideally) on the same instrument ports as will be used in the compressibility run. The pressure range of the blank run must, at a minimum, fully encompass the planned range to be used in the compressibility measurement and have a minimum of seven uniformly spaced (linear basis) data points inside the planned computation range and with the beginning and ending data points within 5% (pressure) of the planned computation range end points. It is also permissible for the “blank run” to consist of a manually entered data file.

The first and second order isostatic pressure coefficients of volumetric compressibility for mercury over the pressure range from zero psia to 60,000 psia must be known and available.

All standard input information such as sample material identity, equilibration times, evacuation information, penetrometer constants, etc. that would be required for standard runs is required for a compressibility run. Note that sample volume, bulk volume/density, and skeletal volume/density as measured during the mercury porosimetry run are, in general, far too imprecise to yield good results if used in the compressibility computations. For this reason, you must enter very accurate material density and sample weight 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%.

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