PoreSizer 9320

Operator's Manual
V2.06

Part No. 932-42801-01
11 November 1994

WARRANTY

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

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

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

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

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

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

1. Certain products sold by MICROMERITICS are the products of reputable manufacturers, sold under their respective brand names or trade names. We, therefore, make no express or implied warranty as to such products. We shall use our best efforts to obtain from the manufacturer, in accordance with his customary practice, the repair or replacement of such of his products that may prove defective in workmanship or materials. Service charges made by such manufacturer are the responsibility of the ultimate purchaser. This states our entire liability in respect to such products, except as an authorized person of MICROMERITICS may otherwise agree to in writing.

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

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

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

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

One Micromeritics Drive • Norcross, GA 30093-1877 • Telex: 682-7018

Domestic Sales — (404) 662-3633
International Sales — (404) 662-3660
Domestic Repair Service — (404) 662-3666
Customer Service — (404) 662-3636

Form No. 008-42104-00
28 Oct 1988

PRECAUTIONS

Generation of hydraulic **pressures in excess of 30,000 psi (206.8 MPa) should never be attempted** even though the PoreSizer 9320 is designed with extra capacity. Critical components are tested to 45,000 psi. An automatic shutdown system is set to operate just beyond 30,000 psi, and, as a further protection, a rupture disk with capacity only slightly greater is built into the system.

A latching switch is provided for operator convenience in making pressure increases in large increments. The **instrument should never be left unattended when this switch is on.** The automatic shutdown should prevent excess pressure being developed but, if it should not function, the rupture disk will give way. Opening the rupture disk will necessitate clean up as well as disk replacement.

Opening the high pressure chamber under pressure is usually precluded by the difficulty of doing so. However, the vent valve on top of the pressure chamber closure can be opened at relatively low pressure and with some difficulty at high pressure. Doing so will result in a vertical discharge of hydraulic fluid with considerable force. **This valve should be opened only after** chamber pressure is reduced to atmospheric pressure.

Mercury vapor is considered potentially harmful if allowed to accumulate in work spaces. The PoreSizer 9320 is provided with an exhaust fan that draws air across the work space and from within its cabinet. A duct leading to an exhaust system or directly outside **should be attached as provided** for on the instrument. Any mercury spilled on the tray of the PoreSizer should be wiped to the hole in the center of the tray from which it will fall into a collector and be covered by a layer of oil. The exhaust from the vacuum pump can contain mercury vapor. Provision should be made whereby this exhaust is discharged from the work area either through the ventilation system of the PoreSizer 9320 or separately. Mercury vapor levels well below the accepted safe level will easily be achieved if these directions are followed and comparable cleanliness is maintained elsewhere.

Removing the back panel exposes connections carrying potentials as great as 230V in some models. The **instrument should be unplugged** from the electric supply before proceeding.

Materials suitable for testing in the PoreSizer **must have a low vapor pressure** at room temperature. Meaningful results are not likely to be obtained with a volatile material.

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

Mercury amalgamates with the noble metals, hence these are generally not evaluated by mercury porosimetry. However, **some materials ordinarily considered reactive with mercury,** notably copper and aluminum, frequently have an oxide surface layer that renders them sufficiently resistive for testing.

The fluted knob through which each penetrometer is inserted into its preparation station **should always be left securely tightened** until the low pressure operation is completed. Tightening initially is necessary to achieve a satisfactory vacuum condition. Leaving it tight and leaving the the capacitance detector in place until the low pressure test is finished prevents the possibility of the penetrometer being expelled and broken during that phase of the operation when the pressure on the penetrometer is raised to as high as 30 psia.

Should a penetrometer be broken and mercury spilled in a high pressure chamber, the glass and mercury should immediately be removed. An aspirating or suction tube with a catch bottle preceding the source of the suction constitutes a suitable device for picking up the mercury. A gold-plated banana plug near the bottom of the chamber provides electrical contact, and, of course, gold amalgamates with mercury. The banana plug will be coated with oil and, in this case, amalgamation is likely to proceed only after an interval of time. Amalgamation may be accelerated by high pressure, however. **Immediately clean any mercury or other debris which collects in the high pressure chamber.**

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 immediately be drained.** If, instead, more mercury is added and the error persists, subsequent quantities of mercury cannot be retained and may result in damage to the vacuum pump and other components.

The mercury supply reservoir located beside the high pressure chamber is sealed by a black stopper cap. **The cap must always be securely in place during a test**; the run cannot be completed otherwise.

The two capacitance detectors used in the preparation and low pressure testing of samples are calibrated according to position of attachment. These positions are not interchangeable although the detector units are removable. **Always see that the detector marked LEFT is attached at the leftmost socket and the one marked RIGHT is attached at the rightmost socket.**

Table of Contents

1. GENERAL DESCRIPTION

2. INSTALLATION

3. GENERAL OPERATING INSTRUCTIONS

4. PERFORMING AN ANALYSIS

5. PRINTING REPORTS

6. ENTERING AND MAINTAINING ANALYSIS INFORMATION

7. USING THE ANALYSIS PROGRAM UTILITIES

8. TROUBLESHOOTING AND MAINTENANCE

9. ORDERING INFORMATION

10. ERROR MESSAGES

APPENDICES

CHAPTER 1

GENERAL DESCRIPTION

- Organization of the Manual
- Equipment Description

GENERAL DESCRIPTION

ORGANIZATION OF THE MANUAL

This manual describes how to install, operate, and maintain the PoreSizer 9320 Mercury Porosimeter. The manual is divided into ten chapters.

Chapter 1	Provides a general description and specifications of the PoreSizer.
Chapter 2	Provides unpacking and inspection information, and installation instructions.
Chapter 3	Provides general operating instructions.
Chapter 4	Provides instructions for performing low pressure and high pressure analyses.
Chapter 5	Describes how to print reports.
Chapter 6	Describes how to enter and maintain Analysis Program information. It includes sample screens and field descriptions for each function in the Analysis Program.
Chapter 7	Describes how to use the Analysis Program utilities to perform tasks such as formatting diskettes and backing up system files.
Chapter 8	Provides user maintenance and troubleshooting information.
Chapter 9	Provides ordering information for the PoreSizer system components.
Chapter 10	Describes Analysis Program error messages.

Several appendices are also included.

Important information concerning the prevention of bodily injury and the protection of equipment is contained in this manual in the form of WARNINGS and CAUTIONS, whereby:

WARNING
Designates information concerning the possibility of bodily injury.

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

EQUIPMENT DESCRIPTION

The PoreSizer 9320 is a 30,000 psia (207 MPa) mercury porosimeter covering the pore diameter range from approximately 360 to 0.006 µm. The unit has two built-in low pressure ports and one high pressure chamber. Data collection, data reduction, and data display are processed by the optional control module. All aspects of the high pressure analysis are also managed by the control module.

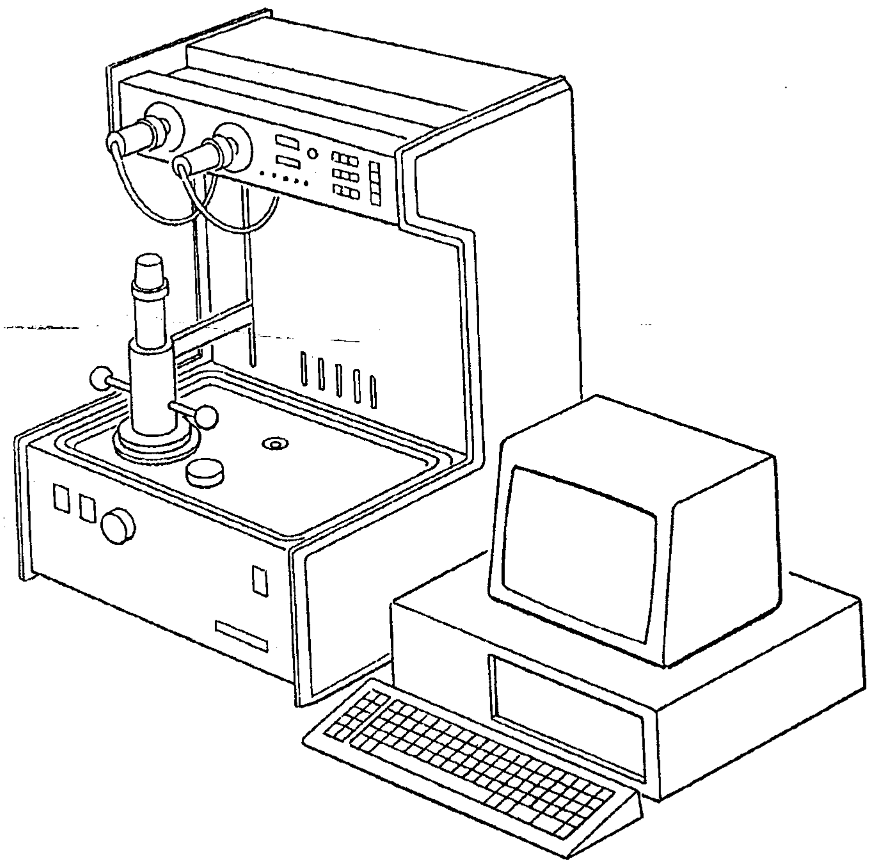


Figure 1-1. PoreSizer 9320 System

The PoreSizer measures the volume distribution of pores in materials by mercury intrusion or extrusion. Mercury has a high surface tension and is non-wetting to all materials with the exception of a few noble metals. These properties cause a mercury surface in contact with a solid to assume the minimum surface area and largest radius of curvature possible at a given pressure. An increase in pressure on the mercury shifts the balance between surface tension and surface area causing the radius of curvature of the mercury contacting the solid to become smaller. When the radius is equal to that of a pore entrance, mercury fills the volume within the pore. A thorough discussion of the theory of porosimetry is given in Appendix B.

Table 1-1. PoreSizer 9320 Specifications

Characteristic	Specification
	—— LOW PRESSURE ——
Low Pressure Measurement	0 to 30 psia (0.207 MPa)
Resolution	±1.67 X 10^{-4} psi
Pore Diameter	360 - 6 µm
Transducer Accuracy	±1% of full scale
	—— HIGH PRESSURE ——
High Pressure Measurement	0 to 30,000 psia (207 MPa)
Resolution	±0.167 psi
Pore Diameter	6 - 0.006 µm
Transducer Accuracy	±1% of full scale or ±2% of reading, whichever is lower
	—— INTRUSION ——
Intrusion Resolution	< 0.1 µL
Intrusion Accuracy	±1% of maximum penetrometer volume
Intrusion Rates	Four intrusion speeds and four extrusion speeds
	—— PENETROMETERS ——
Total Pore Capacity of Penetrometers	
Intrusion Volume	0.38, 1.1, 1.8, 3.1 or 4.0 cc
Accuracy	±1% of capacity
Sample Size	Maximum: a cylinder 2.5 cm in diameter and 2.5 cm long
	—— MEASUREMENT MODES ——
2 Measurement Modes	Scanning or equilibration using either a pressure table or a maximum intrusion increment or both

Table 1-1. PoreSizer 9320 Specifications (continued)

Characteristic	Specification
	POWER REQUIREMENTS
Voltage	100/120 or 220/240 VAC ±10%
Frequency	50/60 Hz
Power	2.5A (100/120 VAC); 1.25A (220/240 VAC)
	PHYSICAL
Height	79 cm (31 in.)
Width	58 cm (23 in.)
Depth	55 cm (21.6 in.)
Weight	86 Kg (190 lbs)
	CONTROL MODULE
Components	Processing unit High-density graphics video monitor Graphics printer Keyboard Plotter (optional)

CHAPTER 2

INSTALLATION

- Unpacking and Inspection
- Selecting the Location
- Additional Equipment Required
- Equipment Setup and Checkout
- Verifying Operation

INSTALLATION

This chapter describes how to unpack and inspect the equipment, how to install the PoreSizer 9320 System, and how to verify operation of the system.

UNPACKING AND INSPECTION

When you receive 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.

NOTE

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, please follow the steps below:

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

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

3. 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. Complete a Return Material Authorization Form (included in Appendix A); be sure to include the RMA number assigned by Micromeritics.

SELECTING THE LOCATION

A sturdy workbench 80 cm (30 in.) high near an exhaust hood or otherwise accessible to external ventilation is the recommended location for the PoreSizer. A square meter or so of free space on one side and a few centimeters to the rear of the instrument should be provided for working space. An equal space on the other side must be reserved for the control module and printer if these accessories are present. An additional 50 cm (20 in.) wide by 35 cm (14 in.) deep space must be provided for the Model 7440A plotter if purchased. For the Model 7550A plotter, a 69 cm (27 in.) wide by 69 cm (27 in.) deep space is needed. A nearby sink will make cleaning the penetrometer after analyses easier.

ADDITIONAL EQUIPMENT REQUIRED

In addition to the components shipped with the PoreSizer, a vacuum pump is required. Also, air pressure up to 20-25 psig (35-40 psia, 0.241-0.275 MPa) may be desired.

VACUUM PUMP

An ordinary, oil-sealed, laboratory vacuum pump capable of producing an ultimate vacuum of 1 x $10E^{-3}$ μmHg is required. The pump must be of the type which does not permit "suckback" of oil into the evacuated space. Usually, a two-stage mechanical pump with an internal solenoid or check valve is adequate. The shorter tube of the accessory vacuum trap is attached to the vacuum pump inlet with 1 cm (7/16 in.) ID vacuum hose (rubber, heavy-wall, preferably). Both end connections should be secured with clamps to provide reliable vacuum-tight seals. Locating the vacuum pump near the PoreSizer permits using a short length of hose and gives the fastest pump-down, but a longer length can be used if necessary.

AIR PRESSURE

Air pressure can be obtained from bottled, dry air, a small air compressor, or with a squeeze bulb. If bottled air is chosen, the tank should be fitted with a regulator set no higher than 25 psig and, for convenience, a manual outlet valve and a manual release valve. Whatever the choice, attachment is made to the labeled port at the lower rear of the instrument. Hose having an inside diameter of 5 mm (3/16 in.) is required.

The intake air should be passed through a drying cartridge if a small compressor or squeeze bulb is used. Otherwise, moisture from the air is likely to accumulate, causing excessively long evacuation periods.

FLEXIBLE DUCTING

Flexible ducting, 10 cm (4 in.), as commonly used with a home clothes dryer, is required for venting the system.

TOOLS REQUIRED

The following tools are required for installation:

1/4-in. flat-blade screwdriver
3/16-in. flat-blade screwdriver
9/16-in. wrench
7/16-in. wrench
Scissors

EQUIPMENT SETUP AND CHECKOUT

The PoreSizer should be checked to make sure it is operating properly before actual analyses are attempted. The remainder of this chapter describes how to install the PoreSizer and verify operation.

SELECTING THE POWER INPUT

All instruments leave 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 PoreSizer can be operated. The PoreSizer is designed to operate with either 100, 120, 200 or 240 VAC at 50 or 60 Hz.

WARNING
The power cord should be disconnected from the PoreSizer 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 PoreSizer.
2. Observe the small clear plastic window to the right of the power cord plug. Inside the window you will see the number 120 since the porosimeter is set to operate at 120 VAC.
3. Slide the window to the left.
4. Remove the small printed circuit card.
5. Orient the card so that the appropriate number, 120 or 240, will be visible when the card is inserted.

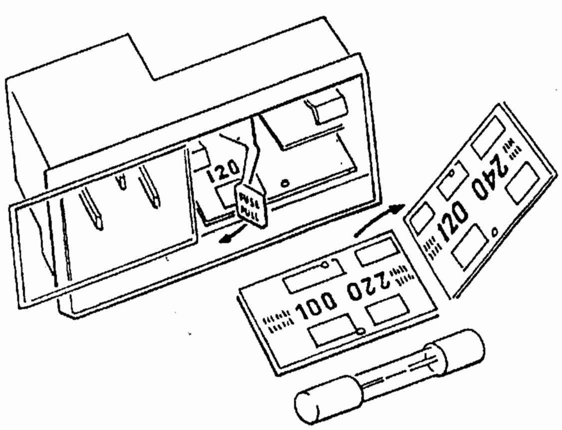


Figure 2-1. Voltage and Fuse Selection

6. Insert the card.

7. The fuse above the card must also be appropriate for the voltage. Refer to the chart below for the appropriate fuse rating.

Power Source	Fuse
100-120 VAC	3AG, 2.5 Amp Slo-Blo
220-240 VAC	3AG, 1.25 Amp Slo-Blo

8. Slide the plastic window to the right.

SELECTING CONTROL MODULE POWER INPUT

The Line Voltage Select switch on the control module must be set to match the input power source (from the wall outlet). The control module operates with either 100-120 VAC or 200-240 VAC at 50 or 60 Hz.

WARNING
Do not connect the control module 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 control module.

The chart below shows the appropriate settings. Refer to your control module operator's manual for instructions on setting the switch.

Input Power	Setting
100-120 VAC	115V
200-240 VAC	230V

SELECTING THE PRESSURE READOUT

The PoreSizer will indicate pressure in pounds per square inch absolute (psia) or in megapascals (MPa). The PoreSizer is shipped with the pressure readout set to pounds per square inch absolute; no adjustment is necessary for this setting.

There are two methods of changing the readout to megapascals. If you are operating the PoreSizer with a control module, the setting is changed on the System Options screen (refer to **Installing the Software** later in this chapter). If you are operating the PoreSizer without a control module, perform the following steps:

1. Remove the pushbutton caps labeled LOW psia and HIGH psia from the front panel. Replace them with caps from the accessory kit labeled LOW MPa and HIGH MPa. The LOW cap goes to the left and the HIGH cap to the right.

2. Make sure the power cord is unplugged.

3. Remove the seven screws holding the upper rear panel.

4. Lift the panel away from the instrument but do not disconnect the fan power leads. Observe the large circuit board with a smaller circuit board mounted on it (refer to the following illustration). These boards are mounted on a hinged plate. Remove the two captive screws at the top of the plate and pull the assembly so that it hinges out and down.

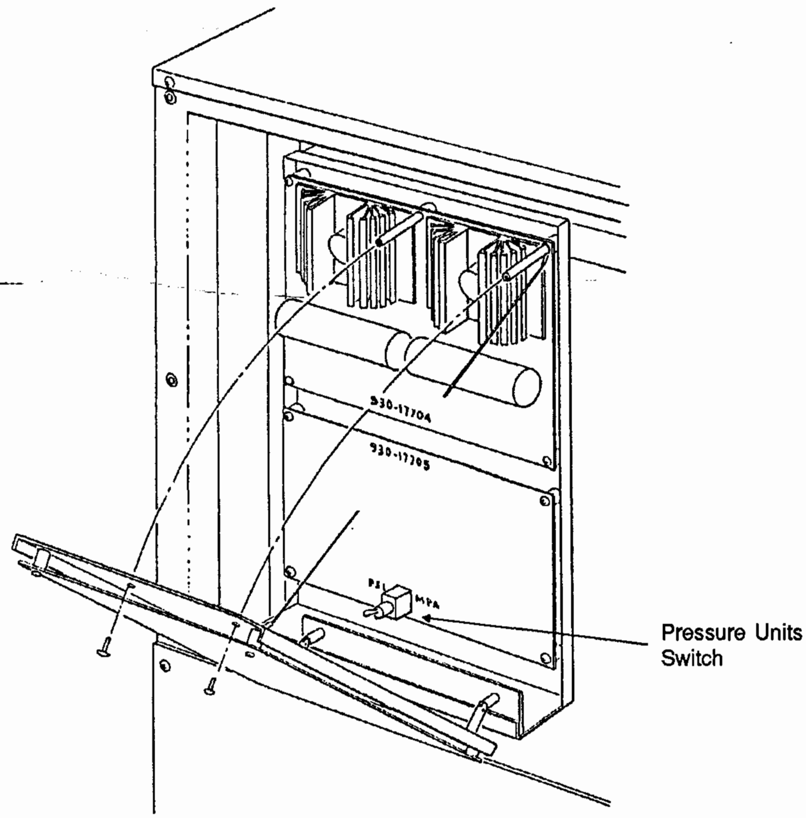


Figure 2-2. Location of Pressure Units Switch

5. Place the pressure units switch in the MPa position.

6. Replace the hinged plate and the rear panel.

CONNECTING CABLES

Connecting the Control Module to the Video Monitor, Keyboard, Printer and Plotter

NOTE

PoreSizer 9320 Software V2.00 or higher runs on both monochrome and color monitors. VGA support is required for use with color monitors.

NOTE

Refer to the instruction manuals supplied with the control module, video monitor, printer and plotter for voltage requirements for these units.

1. Plug the keyboard cable into the connector on the rear panel of the control module. Figure 2-3 shows a typical connection.

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

3. Plug the monitor power cord into the appropriate power source.

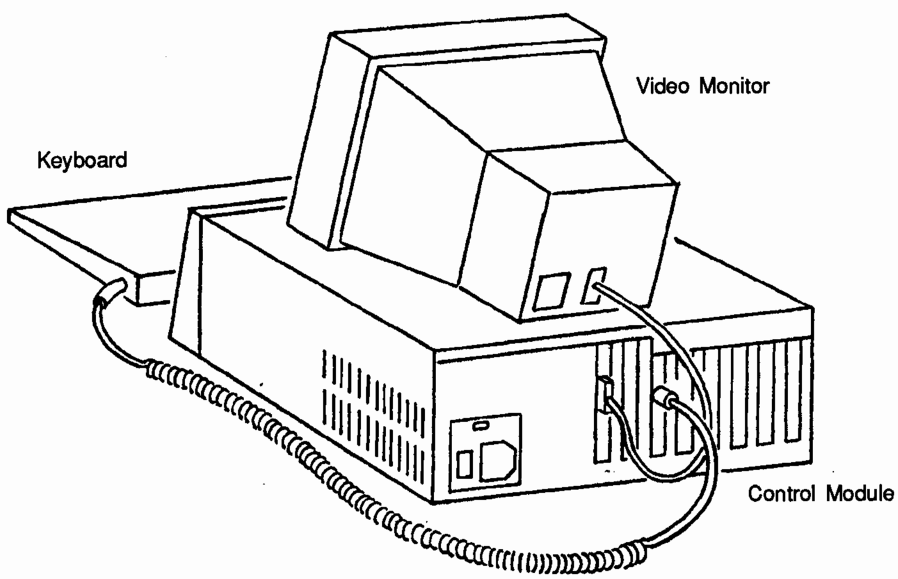


Figure 2-3. Connecting the Keyboard and Video Monitor to the Control Module

4. Connect one end of the printer cable into the input connector at the rear of the printer. Connect the other end to the output connector at the rear of the control module. Figure 2-4 shows a typical connection.

5. Plug the printer power cord into the appropriate power source.

6. If a plotter is to be used, it is connected to the output connector at the rear of the control module labeled COM2. If your control module has only one communications port, it is connected to COM1.

   Observe the COM connector at the rear of the control module nad the connector at the rear of the plotter. Each connector is either a 9-pin or 25-pin connector. Choose the correct cable according to the following chart, then connect it to the plotter and the control module.

Control Module Cable Connector	Plotter Cable Connector	Part Number
9-pin Female	25-pin Male	003-60812-00
25-pin Female	25-pin Male	003-60806-00
9-pin Female	25-pin Female	003-60813-00
25-pin Female	25-pin Female	003-60810-00

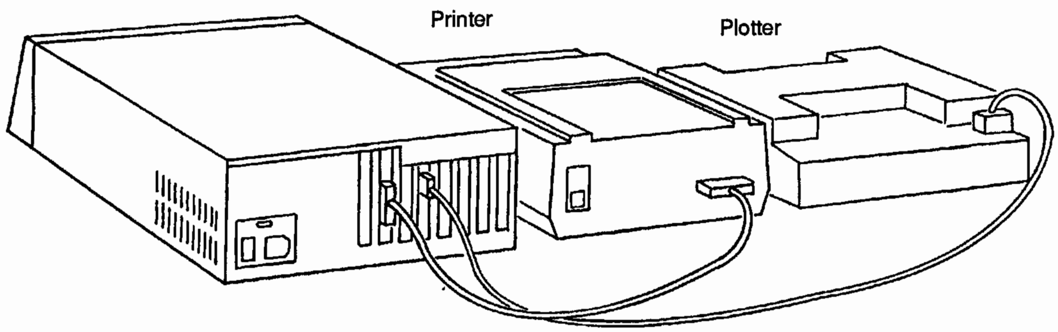


Figure 2-4. Connecting the Printer and Plotter to the Control Module

7. Plug the plotter power cord into the appropriate power source.

8. Install the low pressure capacitance detectors by inserting the plugs into the holes beneath the low pressure ports. Turn clockwise to latch. Install the caps on the low pressure ports and turn clockwise to tighten (refer to Figure 2-8).

NOTE

The capacitance detectors are labeled LEFT and RIGHT (as you are facing the unit). Make sure that they are installed in the proper ports.

9. Plug the control module power cord into the appropriate power source.

PoreSizer Rear Panel Connections

1. Connect one end of a 10 cm (4 in.) duct to the port on the upper rear panel of the PoreSizer and the other end into the venting system. Flexible ducting as commonly used with a home clothes dryer is satisfactory.

2. Connect one end of the cable provided to the connector on the rear panel of the control module for the primary serial port (COM1). Connect the other end of the cable to the connector on the rear of the PoreSizer labeled RS232. Refer to Figure 2-5.

3. Connect the vacuum trap to the connector labeled VACUUM on the rear panel of the PoreSizer. Connect the hose from the vacuum pump to the vacuum trap.

NOTE

The vacuum hose should be as short as possible for faster evacuation.

4. Connect the compressed air source to the connector labeled PRESSURE on the rear panel of the PoreSizer.

5. If you wish to connect optional equipment, such as an X-Y plotter, use, the 9-pin connector on the rear panel. The pin assignments are shown below.

Pin No.	Description
1	+15 VDC analog supply
2	+5 VDC digital supply
3	Digital ground
4	Analog ground
5	-15 VDC analog supply
6	High pressure port, capacitance signal; scaling is 200 pF per volt
7	High pressure port, pressure signal; scaling is 6000 psi per volt
8	Not connected
9	Not connected

6. Plug the PoreSizer power cord into the appropriate power source.

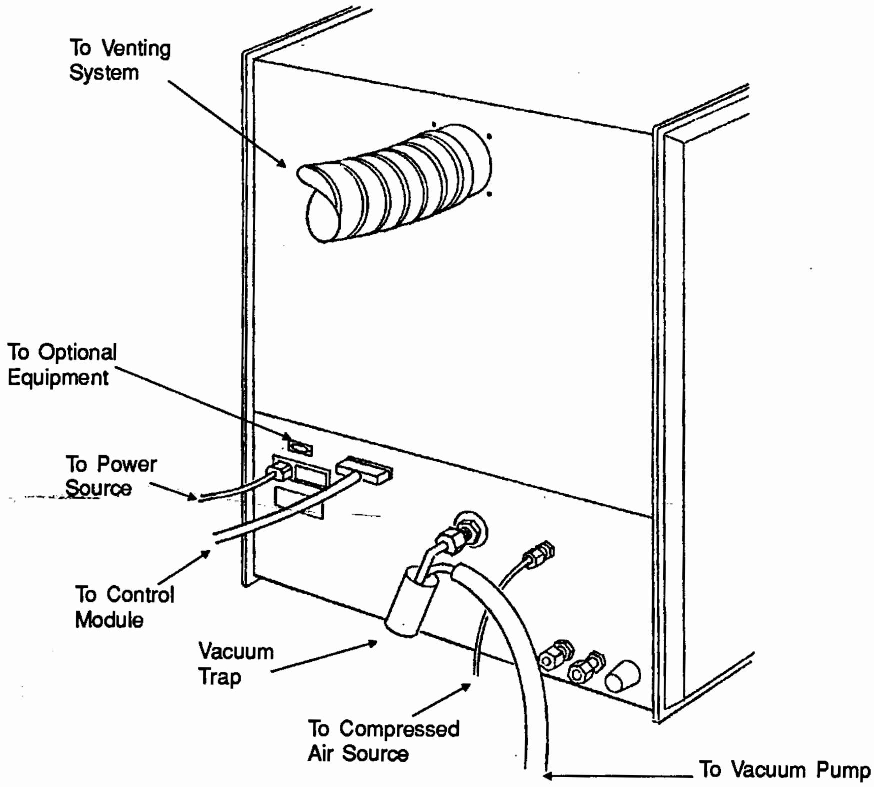


Figure 2-5. Rear Panel Connections

TURNING ON THE SYSTEM

1. Before applying power to the PoreSizer, press down and latch the PRESSURE TRANSDUCER µmHg and PENETROMETER LOCATION LEFT buttons.

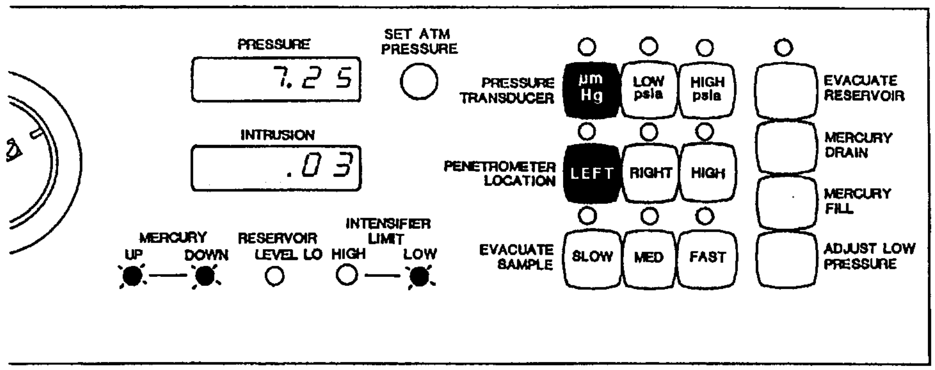


Figure 2-6. Front Panel - Initial Settings

2. Make sure the POWER switch on the lower front panel is in the OFF position and the SCAN switch is in the center (OFF) position.

3. Plug in the power cord and place the POWER switch in the ON (I) position.

4. The PRESSURE and INTRUSION displays should come on; the numbers displayed at this time are not significant. The INTENSIFIER LIMIT LOW indicator should also come on. If it does not, press PRESSURE SCAN down until it does light. Then return the PRESSURE SCAN switch to its center (OFF) position.

5. Turn on the control module, video monitor, printer, and, if used, the plotter.

INSTALLING THE SOFTWARE

The analysis program is supplied on two diskettes (labeled Disk #1 and Disk #2). To install the program:

1. If DOS is not installed on your control module, install it before proceeding.

2. With the DOS C:\> prompt displayed, insert Disk 1 in drive A.

3. Type:

   A:INSTALL

   and press [Enter].

   NOTE

   You may cancel the installation at any time by pressing [Esc]. If you do so, you must start the installation program from the beginning to install the analysis program.

4. A logo screen and a few instructional screens are displayed. Follow the instructions displayed on the screens.

   NOTE

   If Drive C contains insufficient disk space, a message instructing you to free up space prior to installation is displayed. If this occurs, back up seldom used files to diskette, then delete them from Drive C. When sufficient space is available, reinsert Disk 1 in Drive A and type A:INSTALL.

5. When installation is complete, the following message is displayed:

   Installation of the PoreSizer 9320 program is now complete. To run 9320 PoreSizer type "run93" at the DOS prompt.

   Press any key to continue...

   Press any key to return to the DOS prompt.

6. When the DOS prompt is displayed, type:

   RUN93

   and press [Enter].

7. Within five to ten minutes the Analysis Program Main Function Menu will be displayed. Once the program is loaded, subsequent initialization will take less time.

8. The System Options screen enables you to change certain system parameters. The parameters are initially set as follows:

Field	**Default Value**
Pressure units?	psia
Size type?	diameter
Size units?	micrometers
Atmospheric pressure:	14.700 psia
Date:	date from the control module clock/calendar
Time:	time from the control module clock/calendar
Type of plotter?	manual
Show grid?	no

If you wish to change system parameters, press F10, F8 from the Main Function Menu to display the System Options screen. Move the cursor to the field you wish to change. Press 5 on the numeric keypad until the desired value is displayed for fields that end in a question mark (?). Type in the correct value for fields that end in a colon (:).

NOTE

If you wish to change the pressure units to megapascals, remove the pushbutton caps labeled LOW psia and HIGH psia from the front panel. Replace them with the caps from the accessory kit labeled LOW MPa and HIGH MPa and select megapascals at the Pressure units? prompt.

When you have finished, press F2 twice to return to the Main Function Menu.

SETTING UP THE PRINTER

In order to produce analysis results using the printer, the printer operating conditions must be set for the control module system being used. Table 2-1 lists the printer settings that usually require verification or changing. Refer to the manual supplied with the printer to set the operating conditions. Not all printers allow these options to be changed.

Table 2-1. Printer Settings for the PoreSizer 9320 System

Function	Setting
Graphics mode*	IBM graphics or Epson LQ
Form length	11 inches or greater
Lines per inch	6
Characters per inch	10
Character generator	Built in. Select U.S. style if international character choices are available.
Linefeed action	Linefeed only (no automatic carriage return).
Carriage return action	Carriage return only (no automatic linefeed).

*Selections available under **System Options**.

The default settings for other features should work on your printer; experiments may be required for unusual features.

Refer to **Chapter 5, Printing Reports**, for instructions on printing reports.

SETTING UP THE PLOTTER

In order to produce reports using the plotter, the plotter operating conditions must be set as shown in the following table.

Table 2-2. Plotter Settings for the PoreSizer 9320 System

Function	Setting
Baud rate	9600
Parity	None
Data bits	8
Stop bit	1

Refer to **Chapter 5, Printing Reports**, for instructions on printing reports on the plotter.

VERIFYING OPERATION

Once installation of the PoreSizer is complete, perform the following checkout procedure to verify proper operation before attempting to run an analysis. The materials required for verification are listed in the following table.

Table 2-3. Materials Required to Verify Operation

Items Supplied by Micromeritics	Items Supplied by User
High pressure fluid	Analytical balance
3-cc bulb, 0.412-cc capillary penetrometer	Funnel
Reference material	Mercury
Sample Data Sheet	Mercury waste containers
Vacuum grease (Apiezon H)	Oil
	Rubber gloves
	Vacuum pump oil

PLACING THE SYSTEM IN MANUAL MODE

The following procedures require operating the PoreSizer in manual mode. If a control module is attached and running the Analysis Program, you must exit the Analysis Program. To exit the Analysis Program, press F10 twice from the Main Function Menu. Answer yes to the prompt displayed on the screen. Make sure Drive A is open, then press PgDn to exit to DOS.

FILLING MERCURY RESERVOIR

The PoreSizer is shipped without mercury in its reservoir although a few droplets may remain from testing the instrument during manufacturing and quality control checks. To fill the reservoir:

1. Lift out the mercury reservoir plug.

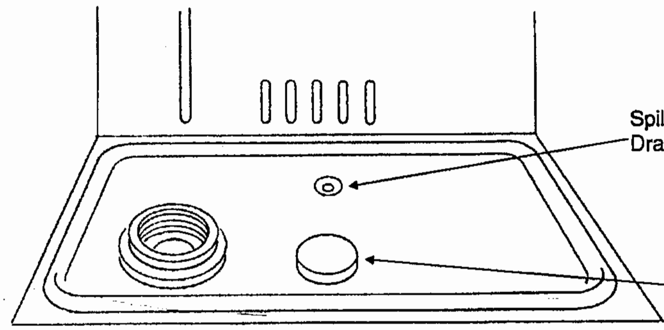


Figure 2-7. Mercury Reservoir and Spilled Mercury Drain

2. Carefully pour mercury into the reservoir using a funnel. Add no more than 250 cc (7.5 lb) and no less than 100 cc (3.0 lb.) of mercury. The level of mercury in the reservoir must not reach the lower opening of the neck of the reservoir filling hole. Filling the reservoir to within 0.5 to 1.0 inch of that level is sufficient.

> **CAUTION**
> **DO NOT OVERFILL. Damage to the vacuum system may result from overfilling.**

3. Replace the plug by pressing it down firmly.

PREPARING SPILLED-MERCURY CONTAINER

The small drain hole in the center of the Poresizer (refer to Figure 2-7) is provided so that any spilled mercury can immediately be brushed into a container directly beneath the hole. To prepare the container, pour 2 to 3 cc of oil through the hole and into the container. Any mercury that falls into the container will thus be covered with a layer of oil and will not be free to vaporize.

CHECKING VACUUM AND LOW PRESSURE TRANSDUCERS

To check the vacuum and low pressure transducers:

1. Remove the capacitance detectors from the low pressure ports by turning them counterclockwise and pulling forward. Lay them on the shelf-like tray on top of the PoreSizer.

2. Press the LOW psia (MPa) button. The PRESSURE display should now show atmospheric pressure (either 14 or 15 psia or 0.096 to 0.103 MPa). If it is not, the low pressure system may be under a slight vacuum. Unscrew the retaining knobs and remove the blank rods.

3. To check reduced pressure indications, use the two blank rods you removed from the low pressure ports. First apply a light coating of vacuum grease to one end of each rod.

4. Insert the rods (greased end first) completely into the low pressure ports and tighten the retaining knobs until the rods are securely held.

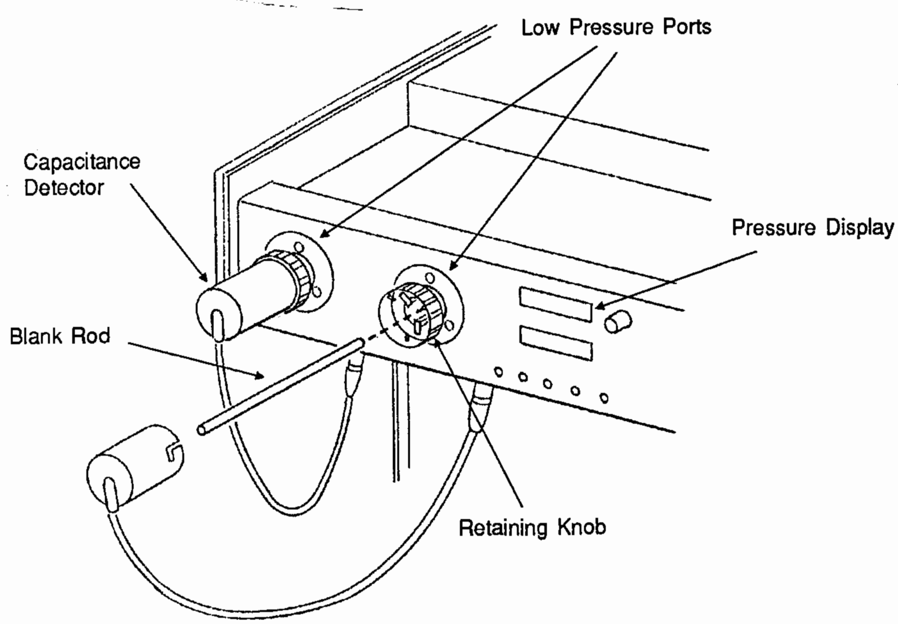


Figure 2-8. Checking Vacuum and Low Pressure Transducers

5. Make sure the vacuum pump is filled with oil (refer to **Maintaining Vacuum Pump Fluid Level** in Chapter 8). Then turn on the vacuum pump.

6. Press the EVACUATE SAMPLE FAST button. If the vacuum pump and sealing rods are properly installed, the indicated pressure should decrease rapidly. It should attain 0.1 psia (0.0006 MPa) within one minute.

7. Press the PRESSURE TRANSDUCER μmHg button. The pressure shown by the indicator should attain 100 μmHg in about five minutes.

8. Press the LOW psia (MPa) button.

9. Unlatch the EVACUATE SAMPLE FAST button.

10. Return the system to atmospheric pressure by lightly tapping the ADJUST LOW PRESSURE button.

11. Remove the blank rods, and replace the capacitance detectors.

CHECKING HIGH PRESSURE TRANSDUCER AND PRESSURE GENERATION CONTROLS

The PoreSizer was filled with the required quantity of high pressure fluid before shipment. Check to ensure that the proper level of fluid is still in the high pressure chamber as follows:

1. Turn the vent valve (refer to Figure 2-9) counterclockwise one or two turns.

2. Turn the lever arms counterclockwise and then lift up to raise the chamber cap to its up position.

3. Wait a few moments to allow high pressure fluid to drain back into the chamber, then observe the level of fluid in the chamber. It should be just below the ledge shown in Figure 2-9. If the fluid is below this level, add fluid.

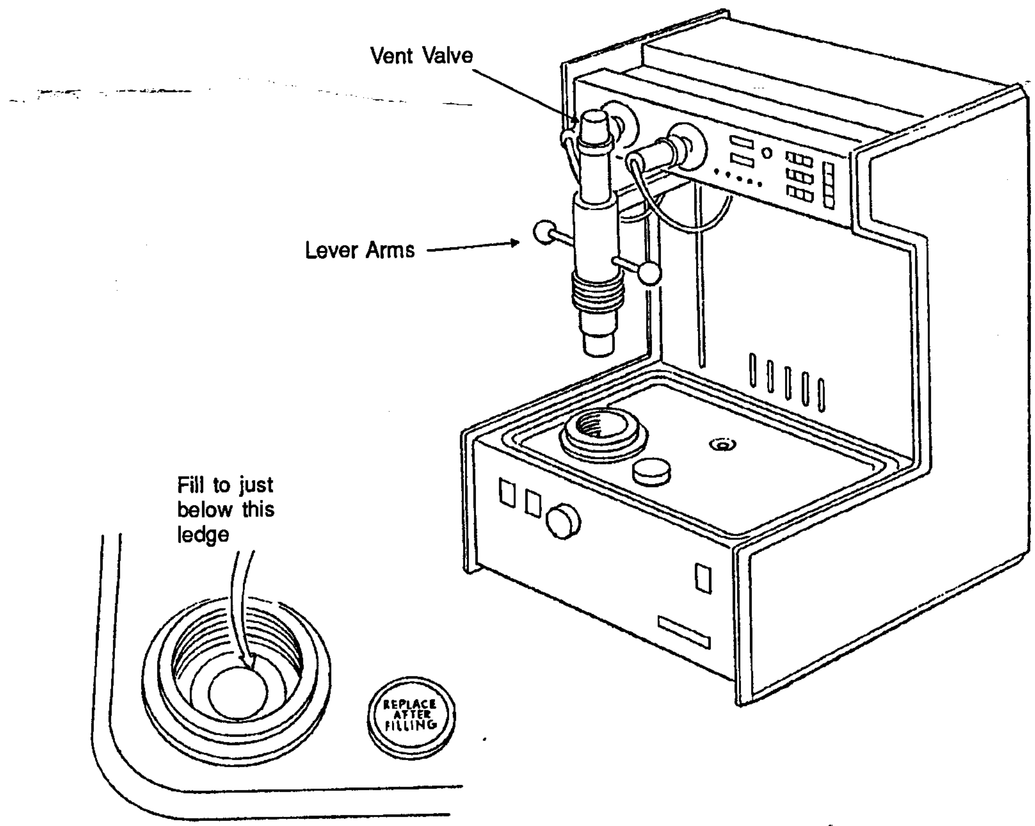


Figure 2-9. Checking Fluid Level

4. Press the HIGH psia (MPa) button.

5. Lower the chamber cap, then slowly turn the arms clockwise. Note the small plastic cup on top of the cap. If air bubbles are present, tighten, then loosen the arms two or three times. If bubbles are still present, open the vent valve and add more high pressure fluid.

6. The PRESSURE display should now show atmospheric pressure. It can be adjusted to within ±0.5 psia (0.003 MPa) of the true atmospheric pressure by turning the SET ATM PRESSURE knob.

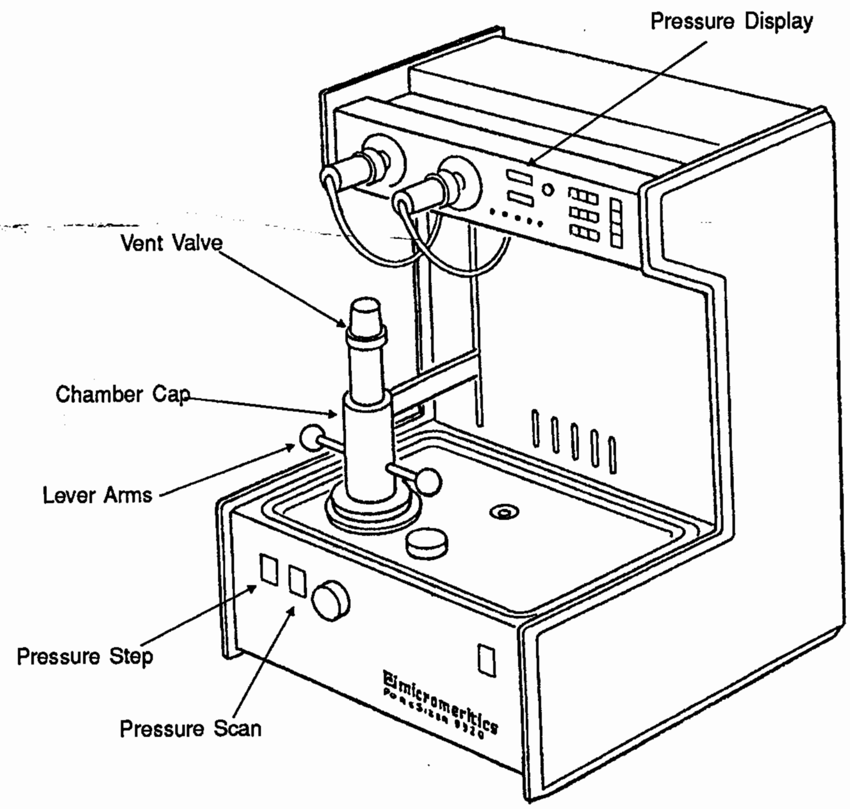


Figure 2-10. Checking High Pressure Transducer and Pressure Generation Controls

7. Close the vent valve on the high pressure chamber.

8. Turn the RATE knob fully clockwise.

9. Place the PRESSURE SCAN switch in the UP position and hold it for a moment. The indicated pressure should increase.

11. Wait until the instrument has generated 30,000 psi (this could take 10 to 15 minutes) and then place the PRESSURE SCAN switch in the OFF position.

> **CAUTION**
> **Do not generate pressures above 30,000 psi.**

12. Place the PRESSURE SCAN switch in the DOWN position until the INTENSIFIER LIMIT LOW indicator comes on. Then return the switch to the OFF (center) position.

13. Open the vent valve on the high pressure chamber.

CHECKING OPERATION OF THE PORESIZER: SYSTEMS WITHOUT A CONTROL MODULE

The following instructions apply to systems that do not use a control module. If you have a control module attached to your PoreSizer, skip to **Checking Operation of the PoreSizer: Systems with a Control Module.**

To ensure that the PoreSizer is operating properly, perform an analysis using the reference material provided as described below.

1. Clean the 3-cc bulb, 0.412-cc capillary powder penetrometer (refer to **Cleaning Penetrometers** in Chapter 4).

2. Remove 10 pellets from the reference material provided. Do not touch the sample because skin oils may be transferred, which can slightly alter results or create evacuation problems.

   Weigh the 10 pellets using an analytical balance. Record the weight on the Pore Size Distribution Data Sheet as **Sample weight.**

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

4. Seal the penetrometer (refer to **Sealing the Penetrometer** in Chapter 4).

5. Install the assembled penetrometer in the left low pressure port. Install a blank rod in the right low pressure port. (Refer to **Installing Penetrometers in Low Pressure Ports** in Chapter 4.)

6. Press the EVACUATE SAMPLE SLOW and LOW psia PRESSURE TRANSDUCER buttons.

7. Turn on power switches for both the PoreSizer and the vacuum pump if not already on.

8. When the indicated pressure drops below 1 psia, release the EVACUATE SAMPLE SLOW button and press the EVACUATE SAMPLE MED and μmHg PRESSURE TRANSDUCER buttons.

9. When the indicated pressure drops to 250 μmHg, release the EVACUATE SAMPLE MED button and press the EVACUATE SAMPLE FAST button.

10. Allow evacuation to continue until the vacuum on the sample stabilizes. This vacuum pressure must be less than 50 μmHg. If it is not, refer to Table 8-1 in Chapter 8 for possible causes and corrective actions.

11. Replace low pressure port capacitance detectors when vacuum conditions have stabilized. Turn the detectors clockwise to tighten the closure.

12. Press the MERCURY FILL button momentarily and release it. If there is a loss of vacuum, press down on the MERCURY RESERVOIR cap; it must be tightly sealed.

    If there is still a loss of vacuum, release the MERCURY FILL button and wait until a good vacuum is established, perhaps five minutes. Repeat the procedure until the MERCURY FILL button can be held in without loss of vacuum. Hold the button in until the MERCURY UP indicator comes on. Release the MERCURY FILL button. If the indicator turns off, press the MERCURY FILL button until the indicator stays on.

> **CAUTION**
> **Release the button as soon as the light comes on.**

13. Release the EVACUATE SAMPLE FAST button and shift from µmHg PRESSURE TRANSDUCER to LOW psia PRESSURE TRANSDUCER.

14. Press ~~and hold~~ the ADJUST LOW PRESSURE button until it has reached 1.5 psia.

15. Press and latch the EVACUATE RESERVOIR button and wait 20 to 30 seconds or until the sound from the vacuum pump returns to its normal operating level. Do not release the EVACUATE RESERVOIR button.

> **CAUTION**
> **Do not release the EVACUATE RESERVOIR button until the sound from the vacuum pump returns to its normal operating level. If you release the button prematurely, you will risk backing up mercury in the system.**

16. Press and hold down the MERCURY DRAIN button until the MERCURY DOWN light comes on.

17. Release the MERCURY DRAIN and EVACUATE RESERVOIR buttons.

18. Press the LEFT PENETROMETER LOCATION button if the sample is in the left low pressure port; otherwise, press the RIGHT button. Record the values shown in the PRESSURE and INTRUSION displays on the Pore Size Distribution Data form.

19. Begin the low pressure intrusion test by pressing the ADJUST LOW PRESSURE button momentarily to create a series of pressures up to 1 atmosphere (approximately 14.7 psia).

20. Continue the series of increasing pressures to between 22 and 25 psia by pressing the ADJUST LOW PRESSURE button and using an external source of gas pressure. Record data at the pressure points shown in the following table.

Table 2-4. Standard Pressure Points

1)	2.000
2)	3.000
3)	4.000
4)	5.500
5)	7.000
6)	8.500
7)	10.500
8)	13.000
9)	16.000
10)	20.000
11)	23.000
12)	25.000

21. Return the system to atmospheric pressure by lightly tapping the SLOW EVACUATION button until a pressure between 15-16 psia is reached.

22. The low pressure run is now complete. Remove the penetrometers as described under **Removing Penetrometers from Low Pressure Ports** in Chapter 4.

23. Install the penetrometer assembly in the high pressure chamber as described in **Installing the Penetrometer in the High Pressure Chamber** in Chapter 4. Press the HIGH psia PRESSURE TRANSDUCER button and the HIGH PENETROMETER LOCATION button.

24. Adjust the SET ATM PRESSURE knob until atmospheric pressure is displayed.

25. Close the vent valve on top of the chamber cap.

26. To record data:

    a. Take one set of pressure and intrusion values at the highest pressure (minus the head correction) recorded in the low pressure portion of the analysis. Adjust the RATE knob as appropriate to set the rate of pressure generation.

    b. Press the PRESSURE STEP UP button momentarily to set increasing pressures. Wait approximately 10 seconds. Record the desired data.

    c. Stop the pressure generation when 30,000 psia is attained.

    d. For equilibrium extrusion data, press the PRESSURE STEP DOWN button at intervals and record values.

27. Press the PRESSURE SCAN DOWN button until the LOWER LIMIT indicator comes on.

28. Remove the penetrometer as described under **Removing the Penetrometer from the High Pressure Chamber** in Chapter 4.

29. Clean the penetrometer (refer to **Cleaning Penetrometers** in Chapter 4).

30. Calculate pore area distribution data. Compare the results with the information in the Product Bulletin supplied with the reference material.

CHECKING OPERATION OF THE PORESIZER: SYSTEMS WITH A CONTROL MODULE

The following instructions apply to systems that have a control module attached. If you do not have a control module attached to your PoreSizer, refer to **Checking Operation of the PoreSizer: Systems without a Control Module** earlier in this chapter.

To ensure that the PoreSizer and the control module are operating properly, perform an analysis using the reference material provided as described below.

NOTE

You may wish to refer to Table 3-1, Keys Used by the Analysis Program, to familiarize yourself with the keys for entering information into the system.

1. Re-enter the Analysis Program by typing **EXIT** at the DOS prompt.
2. Clean the 3-cc bulb, 0.412-cc capillary powder penetrometer (refer to **Cleaning Penetrometers** in Chapter 4).
3. Remove the Sample Data Sheet from Appendix A and make a copy of it. Use the copy and retain the original. You may wish to make a number of copies at this time to be used for future analyses.
4. Fill out the top portion of the Sample Data Sheet as follows:

Sample number:	1 if during installation; the next consecutive number (shown on the Add Sample Information Screen) if sample files already exist in the system.
Sample ID:	REFERENCE MATERIAL
Submitter ID:	N/A
Operator ID:	Your name.

5. Remove 10 pellets from the reference material provided. Do not touch the sample because skin oils may be transferred, which can slightly alter results or create evacuation problems.

   Weigh the 10 pellets using an analytical balance. Record the weight on the Sample Data Sheet as **Sample weight.**
6. Hold the penetrometer with the stem down and carefully pour the sample into the bulb.
7. Seal the penetrometer (refer to **Sealing the Penetrometer** in Chapter 4).
8. Weigh the penetrometer using an analytical balance. Record the weight on the sample data sheet as **Sample + penetrometer weight**. Subtract **Sample weight** from **Sample + penetrometer weight**; record on the sample data sheet as **Penetrometer weight**.

9. With the Main Function Menu displayed on the video monitor, press [F3] twice to display the Add Sample Information screen. The following screen is displayed.

NOTE

Make sure the [Num Lock] key is turned off.

```
PORESIZER 9320 VX.XX              DIR: DATA1            DATE:07/13/89 TIME:09:13:53
LP:     14.70 psia IDLE                                 HP:     14.7 psia IDLE
PORT         1           2                              PORT       1
SAMPLE                                                  SAMPLE
INTVOL      0.000       0.000      mL/g                 INTVOL     0.000     mL/g
% STEM                                                  % STEM

                        3.3p1 ADD SAMPLE INFORMATION

Sample number:     1
Sample ID:  REFERENCE MATERIAL
Submitter ID:
Operator ID:  JANE BARRETT

Pntr number:  05-0673                   Pntr constant:  10.790  µL/pF
Pntr wt:      1.0000  g                 Max head press:  4.680   psi
Pntr vol:   1.0000  mL                  Stem vol:  0.4120  mL

Advancing contact <:  130.000  deg      Receding contact <:  130.000  deg
Hg surf ten:  485.000  dynes/cm         Hg density:  13.5335  g/mL
Samp wt:      1.5000  g

                                                                       [PgDn]
```

Figure 2-11. Add Sample Information Screen

10. Press [Enter] to enter the default sample number. Enter the sample ID and operator ID from the Sample Data Sheet, pressing [Enter] after each entry.

11. Enter information in the remaining fields on the screen as follows:

Pntr number:	Enter the number etched on the penetrometer bulb and press [Enter].
Pntr constant:	Press [Enter] to accept the default value of 10.790 µL/pF.
Pntr wt:	Enter the penetrometer weight from the Sample Data Sheet and press [Enter].
Max head press:	Press [Enter] to accept the default value of 4.680 psi.
Pntr vol:	Press [Enter].

Stem vol:	Press ↵ to accept the default value of 0.4120 mL.
Advancing contact:	Press ↵ to accept the default value of 130.000 deg.
Receding contact:	Press ↵ to accept the default value of 130.000 deg.
Hg surf ten:	Press ↵ to accept the default value of 485.000 dynes/cm.
Hg density:	Press ↵ to accept the default value of 13.5335 g/mL.
Sample weight:	Enter the sample weight from the Sample Data Sheet and press ↵.

Press F2 twice to return to the Main Function Menu.

12. Install the assembled penetrometer in the left low pressure port. Install a blank rod in the right low pressure port. (Refer to **Installing Penetrometers in Low Pressure Ports** in Chapter 4.)

13. From the Main Function Menu, press F7 to display the Start Low Pressure Run screen. The following screen is displayed.

```
PORESIZER 9320 VX.XX                 DIR: DATA1           DATE:07/14/89 TIME:06:22:53
LP:    14.70 psia IDLE                                    HP:      14.7 psia IDLE
PORT       1        2                                     PORT       1
SAMPLE                                                    SAMPLE
INTVOL     0.000    0.000    mL/g                         INTVOL     0.000    mL/g
% STEM                                                    % STEM

                          7p1 START LOW PRESSURE RUN

                  PORT1       PORT2

Sample number:     1             2

Start report after run?  yes

Report destination?  printer



Press [PgDn] to start run, or [Esc] for no action.
```

Figure 2-12. Start Low Pressure Run Screen

14. Enter information in the fields on the screen as follows:

Sample number: Enter again the sample number you entered on the Add Sample Information screen in the PORT1 field. Press ⏎.

Press ⏎ to accept the default value of 0 in the PORT2 field.

Start report after run? Select NO and press ⏎. (Press 5 on the numeric keypad to toggle the field between yes and no.)

15. Press PgDn. Screens containing instructions for the low pressure run are displayed. Follow the instructions on the screens (refer to **Entering and Maintaining Analysis Information** in Chapter 6 for an illustration of these screens).

16. Press F9, F3 from the Main Function Menu to display the Low Pressure Run Control screen.

```
PORESIZER 9320 VX.XX             DIR: DATA1             DATE:07/13/89 TIME:09:20:43
LP:      3.34  psia MANU COLL                           HP:       14.7 psia IDLE
PORT       1        2                                   PORT        1
SAMPLE     31       32                                  SAMPLE
INTVOL     0.003    0.000   mL/g                        INTVOL      0.000    mL/g
% STEM     0                                            % STEM

                          9.3 LOW PRESSURE RUN CONTROL

                      Low Pressure Sample Directory: DATA1
Point number   Pressure( psia )   Left Intrusion( mL/g )   Right Intrusion( mL/g )
     1)             1.50                0.000                     0.000
                    2.17                0.001                     0.000
                    3.34                0.003                     0.000

     F2 - Run Control/Status Menu      F3 - Record Data      F4 - Finish Run
```

Figure 2-13. Low Pressure Run Control Screen

Observe the low pressure shown in the status display portion of the screen. Data should be recorded at the points (or approximately) shown in Table 2-5 as follows:

a. Press the ADJUST LOW PRESSURE switch on the front panel of the PoreSizer while observing the pressure reading in the status display.

b. When the pressure reading equals the first point shown in the following table, release the ADJUST LOW PRESSURE switch.

c. Press F3 on the control module keyboard to record the pressure point.

d. Repeat steps a through c for each pressure point shown in the following table.

e. When you have finished recording pressure points, press F4. Follow the instructions displayed on the screen for returning the system to atmospheric pressure.

Table 2-5. Standard Pressure Points

1)	2.000
2)	3.000
3)	4.000
4)	5.500
5)	7.000
6)	8.500
7)	10.500
8)	13.000
9)	16.000
10)	20.000
11)	23.000
12)	25.000

17. Remove the penetrometer from the low pressure port (refer to **Removing Penetrometers From Low Pressure Ports** in Chapter 4).

18. Install the penetrometer in the high pressure chamber (refer to **Installing the Penetrometer in the High Pressure Chamber** in Chapter 4).

19. Press [F8] from the Main Function Menu to display the Start High Pressure Run screen. The following screen is displayed.

```
PORESIZER 9320 VX.XX            DIR: DATA1          DATE:07/14/89 TIME:08:45:49
LP:    14.70 psia  IDLE                             HP:      14.7 psia  IDLE
PORT      1         2                               PORT       1
SAMPLE                                              SAMPLE
INTVOL    0.000     0.000    mL/g                   INTVOL     0.000    mL/g
% STEM                                              % STEM

                        8p1 START HIGH PRESSURE RUN

Sample number:     1                      Pressure table number:  1

Run type?  automatic                      Run method?  equilibrated

Start report after run?  yes              Equilibration time:      10 seconds

Report destination?  printer              Maximum intrusion volume:    0.000  mL/g

Atmospheric pressure:  14.7000   psia     Last collected point:  14.7000     psia

Sampwt+Pntrwt+Hgwt:      1.0000   g
```

Figure 2-14. Start High Pressure Run Screen

20. Enter information in the fields as follows:

Sample number: Enter the sample number and press [↵] (must be the same number you entered on the Add Sample Information screen).

Pressure tablenumber: Enter 1 to use the pressure table provided or if you wish to use a previously-defined pressure table, enter the number of the pressure table. Press [↵].

NOTE

A pressure table (number 1) is provided with the Analysis Program, but will be present only if copied according to the instructions given under Installing the Software in Chapter 2.

Run type? Select automatic and press [↵]. (Press [5] on the numeric keypad to toggle the field between automatic and manual.)

Run method? Select equilibrated. (Press [5] on the numeric keypad to toggle the field between equilibrated and rate sequence.)

Start report after run?	Select yes and press ↵. (Press 5 on the numeric keypad to toggle the field between yes and no.)
Equilibration time:	Enter 10 and press ↵.
Report destination?	Select printer and press ↵. (Press 5 on the numeric keypad until **printer** is displayed.)
Maximum intrusion volume:	Press ↵ to accept the default value.
Atmospheric pressure:	Press ↵ to accept the default value.

The sample + penetrometer + mercury weight is for density calculations only, so it does not need to be entered for this procedure.

The last point collected is a display field only.

21. Press PgDn. If you wish to monitor the high pressure run, press F9, F5 to display the Auto High Pressure Run Control/Status screen.

22. Remove the penetrometer from the high pressure chamber (refer to **Removing the Penetrometer From the High Pressure Chamber** in Chapter 4).

23. Clean the penetrometer (refer to **Cleaning Penetrometers** in Chapter 4).

24. Remove the reports from the printer. Compare the results with the information in the Product Bulletin supplied with the reference material.

This completes the procedure for verifying operation. If the results match the information contained in the Product Bulletin, the PoreSizer is ready for operation. If the results do not match, repeat steps 1 through 24. If the desired results are not obtained, service to the system or operational assistance may be required.

CHAPTER 3

GENERAL OPERATING INSTRUCTIONS

- Front Panel Controls and Indicators
- Rear Panel Controls
- Turning the System On and Off
- Analysis Program Menu Structure
- Using the Keyboard
- The Analysis Program Input Screens
- Status Display
- Backing Up Files

GENERAL OPERATING INSTRUCTIONS

FRONT PANEL CONTROLS AND INDICATORS

LOW PRESSURE PANEL

The controls on the upper front panel, which are described below, control low pressure analyses. The indicators specify operating conditions for both low and high pressure analyses.

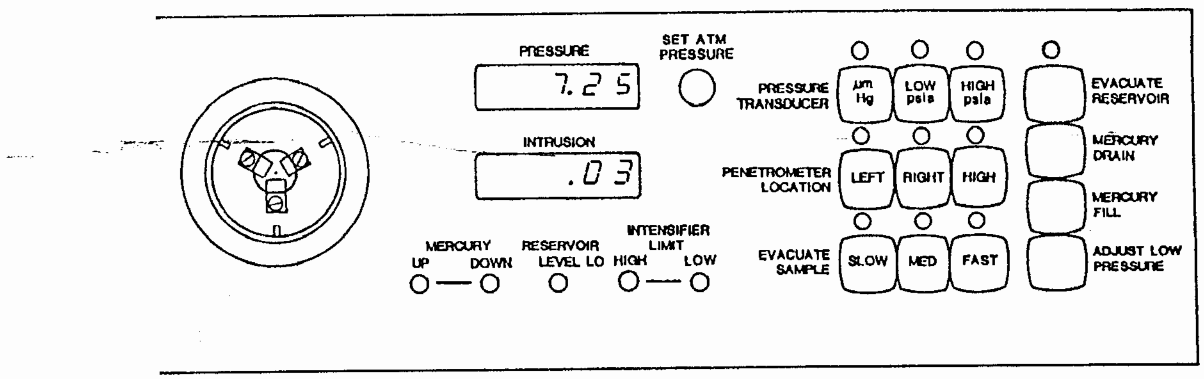


Figure 3-1. Upper Front Panel Controls and Indicators

PRESSURE display	Displays the current pressure in psia or MPa and µmHg when the PoreSizer is being operated without a control module. Displays 8's when the PoreSizer is being operated with a control module. In this case, the pressure is displayed on the video monitor.
INTRUSION display	Displays the current mercury intrusion volume in pF when the PoreSizer is being operated without a control module. Displays 8's when the PoreSizer is being operated with a control module. In this case, the intrusion volume is displayed on the video monitor.
SET ATM PRESSURE knob	Used to adjust front panel high pressure reading to match atmospheric pressure. Turn clockwise to increase the pressure reading or counterclockwise to decrease the pressure reading.

MERCURY UP and DOWN indicators

The UP indicator comes on when the level of mercury in the evacuated sample is sufficient.

The DOWN indicator comes on when the mercury is drained from the manifold.

RESERVOIR LEVEL LOW indicator

This indicator comes on when the level of mercury in the reservoir is below the level needed for normal operation.

When this indicator comes on, add mercury to the reservoir.

INTENSIFIER LIMIT HIGH and LOW indicators

The HIGH indicator comes on when the high pressure intensifier reaches its maximum pressure generating position. When this indicator comes on, the motor is turned off.

The LOW indicator comes on when the high pressure intensifier reaches reset position. When this occurs, the motor is turned off.

PRESSURE TRANSDUCER

μmHg

Use this latch switch to display pressures below 1000 μmHg for the low pressure system.

LOW psia

Use this latch switch to display pressures above 0.1 psia on the low pressure system.

HIGH psia

Use this latch switch to display pressure for the high pressure system.

PENETROMETER LOCATION

LEFT

Use this latch switch to display capacitance in pF for the low pressure station to the left as you are facing the unit.

RIGHT

Use this latch switch to display capacitance in pF for the low pressure station to the right as you are facing the unit.

HIGH

Use this latch switch to display capacitance in pF for the high pressure port.

EVACUATE SAMPLE

SLOW

Use this latch switch to select slow speed evacuation, which should be used until pressure drops to 1 psia.

> **CAUTION**
> Some powders require gentler evacuation. These powders will easily fluidize and could be drawn into the plumbing and valves. To avoid fluidization, the sample must be allowed to attain a lower pressure, about 0.5 psia, under slow evacuation.

Refer to **Low Pressure Operation** in Chapter 4.

MED

Use this latch switch to select medium speed evacuation, which should be used until pressure drops to 250 μmHg.

FAST

Use this latch switch to select fast speed evacuation, which should be used until pressure drops to 50 μmHg or has equilibrated at some lower pressure.

EVACUATE RESERVOIR

Use this latch switch to pull a vacuum in the mercury reservoir.

MERCURY DRAIN

Use this momentary switch to drain excess mercury from the evacuated sample. This switch should be held down until the MERCURY DOWN indicator comes on.

MERCURY FILL

Use this momentary switch to fill the evacuated sample with mercury. This switch should be held down until the MERCURY UP indicator comes on.

> **CAUTION**
> Release this switch as soon as the MERCURY UP indicator comes on.

ADJUST LOW PRESSURE

Use this momentary switch to increase pressure on the low pressure station.

HIGH PRESSURE PANEL

The controls on the lower front panel, which are described below, control high pressure analyses and turn on and off power to the unit.

NOTE

These controls (except the RATE knob) are disabled when an automatic high pressure analysis is in progress. The RATE knob sets the maximum rate of pressure generation available during automatic high pressure operation, and should normally be left at the maximum (clockwise) position during automatic operation.

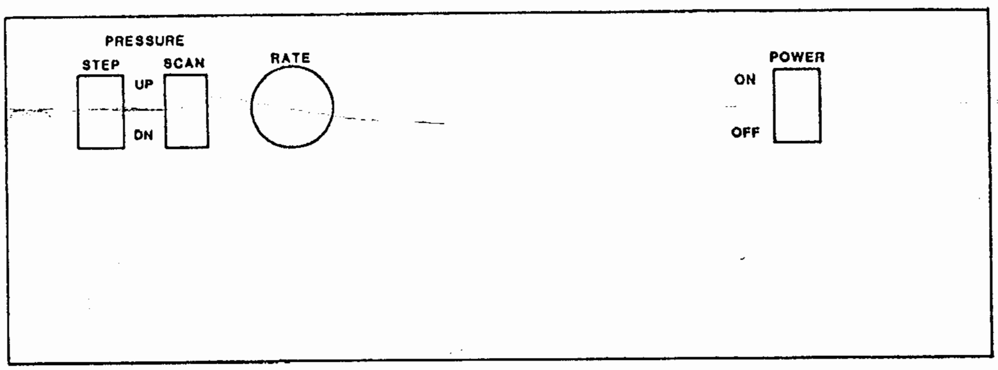


Figure 3-2. Lower Front Panel Controls

PRESSURE

STEP UP and DOWN switch

Use this momentary switch to make small changes in pressure in the high pressure chamber.

Press UP to increase the pressure at the rate selected by the RATE knob.

Press DOWN to decrease the pressure at the rate selected by the RATE knob.

SCAN UP and DOWN switch

Use this latch switch to make large changes in pressure in the high pressure chamber at the rate selected by the RATE knob.

Press UP to increase continually the pressure until you return the switch to the center (OFF) position.

CAUTION

Do not leave the switch unattended in the UP position. This could cause an automatic shutdown slightly above 30,000 psia with possible damage to the unit.

Press DOWN to continually decrease the pressure until you return the switch to center (OFF) position.

RATE knob

Use this knob to control the rate of pressure increase.

NOTE

The RATE knob will control the motor speed for automatic runs as well as manual runs.

Turn clockwise to increase the motor speed and counterclockwise to decrease the speed.

POWER ON and OFF switch

Use this switch to turn the unit on or off.

Place in the ON (up) position to turn on power to the unit.

Place in the OFF (down) position to turn off power to the unit.

REAR PANEL CONTROLS

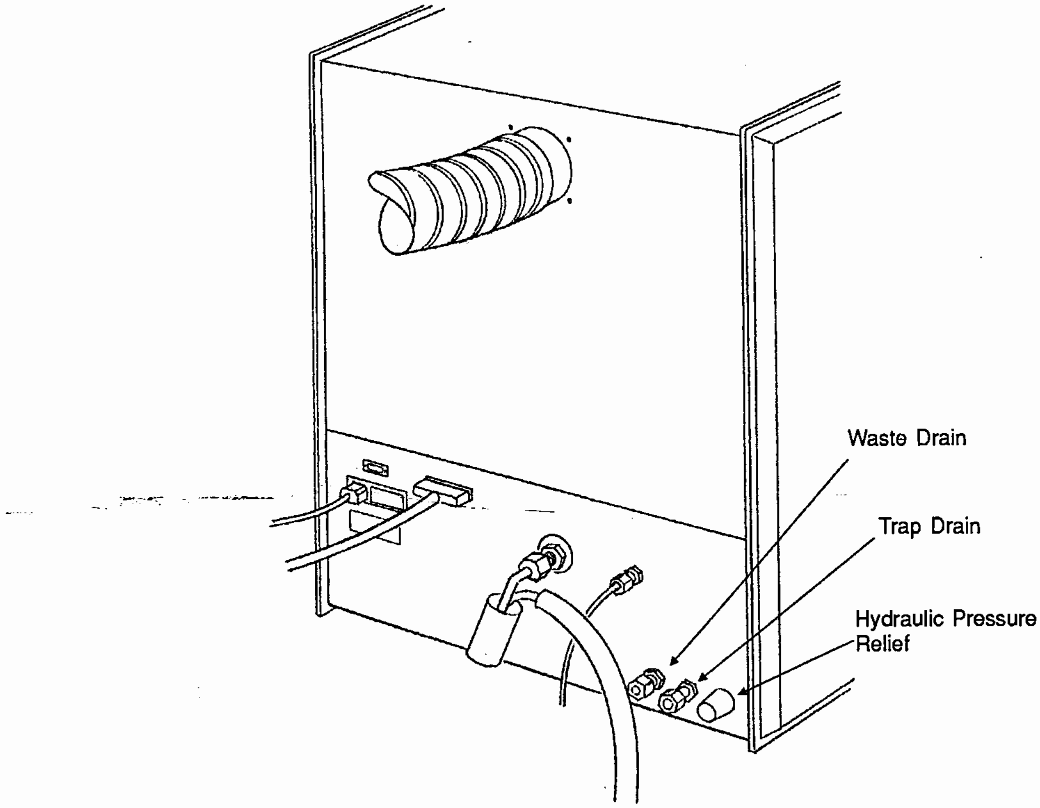


Figure 3-3. Rear Panel Controls

WASTE DRAIN Use this drain to expel mercury that has collected in the mercury spill trap.

TRAP DRAIN When mercury accumulates in the mercury trap located inside the unit, an alarm sounds. Use this drain to expel the mercury collected in the trap.

HYDRAULIC PRESSURE RELIEF This relief valve is released when the pressure exceeds 32,500 psia.

TURNING THE SYSTEM ON AND OFF

TURNING ON THE SYSTEM

Refer to **Turning On the System** in Chapter 2 for instructions on turning on the system.

TURNING OFF THE SYSTEM

The system should not be turned off when an automatic operation is in progress; doing so could cause loss of data. If it becomes necessary to turn off the system while an automatic operation is in progress, you must first cancel the operation before turning off the system. To cancel the operation:

WARNING

Make sure you cancel the current operation before turning off the system. Failure to do so could cause pressure to build to an unsafe level in the system.

- To cancel a low pressure analysis, press F9, F3 from the Main Function Menu and follow the instructions displayed on the screen.
- To cancel a manual high pressure analysis, press F9, F4 from the Main Function Menu and follow the instructions displayed on the screen.
- To cancel an automatic high pressure run, press F9, F5 from the Main Function Menu and follow the instructions displayed on the screen.
- If reports are in progress, press F6, then F4 from the Main Function Menu to display the Cancel Report screen. Press PgDn to cancel the reports.

CAUTION

Always make sure the disk light is off before turning off the control module. Failure to do so could result in loss of data.

1. Press F10 from the Main Function Menu to display the Utilities Menu.
2. Press F10 to display the sign-off screen.
3. Make sure that Drive A is open. Press PgDn.
4. Place the PoreSizer ON/OFF switch in the off (down) position.

5 If the control module is not going to be moved, skip to Step 6. If the control module is to be moved, type **SHIP** and press [↵]. Then press [Y]. This protects the hard disk from damage during movement.

NOTE

Some control modules may use a command other than SHIP, such as DISKSHIP, PARK or RETRACT. Consult the control module operator's manual.

6. Place the control module, monitor, printer, and plotter ON/OFF switches in the OFF position.

ANALYSIS PROGRAM MENU STRUCTURE

All the screens in the Analysis Program are accessed through the Main Function Menu. A menu is a list of tasks that can be performed by the Analysis Program. To choose a task, simply press the function key associated with the task. The appropriate submenu or screen will be displayed.

The following chart shows the menu structure of the Analysis Program. The function key used to access each task is shown next to the task.

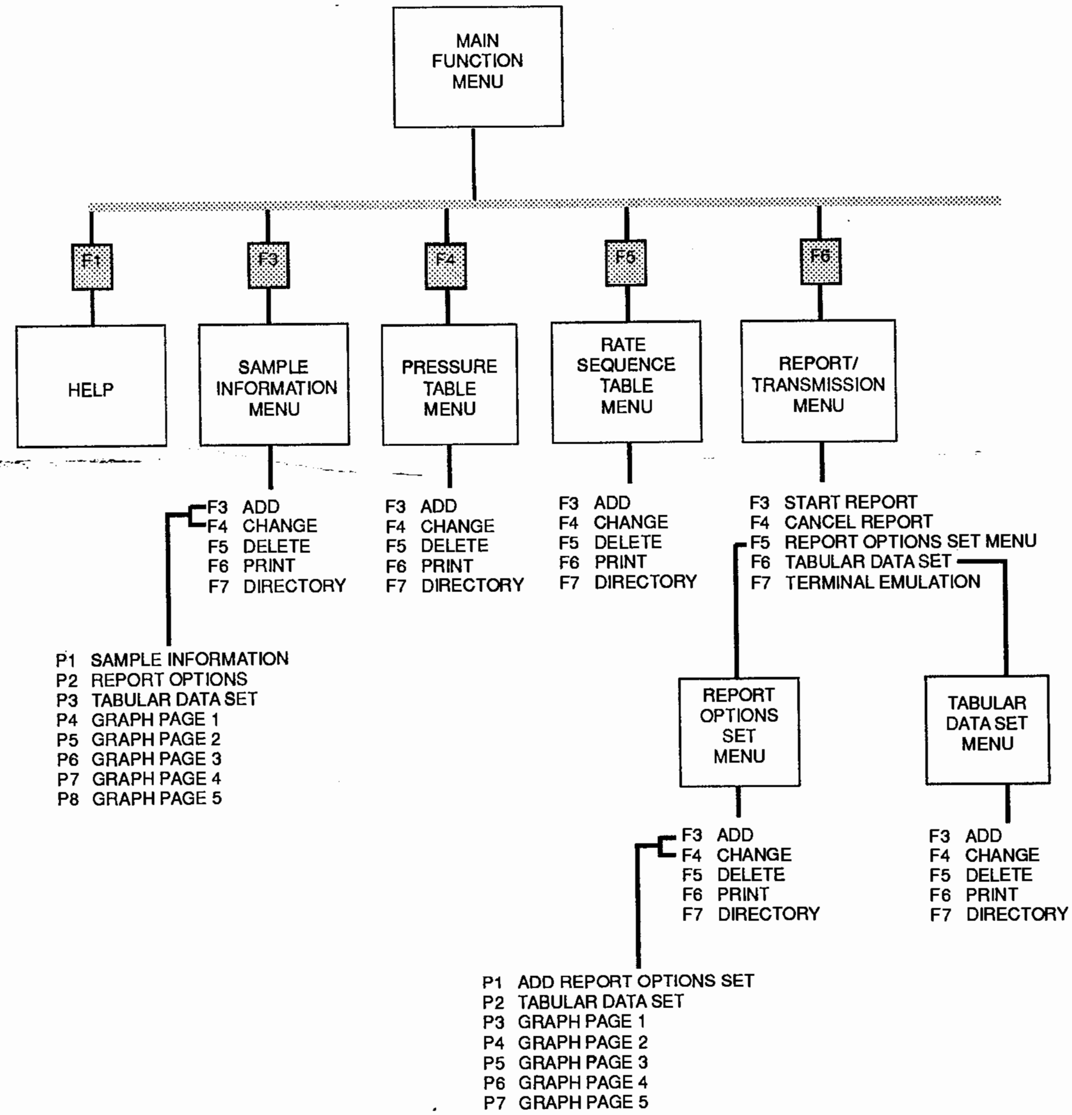
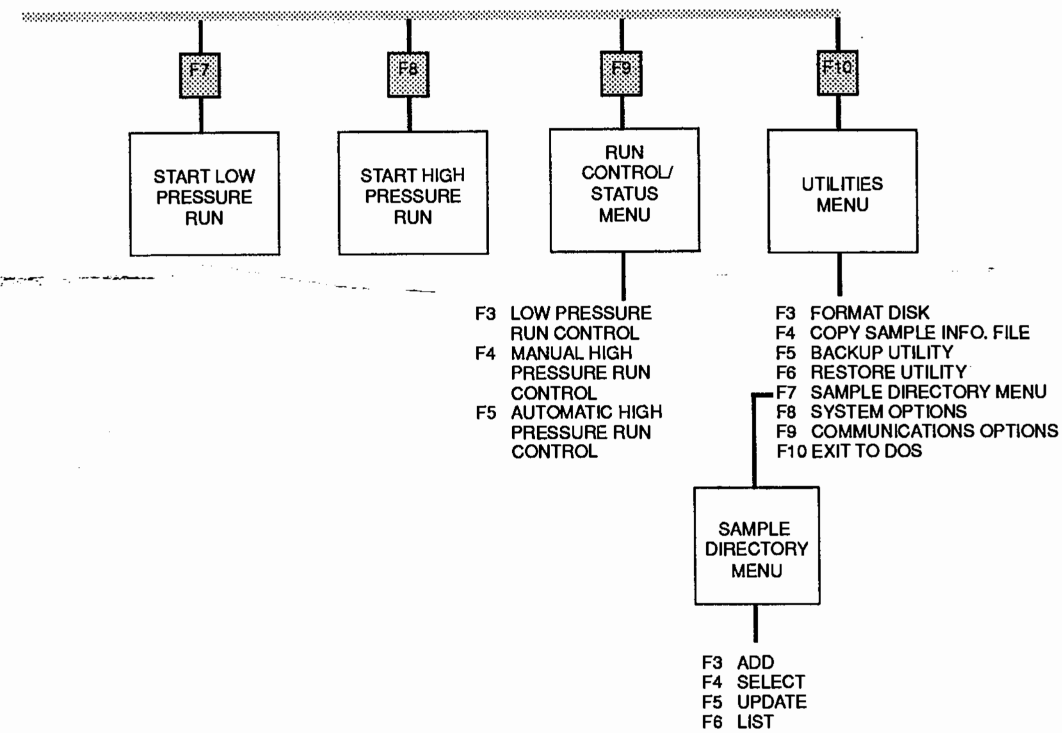


Figure 3-4. Analysis Program Menu Structure



Press F2 from any screen to store information and return to the previous menu.

Figure 3-4. Analysis Program Menu Structure (continued)

USING THE KEYBOARD

Information is entered through the keyboard. Figure 3-5 shows typical keyboards.

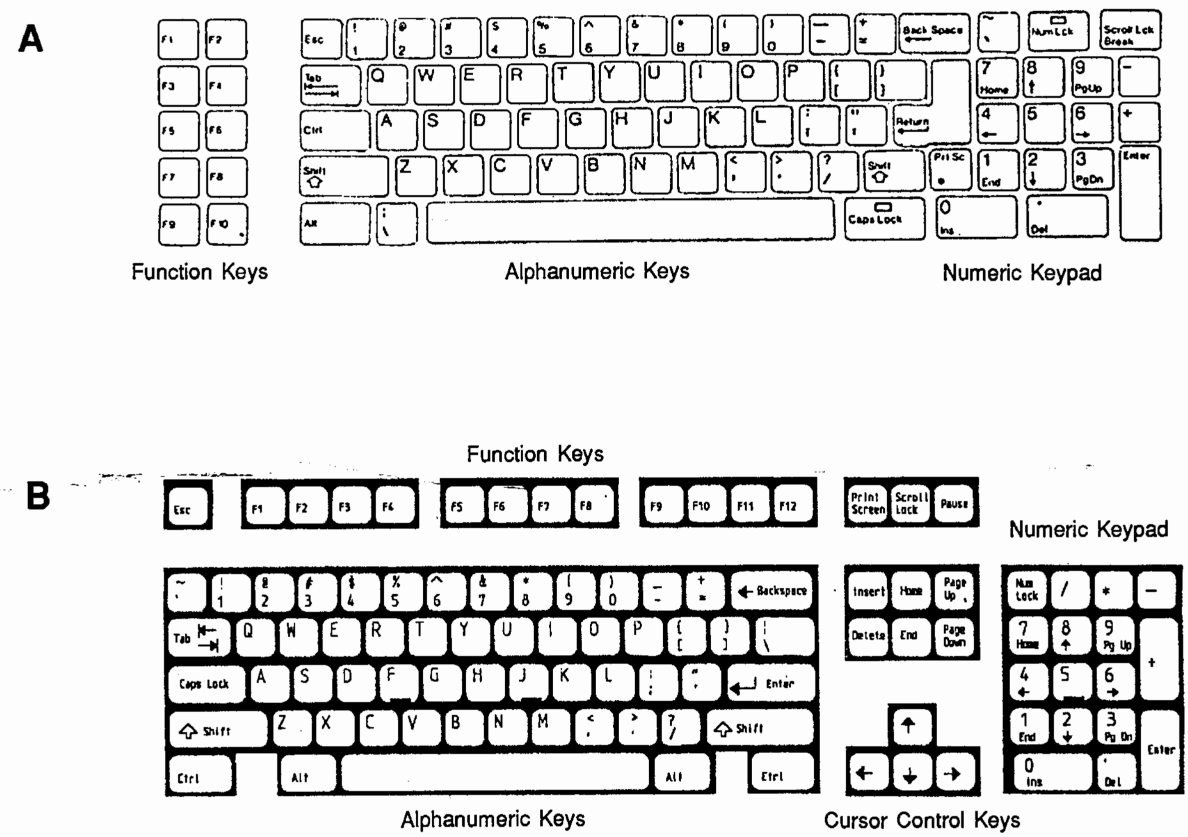


Figure 3-5. Typical Keyboards

The **function keys** are used to select menus and input screens. The **alphanumeric** keys are used to enter upper and lower case letters and numbers. The **cursor control** keys are used to move the cursor from field to field on the input screens.

On some keyboards (refer to Figure 3-5.A), the cursor control keys are located on the **numeric keypad**. The keypad may be operated in one of two modes: numeric or cursor control. When operating the Analysis Program, the keypad must remain in cursor control mode. To change the mode, press Num Lock .

On other keyboards (refer to Figure 3-5.B), a separate keypad also contains cursor control keys. You may use the cursor control keys on this keypad or those on the numeric keypad. In any event, the numeric keypad must remain in cursor control mode when the Analysis Program is running.To change the mode, press Num Lock .

The following table describes the keys used by the Analysis Program.

Table 3-1. Keys Used by the Analysis Program

Key	Description
[Home]	Moves the cursor to the first field on the current page.
[End]	Moves the cursor to the last field on the current page.
[←] [↑] [→] [↓]	Move the cursor to the next field in the direction of the arrow.
[Ins]	Inserts a field in a table.
[PgUp]	Returns to the previous page when a screen contains more than one page.
[PgDn]	Moves to the next page when a screen contains more than one page.
[5] (Cursor Control Keypad)	Toggles data in fields preceded by a question mark (?).
[F2]	Saves information on the current screen and returns to the previous menu.
[Esc]	Discards entries made on the current screen.
[Del]	Deletes all information entered in a field.
[Alt] + [Spacebar]	Restores the screen if it becomes blank due to electrostatic discharge.

THE ANALYSIS PROGRAM INPUT SCREENS

ACCESSING SCREENS

An input screen is a screen that displays analysis information and allows you to change the information.

If more information is required than can be organized on one input screen, other input screens will follow. Each input screen in a series is considered a page. Pages are arranged in a linear sequence and numbered accordingly. Some pages allow branching to other screens which are not in the linear sequence.

A branch from a page is indicated in the screen number by a decimal number following a page number. For example, 3.3p5.4 indicates that a branch was made from page 5 of screen 3.3 by way of the F4 key.

ENTERING DATA IN INFORMATION FILES

Five types of information files are used by the Analysis Program:

Sample Information Files
Pressure Tables
Rate Sequence Tables
Report Options Sets
Tabular Data Sets

The sample information file contains information about a sample, as well as all the information contained in a report options set.

File Number

When entering data in information files, the first field displayed is for a number by which the file is to be identified. For sample information files, the system assigns and displays the next available number. You can retain this default number or enter another number.

File ID

In addition to the file number, you can also enter a description of the file in the File ID field.

Cursor Movement

You can move the cursor to the desired field using the cursor control keys. The selected field is shown in reverse video.

Input fields can be bypassed using the cursor control keys. Information currently displayed in the field will be retained in the file.

Correcting Typographical Errors

If you make a typographical error while entering information in a field, use the following keys:

- Cursor control keys to move to a previously-entered field.
- Del key to erase all the characters in the field.
- Backspace key to erase one or more characters to the left of the cursor.

Toggle Fields

If the entry for a field is limited to certain fixed responses, it is called a toggle field. The program provides the valid entries in a fixed response set. To choose one of the entries, press [5] on the numeric keypad until the correct entry is displayed. Then press [↵] or one of the arrow keys to move to the next field. Toggle fields are indicated by a question mark (?) following the prompt.

Data Entry Fields

Prompts that are followed by a colon (:) require data entry. Some fields require numeric entries (numbers), while others allow alphanumeric entries (letters and numbers). Usually, a default value is shown in the field. Press [↵] to accept the default value or enter another value, then press [↵] to move to the next field. The valid entries for input screens are given in Chapter 6. An invalid entry will cause an error message to be displayed on the bottom of the screen explaining the type of data or range of values required.

Completing Data Entry

When additional pages of information are required to complete the information file, [PgDn] appears in the lower right corner of the screen. If there is a previous page, [PgUp] appears in the upper right corner of the screen. Press [PgDn] to display the next page, [PgUp] to display the previous page.

When you have finished entering information on a page, choose one of the following options:

- To save all the information you entered, press one of the following:

  [PgUp] when on the first page
  [PgDn] when on the last page
  [F2] when on any page

  The previous menu is displayed and the information you entered is written to the information file.

- To delete all the information you entered, press [Esc]. The previous menu is displayed and the information you entered is not written to the information file.

Changing Data in Information Files

The data in information files can be changed by selecting the CHANGE option from the appropriate menu.

NOTE

If a sample is involved and the sample is active, the file cannot be changed.

After selecting the CHANGE option, enter the number of the file to be changed. The contents of the file are then displayed on the screen. You can edit the fields in the same manner as described under **Entering Data in Information Files.**

Deleting Data from Information Files

The data in information files can be deleted by selecting the DELETE option from the appropriate menu.

NOTE

If a sample is involved and the sample is active, the file cannot be deleted.

After selecting the DELETE option, enter the number of the file to be deleted. The contents of the file are then displayed on the screen. Press [PgDn] to delete the file.

STATUS DISPLAY

All screens in the Analysis Program (except help screens and graphs) have the same information displayed at the top of the screen. This collection of information is called the Status Display. A typical Status Display is shown in Figure 3-6.

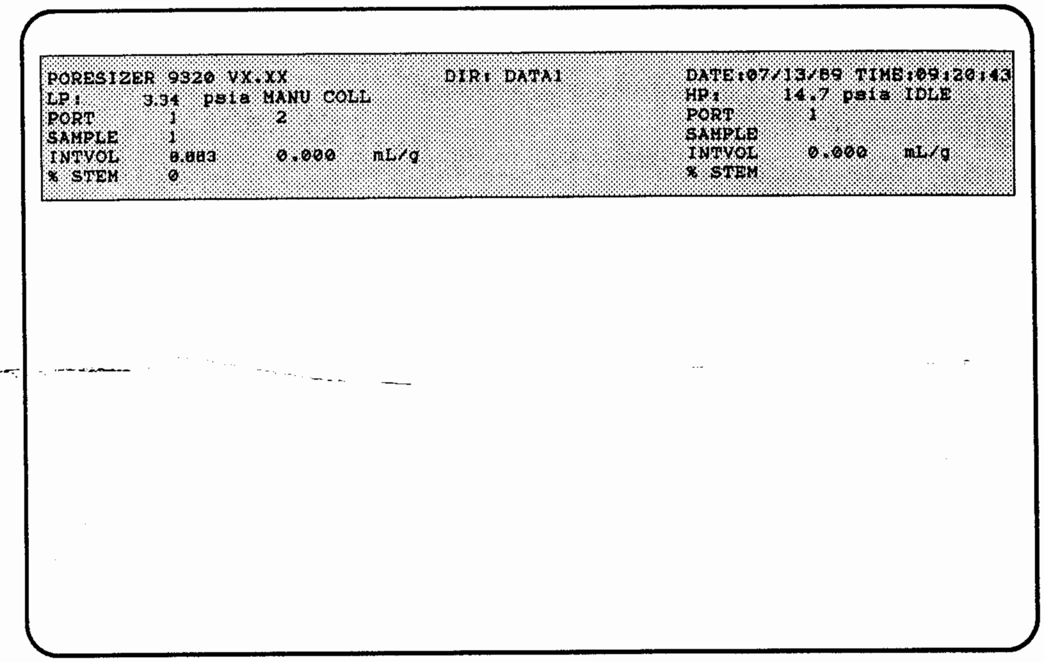


Figure 3-6. Status Display

Each item in the Status Display is described in the following table.

NOTE

All displays of readings from the PoreSizer will be blanked if communication with the PoreSizer is interrupted. Display readings will reappear when communication is restored.

Table 3-2. Status Display

Item	Description
Vx.xx	Software version; x.xx is replaced with the revision number.
DIR	The currently selected sample directory. The current sample directory is selected by means of screen 10.7.4 Select Sample Directory.
DATE	System date. Format: mm\dd\yy. mm = month, dd = day, yy = year. The system date can be set using screen 10.8 System Options. The calendar is backed up by a battery in case of line power failure.
TIME	System time. Format: hh:mm:ss hh = hour, mm = minutes, ss = seconds. The system time can be set using screen 10.8 System Options. The clock is battery-backed also. The time on the display is updated about every second, although the clock is updated every fraction of a second.
LP	The current pressure in the low pressure system, shown in pounds per square inch absolute (psia) or megapascals (MPa). If the pressure is below 500 µmHg, the pressure reading from the vacuum gauge will be shown in µmHg instead. During a low pressure run an abbreviation of the status will be displayed to the right of the pressure display.
HP	The current pressure in the high pressure system, shown in pounds per square inch absolute (psia) or megapascals (MPa). During a high pressure run an abbreviation of the status will be displayed to the right of the pressure display.
PORT	The sample port number. For the low pressure ports: 1 = left port, 2 = right port.
SAMPLE	The sample number currently running on the designated port.
INTVOL	The intrusion volume for the sample currently running on the designated port, shown in mL/g. If there are no samples currently running, the intrusion volume displayed is based on the capacitance reading, a sample weight of one, and a penetrometer constant of ten.

Item	Description
% STEM	The percent of the penetrometer stem that does not currently contain mercury. This value is displayed only during an analysis.
STATUS AND ERROR MESSAGE LINE	Status and error messages are displayed on the bottom line of the status display. If there are no status or error messages, this line is blank. If there is one message, it is constantly displayed on the screen. If there is more than one message, each message is displayed for five seconds. Refer to Chapter 10 for a description of error messages.

BACKING UP FILES

Sample files and system files should be backed up to diskette on a regular basis to ensure that data is not lost in the event of a hard disk problem. Refer to **10.5 Back Up Utility** in Chapter 7 for instructions.

CHAPTER 4

PERFORMING AN ANALYSIS

- Selecting Penetrometers
- Loading the Sample
- Sealing the Penetrometer
- Weighing the Assembled Penetrometer with Sample
- Performing Analyses
- Low Pressure Operation
- High Pressure Operation

PERFORMING AN ANALYSIS

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 4-1 lists parameters and part numbers.

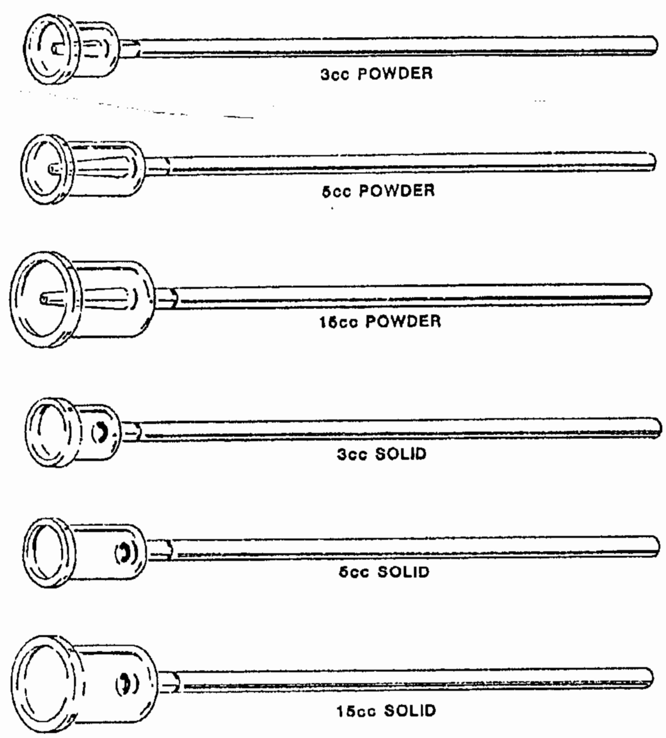
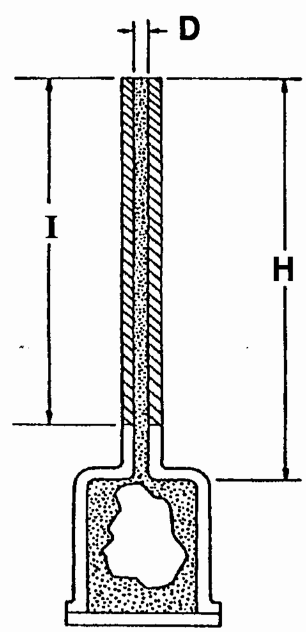


Figure 4-1. Penetrometers

Table 4-1. Penetrometer Selection Guide

Bulb Volume	Sample Type	Maximum Measurable Volume (cc)	Total Stem Volume (cc)	Maximum Head Pressure (psia)	Maximum Head Pressure MPa	Penetrometer Constant (µL/pF)	Physical Dimensions I (mm)	Physical Dimensions H (mm)	Physical Dimensions D (mm)	Part Number
3	Solid	0.387	0.412	4.68	0.0323	10.79	227	242	1.473	920-61713-00
3	Solid	1.116	1.198	4.68	0.0323	21.63	227	242	2.502	920-61715-00
3	Powder	0.387	0.412	4.68	0.0323	10.79	227	242	1.473	920-61714-00
3	Powder	1.116	1.198	4.68	0.0323	21.63	227	242	2.502	920-61716-00
5	Solid	0.366	0.392	4.45	0.0307	10.79	215	230	1.473	920-61707-00
5	Solid	1.057	1.131	4.45	0.0307	21.63	215	230	2.502	920-61709-00
5	Solid	1.716	1.836	4.45	0.0307	27.82	215	230	3.188	920-61711-00
5	Powder	0.366	0.392	4.45	0.0307	10.79	215	230	1.473	920-61708-00
5	Powder	1.057	1.131	4.45	0.0307	21.63	215	230	2.502	920-61710-00
5	Powder	1.716	1.836	4.45	0.0307	27.82	215	230	3.188	920-61712-00
15	Solid	0.366	0.392	4.45	0.0307	10.79	215	230	1.473	920-61701-00
15	Solid	1.057	1.131	4.45	0.0307	21.63	215	230	2.502	920-61703-00
15	Solid	1.716	1.836	4.45	0.0307	27.82	215	230	3.188	920-61705-00
15*	Solid	3.007	(3.263)	4.45	0.0307	33.13	215	230	4.250	920-61724-00
15*	Solid	3.857	(4.185)	4.45	0.0307	34.48	215	230	4.813	920-61725-00
15	Powder	0.366	0.392	4.45	0.0307	10.79	215	230	1.473	920-61702-00
15	Powder	1.057	1.131	4.45	0.0307	21.63	215	230	2.502	920-61704-00
15	Powder	1.716	1.836	4.45	0.0307	27.82	215	230	3.188	920-61706-00



*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) = $[H] \times [0.01934$ psia/mmHg$]$

Maximum Head Pressure (MPa) = $[H] \times [0.000133$ 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.

CAUTION
The larger penetrometers (15 cc volume) are limited to a maximum of about 25 psia in the low pressure tests before leakage of mercury becomes probable.

Best results, generally, will be 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% or be less than 25% of the maximum measurable intrusion volume of the penetrometer (see Table 4-1, Column 3). 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 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% is to be analyzed. 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 4-1 as solid, 5 cc sample volume, 1.057 cc maximum measurable intrusion 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/1.057 cc) x 100% = 59%, 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 4-1 will contain 7.37 cc of sample. Considering the sample size, the one having a maximum measurable intrusion volume of 0.366 cc is most appropriate. The sample requires approximately 34% [0.13/0.36] of the intrusion 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 70% [0.26/0.366] of the maximum measurable intrusion volume.

NOTE

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 um 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.

DETERMINING THE PENETROMETER VOLUME

If density is to be measured, the calibrated empty volume of each penetrometer must be entered on the Add Sample screen.

To determine the penetrometer volume, use the Penetrometer Volume Calibration form (Figure 4-2). A blank form is included in Appendix A. The volume determination need be made only once if penetrometer components are kept together and a record maintained.

The procedure for obtaining a penetrometer filled with mercury is the same as that of performing a low pressure analysis except no sample is loaded into the penetrometer.

PENETROMETER VOLUME CALIBRATION

Penetrometer: ______________________ Date: ______________________
Number: ______________________ 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 ($\bar{V}$) . . . . _______ 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: ____
____
____

micromeritics

Figure 4-2. Penetrometer Volume Calibration

CLEANING PENETROMETERS

Clean and dry penetrometers are essential for accurate, reproducible results. The following steps are recommended:

WARNING
When handling penetrometers, it is recommended that rubber gloves be worn.

1. Dissolve Alconox® (or other suitable detergent) in water. Make sure the detergent is completely dissolved before placing the penetrometer into water.
2. Place the mercury waste container in a shallow pan of water in case of spills.
3. Hold the penetrometer upright over the mercury waste container to allow any accumulated mercury to drain out.

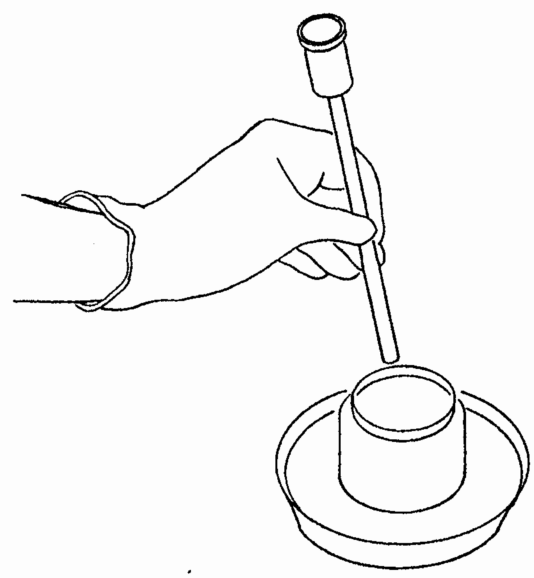


Figure 4-3. Cleaning Penetrometers

4. Remove the cap from the penetrometer.
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 cap, spring, and insulator in the detergent solution. Clean with appropriate brushes and rinse in warm water; make sure that water flows freely through the opening in the cap. Then rinse in isopropyl alcohol.

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 and cap.

LOADING THE SAMPLE

1. Enter the sample ID, submitter ID, and operator ID on a Sample Data Sheet (refer to Figure 4-4). A blank Sample Data Sheet 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.

micromeritics

Date: ____________________

Current Directory: ____________________

PoreSizer 9320
Sample Data Sheet

Press F3 twice from the Main Function Menu to display the Add Sample Information screen.

3.3p1 Add Sample Information

	Port 1	Port 2	
Sample number			(1 to 400)
Sample ID			(40 char. max)
Submitter ID			(40 char. max)
Operator ID			(20 char. max)
Penetrometer number			(10 char. max)
Penetrometer constant			(10.790 µL/pF
Penetrometer weight			(1.0000 g)
Maximum head pressure			(4.680 psi)
Penetrometer volume			(1.0000 mL)
Stem volume			(0.4120 mL)
Advancing contact angle			(130.00 deg)
Receding contact angle			(130.00 deg)
Hg surface tension			(485.0 dynes/cm)
Hg density			(13.5335 g/mL)
Sample weight			(1.0000 g)
Sample + penetrometer weight			

Press PgDn.

3.3p2 Add Sample - Report Options

Report options set number ____________________ (1 to 50)

Press PgDn.

Shaded Value = Default

Figure 4-4. Sample Data Sheet (front page)

SEALING THE PENETROMETER

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:

> **CAUTION**
>
> **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.

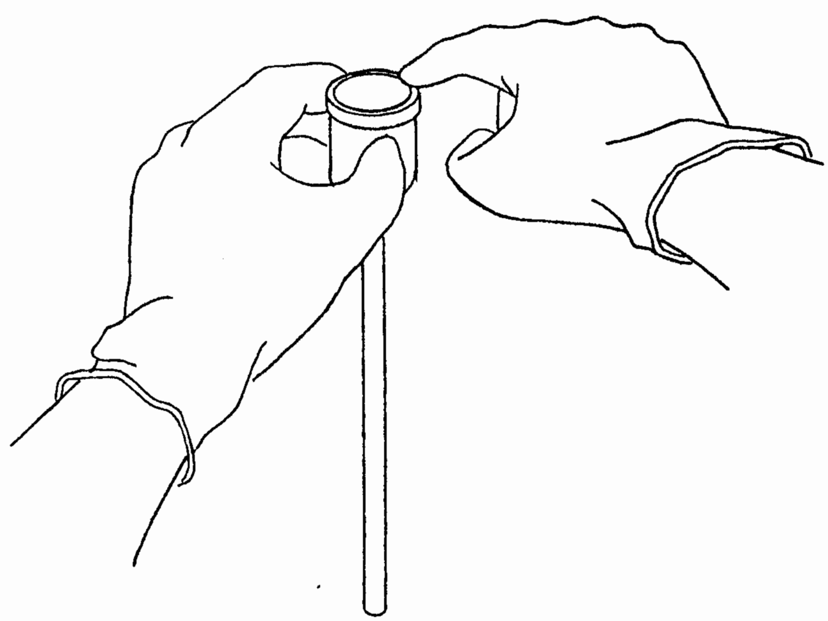


Figure 4-5. Greasing the Penetrometer

   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 seat the cap. Then turn the cap one-half turn to seal.

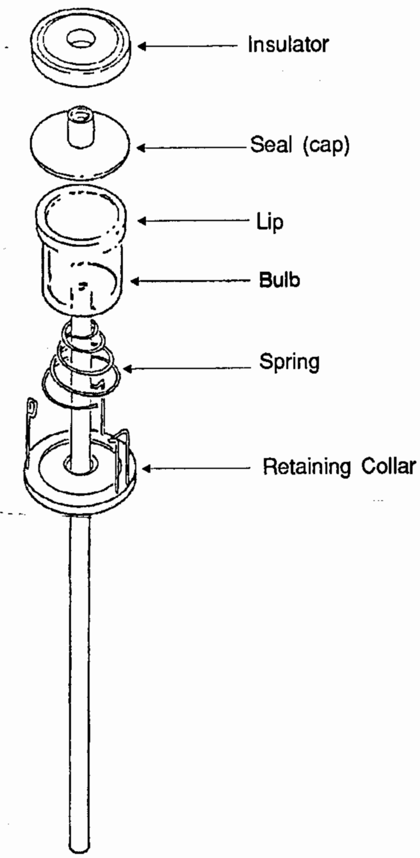


Figure 4-6. Penetrometer Components

3. Insert the insulator over the cap.

4. Slide the spring over the stem and the bulb. Lock into position by pressing the cap down slightly until the spring clamps are securely in place in the groove of the cap as shown in Figure 4-7.

5. Weigh the sample and penetrometer as described in the next section before installing in the low pressure port.

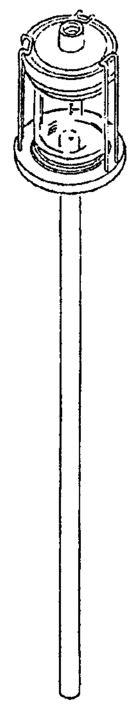


Figure 4-7. Assembled Penetrometer

6. If you are using a 3-cc penetrometer, slide the spacer over the stem.

WEIGHING THE ASSEMBLED PENETROMETER WITH SAMPLE

1. Weigh the assembled penetrometer with sample using an analytical balance.

2. Record the weight on the sample data sheet as **Sample + Penetrometer weight.**

3. Subtract Sample weight from Sample + penetrometer weight; record on the sample data sheet as **Penetrometer weight.**

PERFORMING ANALYSES

The following sections contain instructions for operating the PoreSizer in three modes:

- Without control module assistance

  The instructions describe how to perform both low pressure and high pressure analyses without a control module. Appendix A contains the following forms that can be used for manual analyses: Pore-Size Distribution Data form and Density Determination by Mercury Porosimetry form. Appendix H contains sample forms and formulas used for data reduction.

- With manual control module assistance

  The instructions pertain to operating the PoreSizer using the control module for data collection, but not for control. The control module will also perform data reduction and produce reports. Sample information files must be entered before control module data collection can be performed. Refer to Chapter 6 for more information on sample information files.

- With automatic control module assistance

  The instructions pertain to operating the PoreSizer using the control module to control a high pressure analysis. Sample information files and pressure tables must be entered before automatic high pressure analyses can be performed. If a rate sequence table is to be used rather than an equilibration time, a rate sequence table must be entered also. Refer to Chapter 6 for more information on sample information files, pressure tables and rate sequence tables.

LOW PRESSURE OPERATION

INSTALLING PENETROMETERS IN LOW PRESSURE PORTS

The PoreSizer is designed to perform low pressure analyses on two samples at one time. If only one sample is to be analyzed, a blank rod must be installed in the unused low pressure port. If two penetrometers or one penetrometer and a blank rod are not installed, vacuum conditions cannot be achieved and an analysis cannot be performed. The following instructions refer to one penetrometer; the second is installed in the same manner.

NOTE

These instructions assume installation without prior samples in place. When previous low pressure testing has just been completed, penetrometers have to 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 stem (using a silicone high vacuum grease) on the outside. Wipe off any grease that might get on the flat end of the stem, being careful not to get grease inside the stem.

2. Remove the capacitance detector from the low pressure port by turning it counterclockwise and pulling forward. Lay it on the shelf-like tray on top of the PoreSizer.

3. Turn the retaining knob on the low pressure port counterclockwise until it turns with little resistance. Do not remove the knob; internal components may be disoriented.

4. Insert the penetrometer stem into the port and push it in as far as it will go.

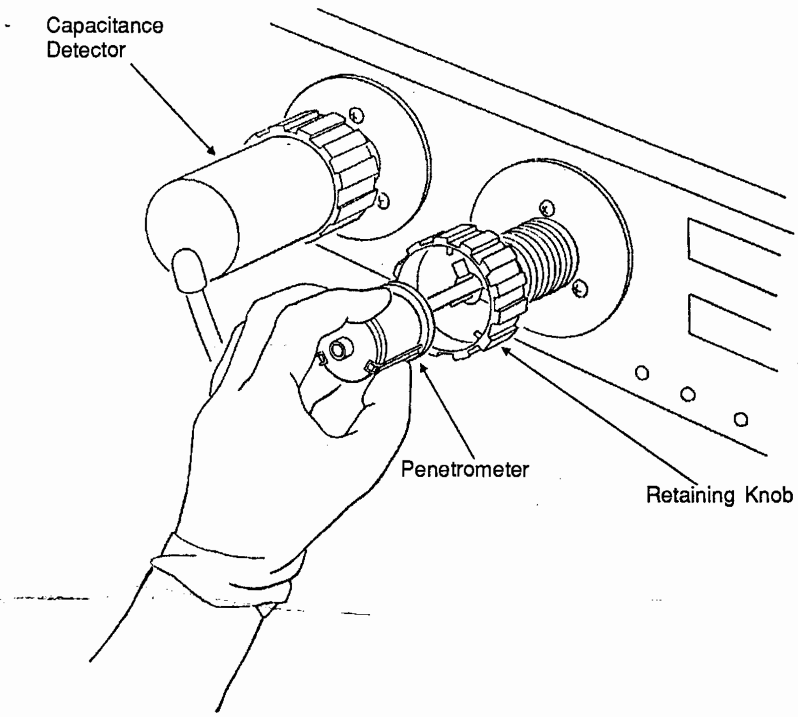


Figure 4-8. Installing Penetrometer in Low Pressure Port

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

6. Before beginning an analysis, check the RESERVOIR LOW indicator on the front panel. If the light is off, sufficient mercury for at least two samples is available. If the light is on, mercury must be added; refer to **Maintaining Mercury Level** in Chapter 8.

FILLING PENETROMETERS WITH MERCURY

1. Ensure that two penetrometers or one penetrometer and one blank rod are installed in the low pressure ports as described in the previous section.

2. Press the EVACUATE SAMPLE SLOW and LOW psia PRESSURE TRANSDUCER buttons.

3. Turn on power switches for both the PoreSizer and the vacuum pump if not already on.

4. When the indicated pressure drops below 1 psia, release the EVACUATE SAMPLE SLOW button and press the EVACUATE SAMPLE MED and μmHg PRESSURE TRANSDUCER buttons.

CAUTION

High surface area samples such as carbon black, fine silica, controlled pore glass, etc. require gentler evacuation. Such materials will easily fluidize and could be drawn into the plumbing and valves. To avoid sample fluidization, the sample must be allowed to attain a lower pressure, about 0.5 psia, under slow evacuation. Press the EVACUATE SAMPLE MED button partially in until you hear the valve open; do not latch the button in. Watch the sample. If it begins to fluidize, release the EVACUATE SAMPLE MED button and allow slow evacuation to continue for a few more minutes. When safe medium evacuation may continue, press the μmHg PRESSURE TRANSDUCER and the EVACUATE SAMPLE MED buttons. Release the EVACUATE SAMPLE SLOW button.

5. When the indicated pressure drops to 250 μmHg, release the EVACUATE SAMPLE MED button and press the EVACUATE SAMPLE FAST button.

6. Allow evacuation to continue until the vacuum on the sample stabilizes. This vacuum pressure must be less than 50 μmHg. If it is not, refer to Table 8-1 in Chapter 8 for possible causes and corrective actions.

7. Replace low pressure port capacitance detectors when vacuum conditions have stabilized. Turn the detectors clockwise to tighten the closure.

8. Press the MERCURY FILL button momentarily and release it. If there is a loss of vacuum, press down on the MERCURY RESERVOIR cap; it must be tightly sealed.

   If there is still a loss of vacuum, release the MERCURY FILL button and wait until a good vacuum is established, perhaps five minutes. Repeat the procedure until the MERCURY FILL button can be held in without loss of vacuum. Hold the button in until the MERCURY UP indicator comes on. Release the MERCURY FILL button. If the indictor turns off, press the MERCURY FILL button until the indicator stays on.

CAUTION

Release the button as soon as the light comes on.

9. Release the EVACUATE SAMPLE FAST button and shift from μmHg PRESSURE TRANSDUCER to LOW psia PRESSURE TRANSDUCER.

10. Press and hold the ADJUST LOW PRESSURE button until the desired filling pressure is reached (usually 0.5 to 1.5 psia).

11. Press and latch the EVACUATE RESERVOIR button and wait 20 to 30 seconds or until the sound from the vacuum pump returns to its normal operating level. Do not release the EVACUATE RESERVOIR button.

CAUTION

Do not release the EVACUATE RESERVOIR button until the sound from the vacuum pump returns to its normal operating level. If you release the button prematurely, you will risk backing up mercury in the system.

12. Press and hold down the MERCURY DRAIN button until the MERCURY DOWN light comes on.

13. Release the MERCURY DRAIN and EVACUATE RESERVOIR buttons.

PERFORMING LOW PRESSURE ANALYSES WITHOUT CONTROL MODULE ASSISTANCE

NOTE

This procedure cannot be performed while a control module is attached and running the Analysis Program as the front panel digital display is disabled. If a control module is attached, exit the Analysis Program before starting this procedure and then turn the PoreSizer off and back on. See Chapter 7 for instructions on exiting the Analysis Program.

1. Fill out the top portion of a Pore Size Distribution Data Sheet (see Appendix H).

2. Ensure that two penetrometers or one penetrometer and one blank rod are installed in the low pressure ports and are properly evacuated and filled with mercury. (Refer to **Filling Penetrometers With Mercury** earlier in this chapter.)

3. Press the LEFT PENETROMETER LOCATION button if the sample is in the left low pressure port; otherwise, press the RIGHT button. Record the values shown in the PRESSURE and INTRUSION displays on the Pore Size Distribution Data form.

4. Begin the low pressure intrusion test by pressing the ADJUST LOW PRESSURE button momentarily to create a series of pressures up to 1 atmosphere (approximately 14.7 psia). Record pressure and intrusion readings for each setting.

5. Continue the series of increasing pressures to between 22 and 25 psia by pressing the ADJUST LOW PRESSURE button and using an external source of gas pressure. Record these data.

6. Return the system to atmospheric pressure by lightly tapping the SLOW EVACUATION button until a pressure between 15-16 psia is reached.

7. The low pressure run is now complete. Remove the penetrometers as described under **Removing Penetrometers From Low Pressure Ports** later in this chapter.

PERFORMING LOW PRESSURE ANALYSES WITH MANUAL CONTROL MODULE ASSISTANCE

Sample information files must be entered before control module data collection can be performed. Refer to Chapter 6 for more information on sample information files.

1. From the Main Function Menu, press [F7] to display the Start Low Pressure Run screen. The following screen is displayed.

```
PORESIZER 9320 VX.XX              DIR: DATA1            DATE:07/13/89 TIME:09:44:23
LP:     14.70 psia IDLE                                 HP:      14.7 psia IDLE
PORT      1         2                                   PORT       1
SAMPLE                                                  SAMPLE
INTVOL    0.000     0.000    mL/g                       INTVOL     0.000    mL/g
% STEM                                                  % STEM

                         7p1 START LOW PRESSURE RUN

                PORT1       PORT2

Sample number:   2           3

Start report after run? yes




Press [PgDn] to start run, or [Esc] for no action.
```

Figure 4-9. Start Low Pressure Run Screen

2. Enter the sample number for the appropriate port and answer the **Start report after run?** and **Report destination?** prompts. Then press [PgDn].

3. Screens containing instructions for the low pressure run are displayed. Follow the instructions on the screens (refer to **Chapter 6, Entering and Maintaining Analysis Information,** for an illustration of these screens).

4 Press [F9], [F3] from the Main Function Menu to display the Low Pressure Run Control screen.

```
PORESIZER 9320 VX.XX              DIR: DATA1              DATE:07/13/89 TIME:09:20:43
LP:        3.34  psia  MANU COLL                           HP:      14.7 psia  IDLE
PORT       1           2                                   PORT     1
SAMPLE     1                                               SAMPLE
INTVOL     0.003       0.000    mL/g                       INTVOL      0.000    mL/g
% STEM     0                                               % STEM

                         9.3 LOW PRESSURE RUN CONTROL

                    Low Pressure Sample Directory: DATA1
Point number   Pressure( psia )   Left Intrusion( mL/g )   Right Intrusion( mL/g )
     1)              1.58                0.000                      0.000
                     2.17                0.001                      0.000
                     3.34                0.003                      0.000



   F2 - Run Control/Status Menu      F3 - Record Data      F4 - Finish Run
```

Figure 4-10. Low Pressure Run Control Screen

Ensure that two penetrometers or one penetrometer and one blank rod are installed in the low pressure ports and are properly evacuated and filled with mercury. (Refer to **Filling Penetrometers With Mercury** earlier in this chapter.)

Observe the low pressure shown in the status display on the video monitor. Record data as follows:

a. Press the ADJUST LOW PRESSURE button on the front panel of the PoreSizer while observing the pressure reading in the status display.

b. Release the ADJUST LOW PRESSURE button and press F3 to record a pressure point.

c. Continue to record pressure points as described above. When complete, press F4. The following message is displayed:

Press [PgDn] to finish run or [Esc] for no action.

Choose one of the following:

- Press PgDn if you wish to end the run. Screen 9.3.4 will be displayed. Follow the instructions on the screen.
- Press Esc to continue the run.

5. Remove the penetrometer as described below.

REMOVING PENETROMETERS FROM LOW PRESSURE PORTS

1. Make sure that the low pressure ports have returned to atmospheric pressure.

2. Turn the capacitance detector counterclockwise to loosen, then remove it.

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

4. 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. Record this weight on the Sample Data Sheet.

HIGH PRESSURE OPERATION

INSTALLING THE PENETROMETER IN THE HIGH PRESSURE CHAMBER

1. Press the PRESSURE SCAN switch to the DOWN position until the INTENSIFIER LIMIT LOW light comes on. Then return the PRESSURE SCAN switch to the center position.

2. Turn the vent valve knob counterclockwise to release.

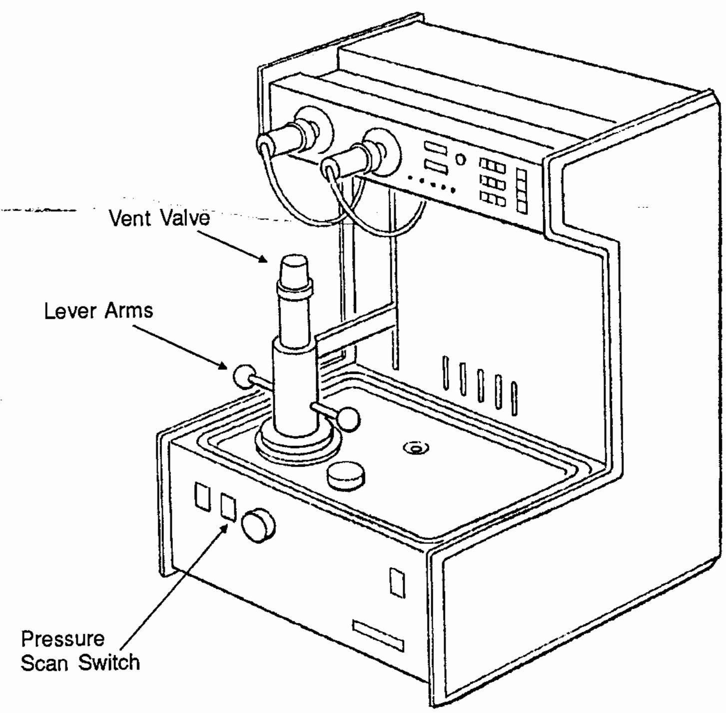


Figure 4 -11. Opening the High Pressure Chamber

3. Unscrew the chamber cap by turning the lever arms counterclockwise. Lift the chamber cap upward as far as it will go.

4. Gently lower the penetrometer assembly, bulb down, into the chamber.

5. Guide the penetrometer stem upward into the chamber cap.

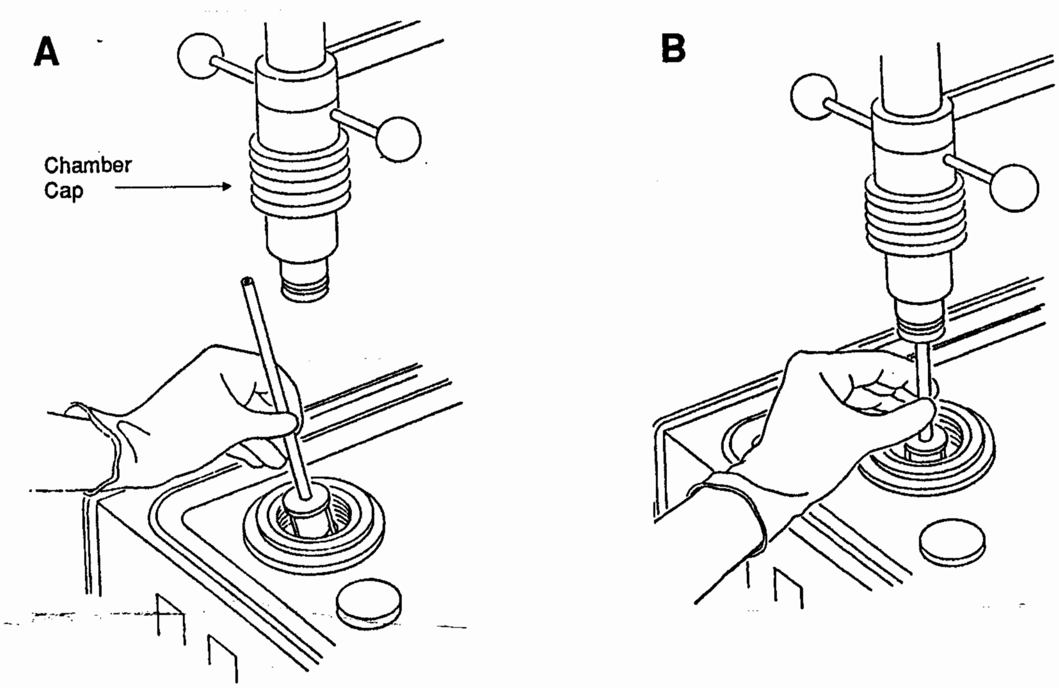


Figure 4-12. Installing the Penetrometer in the High Pressure Chamber

6. Lower the chamber cap 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 cap.

7. Check the high pressure fluid level. If the level is significantly below the visible ledge, add high pressure fluid to bring the level nearly to the ledge. If the level is above the ledge, remove fluid to bring the level to just below the ledge.

8. Lower the chamber cap all the way, then slowly turn the arms clockwise until the cap is completely seated.

CAUTION

Do not turn the arms rapidly. Doing so could generate significant transient pressure which could distort results in the 15-25 psia region.

9. While tightening the chamber cap, note, particularly on the last two turns, whether air bubbles or oil are extruded into the small plastic cup on top of the cap. At first, air bubbles should appear, but later only oil should be observed. Tighten, then loosen the arms two or three times to remove air bubbles. If air bubbles are still present, it means insufficient high pressure fluid was present in the chamber initially. Pour more fluid into the plastic cup and repeat step 9 until all air bubbles are eliminated.

PERFORMING A HIGH PRESSURE ANALYSIS WITHOUT CONTROL MODULE ASSISTANCE

NOTE

This procedure cannot be performed while a control module is attached and running the Analysis Program as the front panel digital display is disabled. If a control module is attached, exit the Analysis Program before starting this procedure and turn the PoreSizer off and back on. See Chapter 7 for instructions on exiting the Analysis Program.

1. Install the penetrometer assembly in the high pressure chamber as described in the previous section. Press the HIGH psia PRESSURE TRANSDUCER button and the HIGH PENETROMETER LOCATION button.

2. Adjust the SET ATM PRESSURE knob until atmospheric pressure is displayed.

3. Close the vent valve on top of the chamber cap.

4. Choose one of the following options:

- If continuous pressure generation is desired:

   a. Take one set of pressure and intrusion values at the highest pressure recorded in the low pressure portion of the analysis (minus the head correction, see Appendix D). Adjust the RATE knob as appropriate to set the rate of pressure generation.

   b. Press the PRESSURE SCAN UP button, wait for the desired equilibration time, and record the data.

   c. Stop the pressure generation at 30,000 psia by placing the PRESSURE SCAN switch in the center (OFF) position.

   d. Take extrusion data if desired by pressing the PRESSURE SCAN DOWN button and recording values at intervals.

- If equilibrium data are desired:

   a. Take one set of pressure and intrusion values at the highest pressure recorded in the low pressure portion of the analysis. Adjust the RATE knob as appropriate to set the rate of pressure generation.

   b. Press the PRESSURE STEP UP button momentarily to set increasing pressures. Record the desired data.

c. Stop the pressure generation when 30,000 psia is attained.

d. For equilibrium extrusion data, press the PRESSURE STEP DOWN button at intervals and record values.

5. Press the PRESSURE SCAN DOWN button until the LOWER LIMIT indicator comes on.

6. Remove the penetrometer as described under **Removing the Penetrometer From the High Pressure Chamber**.

PERFORMING A HIGH PRESSURE ANALYSIS WITH MANUAL CONTROL MODULE ASSISTANCE

NOTE

Control module data collection for high pressure analysis requires that low pressure data have been previously collected for the sample and stored in a sample information file.

1. Install the penetrometer in the high pressure chamber as described in **Installing the Penetrometer in the High Pressure Chamber** earlier in this chapter.

2. From the Main Function Menu, press [F8] to display the Start High Pressure Run screen. The following screen is displayed.

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/13/89 TIME:09:57:14
LP:     14.70 psia IDLE                               HP:      14.7 psia IDLE
PORT       1          2                               PORT        1
SAMPLE                                                SAMPLE
INTVOL     0.000      0.000    mL/g                   INTVOL      0.000    mL/g
% STEM                                                % STEM

                         8p1 START HIGH PRESSURE RUN

Sample number:     1

Run type? manual

Start report after run? yes

Atmospheric pressure:  14.7   psia

Sampwt+Pntrwt+Hgwt:     1.0000  g
```

Figure 4-13. Start High Pressure Run Screen

3. Enter the sample number and answer the other prompts on the screen (select **manual** for run type). Then press [PgDn].

4. Screens containing instructions for the high pressure run are displayed. Follow the instructions on the screens (refer to **Chapter 6, Entering and Maintaining Analysis Information** for an illustration of these screens).

5. Press [F9], [F4] from the Main Function Menu to display the Manual High Pressure Run Control screen.

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/13/89 TIME:09:55:17
LP:     14.70 psia IDLE                               HP:      101.0 psia MANU COLL
PORT      1         2                                 PORT       1
SAMPLE                                                SAMPLE     2
INTVOL    0.000     0.000    mL/g                     INTVOL     0.000    mL/g
% STEM                                                % STEM     0

                    9.4 MANUAL HIGH PRESSURE RUN CONTROL

                   High Pressure Sample Directory: DATA1
      Point number          Pressure( psia )         Intrusion Volume( mL/g )
         MATCH                    30.3                          0.000
          (21)                   104.8                          0.005




     F2 - Run Control/Status Menu       F3 - Record Data      F4 - Finish Run
```

Figure 4-14. Manual High Pressure Run Control Screen

Observe the high pressure shown in the status display. Record data as follows:

a. Use the PRESSURE STEP or SCAN switch on the front panel of the PoreSizer to adjust the high pressure, while observing the pressure reading in the status display on the video monitor.

b. Release the PRESSURE STEP or SCAN switch and press [F3] to record a pressure point. If continuous pressure generation is desired, the SCAN switch may be left on during data recording.

c. Continue to record pressure points as described above. When complete, press [F4]. The following message is displayed:

   Press [PgDn] to finish run or [Esc] for no action.

   Choose one of the following:

   - Press [PgDn] if you wish to end the run. Screen 9.4.4 will be displayed. Follow the instructions on the screen.
   - Press [Esc] to continue the run.

6. Remove the penetrometer as described under **Removing the Penetrometer from the High Pressure Chamber.**

PERFORMING AN AUTOMATIC HIGH PRESSURE ANALYSIS

NOTE

Control module data collection for high pressure analysis requires that low pressure data have been previously collected for the sample and stored in a sample information file.

Pressure tables must be entered before an automatic high pressure analysis can be performed. If a rate sequence table is to be used, a rate sequence table must be entered also. (Refer to Chapter 6.)

1. Install the penetrometer in the high pressure chamber as described in **Installing the Penetrometer in the High Pressure Chamber** earlier in this chapter.

2. From the Main Function Menu, press F8 to display the Start High Pressure Run screen. The following screen is displayed.

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/14/89 TIME:06:45:49
LP:     14.70 psia IDLE                               HP:     14.7 psia IDLE
PORT        1         2                               PORT        1
SAMPLE     31        32                               SAMPLE
INTVOL     0.000     0.000    mL/g                    INTVOL     0.000     mL/g
% STEM                                                % STEM

                         8p1 START HIGH PRESSURE RUN

Sample number:     1                       Pressure table number:  1

Run type?  automatic                       Run method?  equilibrated

Start report after run?  yes               Equilibration time:       10  seconds

Report destination?  printer               Maximum intrusion volume:    0.000  mL/g

Atmospheric pressure:  14.7000  psia       Last collected point:  14.7000    psia

Sampwt+Pntrwt+Hgwt:     1.0000  g
```

Figure 4-15. Start High Pressure Run Screen

3. Enter the sample number and answer the other prompts on the screen (select **automatic** for run type). Then press PgDn.

4. If you wish to monitor the run, press F9, F5 to display the Auto High Pressure Run Control/Status screen.

5. Remove the penetrometer as described below.

REMOVING THE PENETROMETER FROM THE HIGH PRESSURE CHAMBER

1. Make sure the INTENSIFIER LIMIT LOW indicator is on. When the indicator is on, open the vent valve and loosen the sealing valve at the top of the high pressure chamber by turning the arms counterclockwise.

2. Lift the chamber cap up as far as it will go. Retained high pressure fluid will drain back into the chamber.

3. Remove the penetrometer assembly by lifting it upward. Hold it a few moments over the chamber to allow the high pressure fluid to drain.

4. Clean the penetrometer as described earlier in this chapter (refer to **Cleaning Penetrometers**).

CHAPTER 5

PRINTING REPORTS

- How to Print Reports
- How to Cancel Reports
- Sample Reports

PRINTING REPORTS

The Start Report screen enables you to generate reports to be printed on the printer or the plotter, displayed on the screen, or sent to the serial line, to drive a: or to drive c:.

NOTE

Reports can be printed only for analyses that were performed using the control module.

This chapter describes how to print and cancel reports. It also contains samples of the reports generated by the Analysis Program.

HOW TO PRINT REPORTS

To access the Start Report screen from the Main Function Menu, press F6, then F3.

```
PORESIZER 9320 VX.XX            DIR: DATA1            DATE:07/13/89 TIME:10:02:0
LP:     14.70 psia IDLE                               HP:     14.7 psia IDLE
PORT      1        2                                  PORT      1
SAMPLE                                                SAMPLE
INTVOL    0.000    0.000    mL/g                      INTVOL    0.000    mL/g
% STEM                                                % STEM

                          6.3 START REPORT

Sample number:     2

Report destination?  printer

Type of data?  reduced




Press [PgDn] to produce report, or [Esc] for no action.
```

FIELD	DESCRIPTION
Sample number:	*Range:* *1 to 400* Enter a sample file number that exists in the current sample directory.
Report destination?	*Toggle* *Choices:* *printer*, *printer/plotter*, *serial line, screen*, *drive a:, drive c:* If you choose drive a: or c:, the report is written to a file named: (directory name).(sample number) For example, for the directory DATA1, sample number1, the name would be: **DATA1.001**.
Type of data?	*Toggle* *Choices:* *reduced or unreduced*

DISPLAYING REPORTS ON THE SCREEN OR SENDING REPORTS TO THE SERIAL LINE

1. On the Start Report screen, enter the sample file number.

2. Select **screen** or **serial line** for report destination.

3. Select the type of data.

4. Press PgDn.

5. If you selected **serial line**, the reports are generated and the system returns to the Report/Transmission Menu.

   If you selected **screen**, the first page of the report is displayed and you are instructed to press any key to display additional pages. When all pages have been displayed, the system returns to the Report/Transmission Menu. You may press Esc at any time to cancel reports and return to the Report/Transmission Menu.

PRINTING REPORTS ON THE PRINTER

1. On the Start Report screen, enter the sample file number.

2. Select **printer** for report destination.

3. Select the type of data.

4. Press PgDn.

5. The reports are generated and the system returns to the Report Transmission Menu.

PRINTING REPORTS ON THE MODEL 7440A PLOTTER

NOTE

The plotter type on screen 10.8 must be set to manual when using a Model 7440A Plotter.

1. Insert a sheet of paper into the plotter. Make sure the top of the paper is lined up with the white line on the plotter.
2. Press the LOAD/UNLOAD button on the plotter.
3. On the Start Report screen, enter the sample file number.
4. Select **printer/plotter** for report destination.
5. Select the type of data.
6. Press [PgDn].
7. The system returns to the Report/Transmission Menu. If a summary page or tabular page was selected, these are printed on the printer. After about one to three minutes, the following prompt is displayed at the top of the screen:

   Unload plotter paper

   Press the LOAD/UNLOAD button on the plotter.
8. The following prompt is displayed:

   Load a sheet of plotter paper

   CAUTION

   Make sure the top edge of the paper is lined up with the white line on the plotter before proceeding. Failure to do so may distort the scale of the plot.

   Press the LOAD/UNLOAD button on the plotter.
9. When the first plot is complete, press the LOAD/UNLOAD button on the plotter and remove the paper.
10. If you selected more than one plot, repeat steps 8 and 9.

PRINTING REPORTS ON THE MODEL 7550A PLOTTER

NOTE

The plotter type on screen 10.8 must be set to automatic when using a Model 7550A Plotter.

1. On the Start Report screen, enter the sample file number.
2. Select **printer/plotter** for report destination.
3. Select the type of data.
4. Press PgDn.
5. Tabular reports are printed on the printer and plots are printed on the plotter; the system returns to the Report Transmission Menu.
6. When the last report is complete, press the LOAD/UNLOAD button on the plotter to remove the paper.

HOW TO CANCEL REPORTS

To access the Cancel Reports screen from the Main Function Menu, press [F6], then [F4].

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/13/89 TIME:10:03:01
LP:     14.70 psia IDLE                               HP:      14.7 psia IDLE
PORT      1        2                                  PORT       1
SAMPLE                                                SAMPLE
INTVOL    0.000    0.000    mL/g                      INTVOL     0.000    mL/g
% STEM                                                % STEM

                          6.4 CANCEL REPORTS


                Press [PgDn] to cancel all reports,

                     or [Esc] for no action
```

Press This Key	To
[PgDn]	Cancel all reports that have been sent to the printer, plotter, serial line, drive a: or drive b: and return to the Report Transmission Menu.
[Esc]	Continue transmitting reports and return to the Report Transmission Menu.

SAMPLE REPORTS

The remainder of this chapter contains samples of reports that may be generated by the Analysis Program. Note that points taken during high pressure runs have the mercury head correction factored in.

EXAMPLE 1

Example 1 contains a report produced by an automatic high pressure analysis using the default values.

SUMMARY REPORT

```
PORESIZER 9320  VX.XX                                                PAGE  1

SAMPLE DIRECTORY/NUMBER: DATA1     /9
OPERATOR: PBT                                          LP   16:15:19 09/18/89
SAMPLE ID: Reference Sandstone                         HP   17:27:03 09/18/89
SUBMITTER: MATERIALS ANALYSIS LAB                      REP  07:33:44 10/16/91

PENETROMETER NUMBER: 32                 ADVANCING CONTACT ANGLE: 130.0 deg
PENETROMETER CONSTANT:  10.79 µL/pF     RECEDING CONTACT ANGLE:  130.0 deg
PENETROMETER WEIGHT:   69.2711 g        MERCURY SURFACE TENSION: 485.0 dyn/cm
STEM VOLUME:           0.4120 mL        MERCURY DENSITY:          13.5335 g/mL
MAXIMUM HEAD PRESSURE: 4.6800 psi       SAMPLE WEIGHT:             1.8323 g
PENETROMETER VOLUME:   3.8409 mL        SAMPLE+PEN+Hg WEIGHT: 111.5026 g

LOW PRESSURE:
       MERCURY FILLING PRESSURE: 0.5167 psia
       LAST LOW PRESSURE POINT: 30.0181 psia

HIGH PRESSURE:
       RUN TYPE:                    AUTOMATIC
       RUN METHOD:                  EQUILIBRATED
       EQUILIBRATION TIME:              10 seconds

                           INTRUSION DATA SUMMARY

                   TOTAL INTRUSION VOLUME =     0.0884 mL/g
                          TOTAL PORE AREA =      2.312 sq-m/g
             MEDIAN PORE DIAMETER (VOLUME) =    8.5569 µm
               MEDIAN PORE DIAMETER (AREA) =    0.0096 µm
              AVERAGE PORE DIAMETER (4V/A) =    0.1530 µm
                              BULK DENSITY =    2.1411 g/mL
               APPARENT (SKELETAL) DENSITY =    2.6412 g/mL
                                  POROSITY =     18.93 %
                          STEM VOLUME USED =        39 %
```

TABULAR DATA

PORESIZER 9320 VX.XX PAGE 2

SAMPLE DIRECTORY/NUMBER: DATA1 /9
OPERATOR: PBT LP 16:15:19 09/18/89
SAMPLE ID: Reference Sandstone HP 17:27:03 09/18/89
SUBMITTER: MATERIALS ANALYSIS LAB REP 07:33:44 10/15/91

PRESSURE psia	PORE DIAMETER µm	MEAN DIAMETER µm	CUMULATIVE VOLUME mL/g	INCREMENTAL VOLUME mL/g	DIFFEREN. VOL dV/dD mL/g-µm
0.52	350.0577	350.0577	0.0000	0.0000	1.012E-05
1.63	110.7665	230.4121	0.0042	0.0042	3.036E-05
2.00	90.2586	100.5125	0.0048	0.0006	3.046E-05
2.99	60.4456	75.3521	0.0058	0.0010	4.375E-05
4.00	45.2252	52.8354	0.0067	0.0008	6.009E-05
5.52	32.7849	39.0050	0.0075	0.0009	9.284E-05
7.05	25.6537	29.2193	0.0084	0.0009	1.632E-04
8.65	20.9002	23.2770	0.0094	0.0010	2.835E-04
10.51	17.2038	19.0520	0.0109	0.0014	5.661E-04
13.04	13.8662	15.5350	0.0142	0.0033	2.361E-03
15.98	11.3184	12.5923	0.0251	0.0109	5.758E-03
19.97	9.0560	10.1872	0.0411	0.0161	6.384E-03
22.99	7.8671	8.4615	0.0477	0.0066	4.891E-03
25.13	7.1981	7.5326	0.0507	0.0029	4.181E-03
30.02	6.0252	6.6116	0.0550	0.0043	3.149E-03
39.95	4.5270	5.2761	0.0590	0.0040	2.512E-03
49.90	3.6243	4.0756	0.0613	0.0024	2.735E-03
59.53	3.0380	3.3311	0.0631	0.0017	2.908E-03
74.66	2.4224	2.7302	0.0649	0.0019	3.228E-03
89.63	2.0178	2.2201	0.0664	0.0014	3.666E-03
114.60	1.5783	1.7981	0.0682	0.0018	4.372E-03
139.23	1.2990	1.4386	0.0695	0.0013	5.046E-03
174.54	1.0362	1.1676	0.0710	0.0015	5.929E-03
219.17	0.8252	0.9307	0.0723	0.0014	7.015E-03
268.98	0.6724	0.7488	0.0735	0.0012	8.030E-03
328.30	0.5509	0.6117	0.0746	0.0011	9.062E-03
418.27	0.4324	0.4917	0.0757	0.0012	1.006E-02
517.59	0.3494	0.3909	0.0766	0.0009	1.128E-02
637.24	0.2838	0.3166	0.0774	0.0008	1.293E-02
796.05	0.2272	0.2555	0.0782	0.0008	1.437E-02
985.87	0.1835	0.2053	0.0789	0.0007	1.623E-02
1244.03	0.1454	0.1644	0.0796	0.0007	1.786E-02
1393.02	0.1298	0.1376	0.0799	0.0003	1.803E-02
1596.18	0.1133	0.1216	0.0802	0.0003	2.035E-02
1894.84	0.0955	0.1044	0.0806	0.0004	2.507E-02
2297.50	0.0787	0.0871	0.0811	0.0005	2.929E-02
2595.83	0.0697	0.0742	0.0813	0.0003	3.229E-02
2996.65	0.0604	0.0650	0.0817	0.0003	3.723E-02
3491.48	0.0518	0.0561	0.0820	0.0004	4.474E-02
3991.31	0.0453	0.0486	0.0823	0.0003	5.007E-02
4588.97	0.0394	0.0424	0.0827	0.0003	5.453E-02
5382.96	0.0336	0.0365	0.0830	0.0003	6.783E-02
6183.29	0.0293	0.0314	0.0834	0.0004	7.983E-02
7078.95	0.0255	0.0274	0.0837	0.0003	8.344E-02
8195.28	0.0221	0.0238	0.0840	0.0003	9.811E-02

TABULAR DATA (continued)

```
PORESIZER 9320 VX.XX                                        PAGE  3

SAMPLE DIRECTORY/NUMBER: DATA1    /9
OPERATOR: PBT                                     LP  16:15:19 09/18/89
SAMPLE ID: Reference Sandstone                    HP  17:27:03 09/18/89
SUBMITTER: MATERIALS ANALYSIS LAB                 REP 07:33:44 10/15/91
```

PRESSURE psia	PORE DIAMETER μm	MEAN DIAMETER μm	CUMULATIVE VOLUME mL/g	INCREMENTAL VOLUME mL/g	DIFFEREN. VOL dV/dD mL/g-μm
9491.44	0.0191	0.0206	0.0843	0.0003	1.247E-01
10784.61	0.0168	0.0179	0.0846	0.0003	1.452E-01
12479.77	0.0145	0.0156	0.0850	0.0004	1.798E-01
14379.10	0.0126	0.0135	0.0854	0.0004	2.309E-01
16579.26	0.0109	0.0117	0.0858	0.0004	2.975E-01
18971.75	0.0095	0.0102	0.0863	0.0005	3.799E-01
21966.75	0.0082	0.0089	0.0869	0.0006	4.988E-01
24963.74	0.0072	0.0077	0.0875	0.0006	6.607E-01
29852.07	0.0061	0.0067	0.0884	0.0010	9.036E-01
24983.39	0.0072	0.0066	0.0884	0.0000	0.000E+00
21997.04	0.0082	0.0077	0.0884	0.0000	0.000E+00
19003.70	0.0095	0.0089	0.0884	0.0000	0.000E+00
16610.20	0.0109	0.0102	0.0884	0.0000	0.000E+00
14408.69	0.0126	0.0117	0.0884	0.0000	0.000E+00
12505.69	0.0145	0.0135	0.0884	0.0000	0.000E+00
10811.19	0.0167	0.0156	0.0884	0.0000	0.000E+00
9507.19	0.0190	0.0179	0.0884	0.0000	1.250E-02
8205.19	0.0220	0.0205	0.0883	-0.0001	3.123E-02
7104.86	0.0255	0.0237	0.0882	-0.0001	4.089E-02
6208.53	0.0291	0.0273	0.0881	-0.0002	4.899E-02
5410.20	0.0334	0.0313	0.0879	-0.0002	3.943E-02
4605.53	0.0393	0.0364	0.0877	-0.0002	3.158E-02
4006.53	0.0451	0.0422	0.0875	-0.0002	2.735E-02
3504.04	0.0516	0.0484	0.0873	-0.0002	2.307E-02
3006.21	0.0602	0.0559	0.0872	-0.0002	2.210E-02
2604.21	0.0695	0.0648	0.0870	-0.0002	2.229E-02
2302.71	0.0785	0.0740	0.0868	-0.0002	1.964E-02
1908.22	0.0948	0.0867	0.0865	-0.0003	1.777E-02
1607.06	0.1125	0.1037	0.0862	-0.0003	1.466E-02
1404.06	0.1288	0.1207	0.0860	-0.0002	1.415E-02
1252.56	0.1444	0.1366	0.0857	-0.0003	1.255E-02
991.74	0.1824	0.1634	0.0854	-0.0003	7.943E-03
801.74	0.2256	0.2040	0.0851	-0.0003	6.609E-03
642.08	0.2817	0.2536	0.0848	-0.0003	5.409E-03
518.09	0.3491	0.3154	0.0844	-0.0003	4.731E-03
421.60	0.4290	0.3890	0.0841	-0.0004	5.505E-03
330.78	0.5468	0.4879	0.0833	-0.0008	4.631E-03
270.62	0.6683	0.6076	0.0829	-0.0004	3.370E-03
220.63	0.8198	0.7440	0.0824	-0.0005	3.044E-03
175.48	1.0307	0.9252	0.0819	-0.0006	2.440E-03
138.16	1.3091	1.1699	0.0813	-0.0006	2.108E-03
114.50	1.5796	1.4444	0.0807	-0.0006	2.450E-03
90.36	2.0017	1.7906	0.0796	-0.0010	1.986E-03
74.87	2.4157	2.2087	0.0790	-0.0007	1.561E-03
60.22	3.0033	2.7095	0.0781	-0.0009	1.448E-03

TABULAR DATA (continued)

PORESIZER 9320 VX.XX PAGE 4

SAMPLE DIRECTORY/NUMBER: DATA1 /9
OPERATOR: PBT
SAMPLE ID: Reference Sandstone
SUBMITTER: MATERIALS ANALYSIS LAB

LP 16:15:19 09/18/89
HP 17:27:03 09/18/89
REP 07:33:44 10/15/91

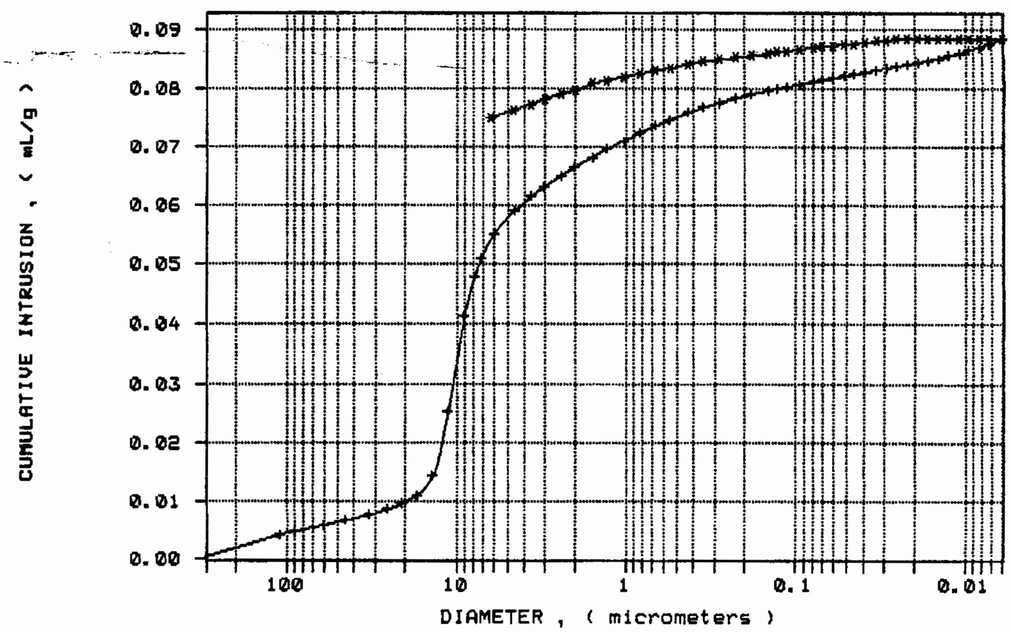
PRESSURE psia	PORE DIAMETER µm	MEAN DIAMETER µm	CUMULATIVE VOLUME mL/g	INCREMENTAL VOLUME mL/g	DIFFEREN. VOL dV/dD mL/g-µm
49.41	3.6607	3.3320	0.0772	-0.0009	1.224E-03
39.43	4.5873	4.1240	0.0762	-0.0010	9.293E-04
28.79	6.2828	5.4351	0.0749	-0.0013	6.681E-04

CUMULATIVE INTRUSION vs DIAMETER

```
PORESIZER 9320  VX.XX                                              PAGE  5

SAMPLE DIRECTORY/NUMBER: DATA1    /9
OPERATOR: PBT                                       LP  16:15:19 09/18/89
SAMPLE ID: Reference Sandstone                      HP  17:27:03 09/18/89
SUBMITTER: MATERIALS ANALYSIS LAB                   REP 07:33:44 10/15/91
```

CUMULATIVE INTRUSION vs DIAMETER
+ intrusion, * extrusion



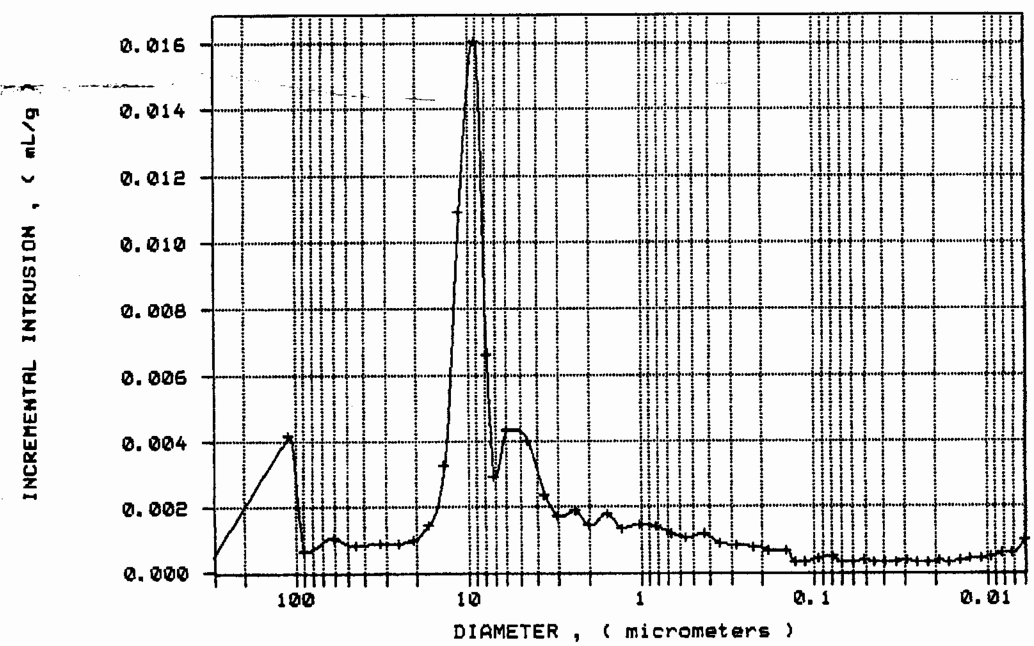
INCREMENTAL INTRUSION vs DIAMETER

PORESIZER 9320 VX.XX PAGE 6

SAMPLE DIRECTORY/NUMBER: DATA1 /9
OPERATOR: PBT
SAMPLE ID: Reference Sandstone
SUBMITTER: MATERIALS ANALYSIS LAB

LP 16:15:19 09/18/89
HP 17:27:03 09/18/89
REP 07:33:44 10/15/91

INCREMENTAL INTRUSION vs DIAMETER

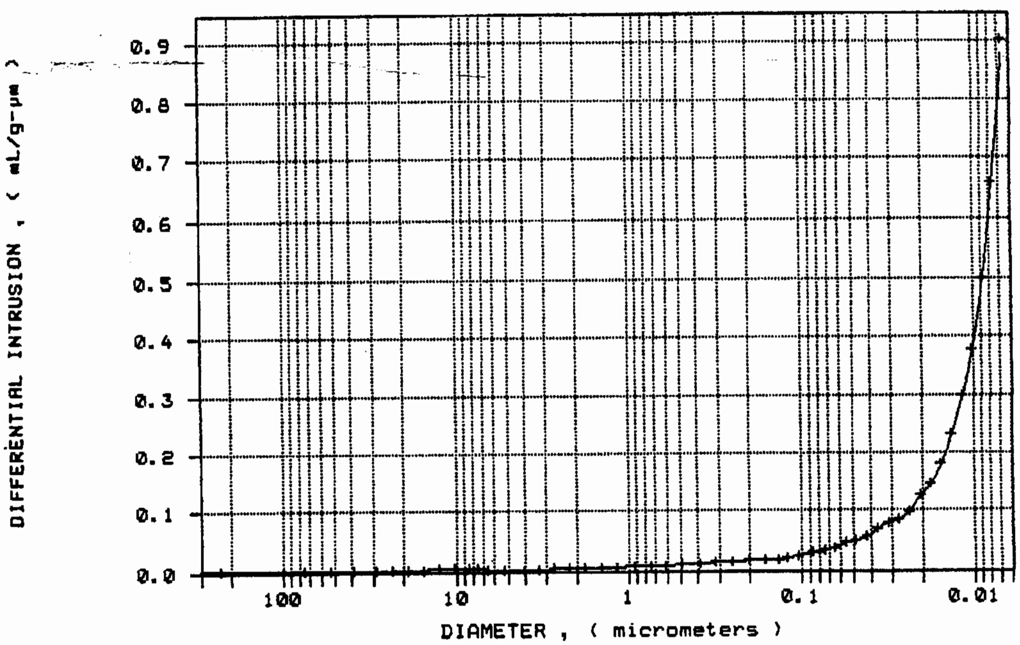


DIFFERENTIAL INTRUSION vs DIAMETER

```
PORESIZER 9320 VX.XX                                              PAGE  7

SAMPLE DIRECTORY/NUMBER: DATA1    /9
OPERATOR: PBT                                        LP   16:15:19 09/18/89
SAMPLE ID: Reference Sandstone                       HP   17:27:03 09/18/89
SUBMITTER: MATERIALS ANALYSIS LAB                    REP  07:33:44 10/15/91
```

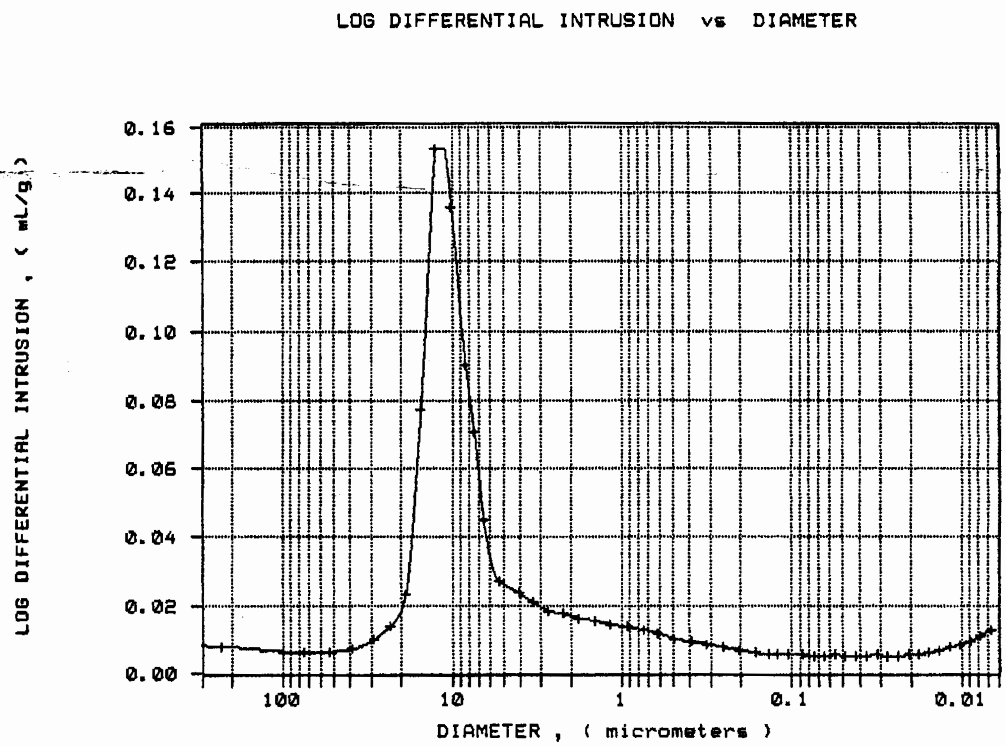
DIFFERENTIAL INTRUSION vs DIAMETER



LOG DIFFERENTIAL INTRUSION vs DIAMETER

```
PORESIZER 9320 VX.XX                                          PAGE  8

SAMPLE DIRECTORY/NUMBER: DATA1    /9
OPERATOR: PBT                                   LP   16:15:19 09/18/89
SAMPLE ID: Reference Sandstone                  HP   17:27:03 09/18/89
SUBMITTER: MATERIALS ANALYSIS LAB               REP  07:33:44 10/15/91
```



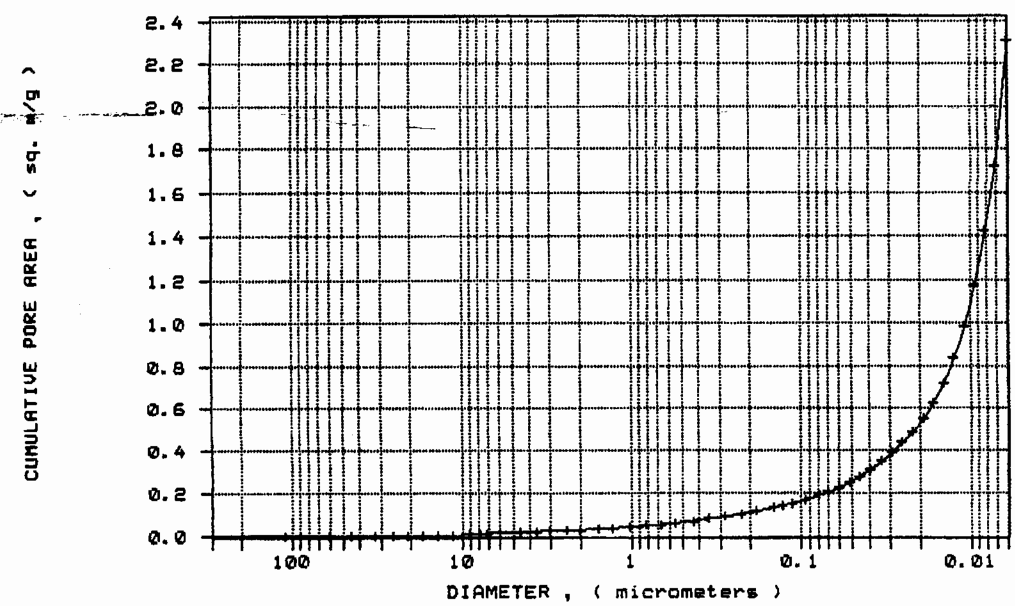
CUMULATIVE PORE AREA vs DIAMETER

PORESIZER 9320 VX.XX PAGE 9

SAMPLE DIRECTORY/NUMBER: DATA1 /9
OPERATOR: PBT
SAMPLE ID: Reference Sandstone
SUBMITTER: MATERIALS ANALYSIS LAB

LP 16:15:19 09/18/89
HP 17:27:03 09/18/89
REP 07:33:44 10/15/91

CUMULATIVE PORE AREA vs DIAMETER



EXAMPLE 2

Example 2 contains a reduced report, from the same sample, that contains only the tabular data pages and one graph. The tabular data pages contain quantities not reported on the previous tabular pages. The graph is of incremental pore area vs. diameter.

TABULAR DATA

```
PORESIZER 9320  VX.XX                                         PAGE  1

SAMPLE DIRECTORY/NUMBER: DATA1    /9
OPERATOR: PBT                                   LP  16:15:19 09/18/89
SAMPLE ID: Reference Sandstone                  HP  17:27:03 09/18/89
SUBMITTER: MATERIALS ANALYSIS LAB               REP 08:26:43 10/15/91
```

PRESSURE psia	LOG DIFF. VOL dV/dlogD mL/g	CUMULATIVE PORE AREA sq-m/g	INCREMENTAL PORE AREA sq-m/g	% OF TOTAL INTRUSION VOLUME
0.52	8.340E-03	0.000	0.000	0.0000
1.63	7.915E-03	0.000	0.000	4.7126
2.00	6.469E-03	0.000	0.000	5.4376
2.99	6.225E-03	0.000	0.000	6.6139
4.00	6.397E-03	0.000	0.000	7.5239
5.52	7.165E-03	0.000	0.000	8.5374
7.05	9.856E-03	0.000	0.000	9.5509
8.65	1.395E-02	0.001	0.000	10.6680
10.51	2.292E-02	0.001	0.000	12.2956
13.04	7.710E-02	0.002	0.001	16.0169
15.98	1.534E-01	0.005	0.003	28.3273
19.97	1.361E-01	0.012	0.006	46.5192
22.99	9.059E-02	0.015	0.003	53.9913
25.13	7.085E-02	0.016	0.002	57.3056
30.02	4.467E-02	0.019	0.003	62.1958
39.95	2.676E-02	0.022	0.003	66.7086
49.90	2.334E-02	0.024	0.002	69.3719
59.53	2.080E-02	0.026	0.002	71.3102
74.66	1.841E-02	0.029	0.003	73.4335
89.63	1.741E-02	0.032	0.003	75.0462
114.60	1.624E-02	0.036	0.004	77.0659
139.23	1.543E-02	0.039	0.004	78.5825
174.54	1.446E-02	0.044	0.005	80.2397
219.17	1.363E-02	0.050	0.006	81.8081
268.98	1.271E-02	0.057	0.006	83.1398
328.30	1.175E-02	0.063	0.007	84.3308
418.27	1.024E-02	0.073	0.010	85.6551
517.59	9.276E-03	0.082	0.009	86.6539
637.24	8.640E-03	0.092	0.010	87.5786
796.05	7.685E-03	0.105	0.012	88.4664
985.87	7.010E-03	0.118	0.013	89.2358
1244.03	6.114E-03	0.134	0.016	89.9978
1393.02	5.510E-03	0.143	0.008	90.3233
1596.18	5.430E-03	0.152	0.010	90.6562
1894.84	5.633E-03	0.169	0.017	91.1445
2297.50	5.428E-03	0.190	0.021	91.6624
2595.83	5.296E-03	0.205	0.016	91.9879
2996.65	5.290E-03	0.225	0.020	92.3504
3491.48	5.451E-03	0.251	0.026	92.7573
3991.31	5.341E-03	0.277	0.026	93.1198
4588.97	5.059E-03	0.307	0.030	93.4749
5382.96	5.366E-03	0.344	0.037	93.8596
6183.29	5.496E-03	0.389	0.045	94.2591
7078.95	5.019E-03	0.434	0.045	94.6068
8195.28	5.097E-03	0.485	0.051	94.9471

TABULAR DATA (continued)

PORESIZER 9320 VX.XX PAGE 2

SAMPLE DIRECTORY/NUMBER: DATA1 /9
OPERATOR: PBT LP 16:15:19 09/18/89
SAMPLE ID: Reference Sandstone HP 17:27:03 09/18/89
SUBMITTER: MATERIALS ANALYSIS LAB REP 08:26:43 10/15/91

PRESSURE psia	LOG DIFF. VOL dV/dlogD mL/g	CUMULATIVE PORE AREA sq-m/g	INCREMENTAL PORE AREA sq-m/g	% OF TOTAL INTRUSION VOLUME
9491.44	5.595E-03	0.552	0.067	95.3392
10784.61	5.733E-03	0.624	0.072	95.7017
12479.77	6.133E-03	0.716	0.092	96.1086
14379.10	6.838E-03	0.836	0.120	96.5673
16579.26	7.638E-03	0.985	0.149	97.0630
18971.75	8.526E-03	1.172	0.187	97.6030
21966.75	9.667E-03	1.428	0.256	98.2467
24963.74	1.127E-02	1.722	0.294	98.8903
29852.07	1.289E-02	2.312	0.590	100.0000
24983.39	0.000E+00	2.312	0.000	100.0000
21997.04	0.000E+00	2.312	0.000	100.0000
19003.70	0.000E+00	2.312	0.000	100.0000
16610.20	0.000E+00	2.312	0.000	100.0000
14408.69	0.000E+00	2.312	0.000	100.0000
12505.69	0.000E+00	2.312	0.000	100.0000
10811.19	0.000E+00	2.312	0.000	100.0000
9507.19	5.403E-04	2.312	0.000	100.0000
8205.19	1.563E-03	2.296	-0.017	99.9038
7104.86	2.365E-03	2.278	-0.018	99.7855
6208.53	3.241E-03	2.252	-0.026	99.5857
5410.20	2.993E-03	2.227	-0.025	99.3638
4605.53	2.816E-03	2.206	-0.021	99.1492
4006.53	2.804E-03	2.190	-0.017	98.9495
3504.04	2.704E-03	2.177	-0.013	98.7719
3006.21	3.020E-03	2.163	-0.013	98.5648
2604.21	3.515E-03	2.151	-0.013	98.3280
2302.71	3.503E-03	2.140	-0.010	98.1135
1908.22	3.824E-03	2.127	-0.013	97.7954
1607.06	3.746E-03	2.115	-0.012	97.4329
1404.06	4.140E-03	2.109	-0.006	97.2257
1252.56	4.118E-03	2.101	-0.007	96.9372
991.74	3.290E-03	2.094	-0.008	96.5895
801.74	3.385E-03	2.088	-0.006	96.2270
642.08	3.460E-03	2.082	-0.005	95.8645
518.09	3.750E-03	2.078	-0.004	95.4798
421.60	5.359E-03	2.075	-0.004	95.0803
330.78	5.752E-03	2.068	-0.006	94.2073
270.62	5.114E-03	2.066	-0.002	93.7782
220.63	5.667E-03	2.063	-0.003	93.2085
175.48	5.710E-03	2.061	-0.002	92.5723
138.16	6.268E-03	2.059	-0.002	91.8843
114.50	8.791E-03	2.057	-0.002	91.2185
90.36	9.028E-03	2.055	-0.002	90.0496
74.87	8.562E-03	2.053	-0.001	89.2950
60.22	9.878E-03	2.052	-0.001	88.3110

TABULAR DATA (continued)

```
PORESIZER 9320  VX.XX                                               PAGE  3

SAMPLE DIRECTORY/NUMBER: DATA1    /9
OPERATOR: PBT                                          LP  16:16:19 09/18/89
SAMPLE ID: Reference Sandstone                         HP  17:27:03 09/18/89
SUBMITTER: MATERIALS ANALYSIS LAB                      REP 08:26:43 10/15/91
```

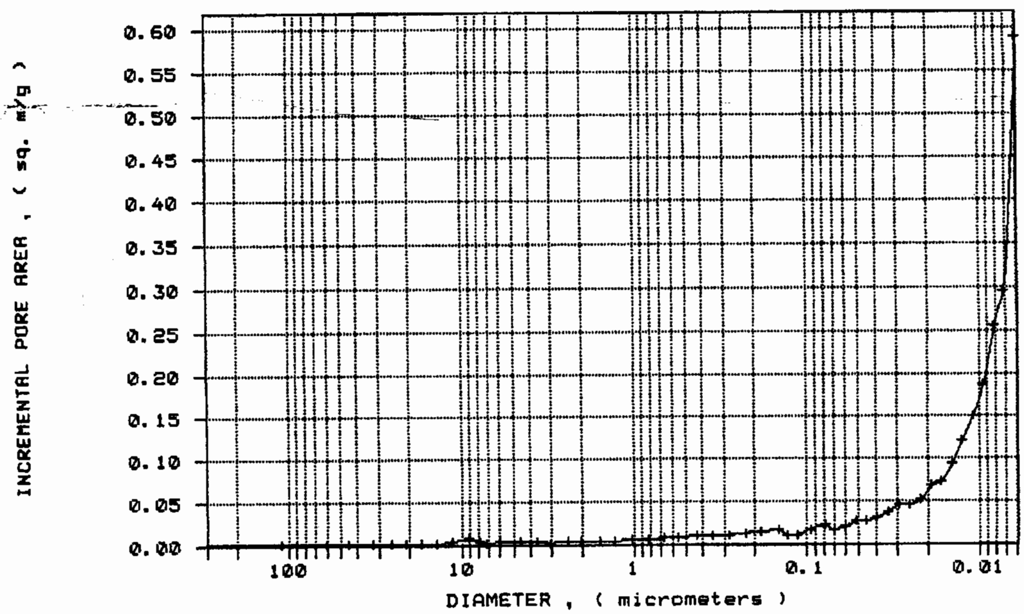
PRESSURE psia	LOG DIFF. VOL dV/dlogD mL/g	CUMULATIVE PORE AREA sq-m/g	INCREMENTAL PORE AREA sq-m/g	% OF TOTAL INTRUSION VOLUME
49.41	1.017E-02	2.051	-0.001	87.3197
39.43	9.680E-03	2.050	-0.001	86.2100
28.79	9.532E-03	2.049	-0.001	84.7377

INCREMENTAL PORE AREA vs DIAMETER

```
PORESIZER 9320  VX.XX                                          PAGE  4

SAMPLE DIRECTORY/NUMBER: DATA1    /9
OPERATOR: PBT                                    LP   16:15:19 09/18/89
SAMPLE ID: Reference Sandstone                   HP   17:27:03 09/18/89
SUBMITTER: MATERIALS ANALYSIS LAB                REP  08:26:43 10/15/91
```

INCREMENTAL PORE AREA vs DIAMETER



EXAMPLE 3

Example 3 contains a reduced report from the same sample that contains only the summary page and a tabular data page.

The tabular data page illustrates the use of a tabular data set to report tabular data at pre-selected points interpolated from the collected data. This is useful for direct comparison of quality control applications. This tabular data set specified pore diameters of 100, 50, 20, 10, etc.

All interpolations in the PoreSizer 9320 data reduction are accomplished with an Akima semi-spline function which produces a cubic spline that is forced to pass through all the collected data points.

SUMMARY DATA

```
PORESIZER 9320  VX.XX                                                PAGE  1

SAMPLE DIRECTORY/NUMBER: DATA1    /9
OPERATOR: PBT                                            LP   16:15:19 09/18/89
SAMPLE ID: Reference Sandstone                           HP   17:27:03 09/18/89
SUBMITTER: MATERIALS ANALYSIS LAB                        REP  08:31:31 10/15/91


PENETROMETER NUMBER: 32              ADVANCING CONTACT ANGLE: 130.0 deg
PENETROMETER CONSTANT:  10.79 µL/pF  RECEDING CONTACT ANGLE:  130.0 deg
PENETROMETER WEIGHT:    69.2711 g    MERCURY SURFACE TENSION: 485.0 dyn/cm
STEM VOLUME:            0.4120 mL    MERCURY DENSITY:        13.5335 g/mL
MAXIMUM HEAD PRESSURE: 4.6800 psi    SAMPLE WEIGHT:           1.8323 g
PENETROMETER VOLUME:    3.8409 mL    SAMPLE+PEN+Hg WEIGHT: 111.5026 g


LOW PRESSURE:
       MERCURY FILLING PRESSURE:  0.5167 psia
       LAST LOW PRESSURE POINT: 30.0181 psia

HIGH PRESSURE:
       RUN TYPE:                   AUTOMATIC
       RUN METHOD:                 EQUILIBRATED
       EQUILIBRATION TIME:             10 seconds

                          INTRUSION DATA SUMMARY

                 TOTAL INTRUSION VOLUME =    0.0884 mL/g
                        TOTAL PORE AREA =     2.312 sq-m/g
          MEDIAN PORE DIAMETER (VOLUME) =    8.5569 µm
            MEDIAN PORE DIAMETER (AREA) =    0.0096 µm
           AVERAGE PORE DIAMETER (4V/A) =    0.1530 µm
                           BULK DENSITY =    2.1411 g/mL
            APPARENT (SKELETAL) DENSITY =    2.6412 g/mL
                               POROSITY =     18.93 %
                       STEM VOLUME USED =        39 %
```

TABULAR DATA

PORESIZER 9320 VX.XX PAGE 2

SAMPLE DIRECTORY/NUMBER: DATA1 /9
OPERATOR: PBT
SAMPLE ID: Reference Sandstone
SUBMITTER: MATERIALS ANALYSIS LAB

LP 16:15:19 09/18/89
HP 17:27:03 09/18/89
REP 08:31:31 10/15/91

PRESSURE psia	PORE DIAMETER µm	MEAN DIAMETER µm	CUMULATIVE VOLUME mL/g	INCREMENTAL VOLUME mL/g	DIFFEREN. VOL dV/dD mL/g-µm
1.81	100.0000	100.0000	0.0045	0.0045	2.636E-05
3.62	50.0000	75.0000	0.0064	0.0019	2.754E-05
9.04	20.0000	35.0000	0.0097	0.0033	1.474E-03
18.09	10.0000	15.0000	0.0337	0.0241	3.407E-03
36.17	5.0000	7.5000	0.0578	0.0240	6.213E-03
90.43	2.0000	3.5000	0.0664	0.0086	2.873E-03
180.86	1.0000	1.5000	0.0712	0.0047	6.075E-03
361.73	0.5000	0.7500	0.0751	0.0039	9.472E-03
904.32	0.2000	0.3500	0.0786	0.0036	1.422E-02
1808.64	0.1000	0.1500	0.0805	0.0018	2.363E-02
3617.27	0.0500	0.0750	0.0821	0.0016	4.556E-02
9043.18	0.0200	0.0350	0.0842	0.0021	1.172E-01
18086.35	0.0100	0.0150	0.0861	0.0019	3.004E-01
9043.18	0.0200	0.0150	0.0884	0.0023	2.168E-02
3617.27	0.0500	0.0350	0.0874	-0.0010	2.853E-02
1808.64	0.1000	0.0750	0.0864	-0.0010	1.542E-02
904.32	0.2000	0.1500	0.0853	-0.0011	8.452E-03
361.73	0.5000	0.3500	0.0836	-0.0017	4.023E-03
180.86	1.0000	0.7500	0.0819	-0.0016	2.783E-03
90.43	2.0000	1.5000	0.0796	-0.0023	2.051E-03
36.17	5.0000	3.5000	0.0759	-0.0038	8.325E-04

EXAMPLE 4

Example 4 contains a reduced report, from the same sample, that contains the summary page, tabular data page, and one graph. The graph is of cumulative intrusion vs. pressure. The calculation range has been specified as 0.0 to 25000.0 psia, and the system parameter "size type" has been specified as radius.

SUMMARY PAGE

```
PORESIZER 9320 VX.XX                                              PAGE  1

SAMPLE DIRECTORY/NUMBER: DATA1    /9
OPERATOR: PBT                                      LP   16:15:19 09/18/89
SAMPLE ID: Reference Sandstone                     HP   17:27:03 09/18/89
SUBMITTER: MATERIALS ANALYSIS LAB                  REP  08:33:58 10/15/91

PENETROMETER NUMBER: 32                ADVANCING CONTACT ANGLE: 130.0 deg
PENETROMETER CONSTANT:  10.79 µL/pF    RECEDING CONTACT ANGLE:  130.0 deg
PENETROMETER WEIGHT:   69.2711 g       MERCURY SURFACE TENSION: 485.0 dyn/cm
STEM VOLUME:           0.4120 mL       MERCURY DENSITY:        13.5335 g/mL
MAXIMUM HEAD PRESSURE: 4.6800 psi      SAMPLE WEIGHT:           1.8323 g
PENETROMETER VOLUME:   3.8409 mL       SAMPLE+PEN+Hg WEIGHT: 111.5026 g

LOW PRESSURE:
       MERCURY FILLING PRESSURE:  0.5167 psia
       LAST LOW PRESSURE POINT: 30.0181 psia

HIGH PRESSURE:
       RUN TYPE:                      AUTOMATIC
       RUN METHOD:                    EQUILIBRATED
       EQUILIBRATION TIME:                10 seconds

                        INTRUSION DATA SUMMARY
               (FROM PRESSURE      0.00 TO 25000.00 psia )

                  TOTAL INTRUSION VOLUME =     0.0875 mL/g
                         TOTAL PORE AREA =      1.726 sq-m/g
             MEDIAN PORE  RADIUS  (VOLUME) =     4.3242 µm
               MEDIAN PORE  RADIUS  (AREA) =     0.0061 µm
              AVERAGE PORE  RADIUS  (4V/A) =     0.1013 µm
                            BULK DENSITY =     2.1411 g/mL
             APPARENT  (SKELETAL) DENSITY =     2.6344 g/mL
                                POROSITY =      18.73 %
                        STEM VOLUME USED =         39 %
```

TABULAR DATA

PORESIZER 9320 VX.XX PAGE 2

SAMPLE DIRECTORY/NUMBER: DATA1 /9
OPERATOR: PBT
SAMPLE ID: Reference Sandstone
SUBMITTER: MATERIALS ANALYSIS LAB

LP 16:15:19 09/18/89
HP 17:27:03 09/18/89
REP 08:33:58 10/15/91

PRESSURE psia	LOG DIFF. VOL dV/dlogR mL/g	CUMULATIVE PORE AREA sq-m/g	INCREMENTAL PORE AREA sq-m/g	% OF TOTAL INTRUSION VOLUME
0.52	8.340E-03	0.000	0.000	0.0000
1.63	7.922E-03	0.000	0.000	4.7651
2.00	6.462E-03	0.000	0.000	5.4982
2.99	6.226E-03	0.000	0.000	6.6876
4.00	6.397E-03	0.000	0.000	7.6077
5.52	7.157E-03	0.000	0.000	8.6325
7.05	9.853E-03	0.000	0.000	9.6573
8.65	1.394E-02	0.001	0.000	10.7869
10.51	2.285E-02	0.001	0.000	12.4326
13.04	7.699E-02	0.002	0.001	16.1953
15.98	1.538E-01	0.005	0.003	28.6428
19.97	1.361E-01	0.012	0.006	47.0373
22.99	9.037E-02	0.015	0.003	54.5926
25.13	7.075E-02	0.016	0.002	57.9439
30.02	4.465E-02	0.019	0.003	62.8885
39.95	2.674E-02	0.022	0.003	67.4516
49.90	2.334E-02	0.024	0.002	70.1445
59.53	2.080E-02	0.026	0.002	72.1044
74.66	1.840E-02	0.029	0.003	74.2513
89.63	1.741E-02	0.032	0.003	75.8821
114.60	1.624E-02	0.036	0.004	77.9242
139.23	1.543E-02	0.039	0.004	79.4577
174.54	1.446E-02	0.044	0.005	81.1334
219.17	1.363E-02	0.050	0.006	82.7192
268.98	1.271E-02	0.057	0.006	84.0667
328.30	1.175E-02	0.063	0.007	85.2701
418.27	1.024E-02	0.073	0.010	86.6091
517.59	9.276E-03	0.082	0.009	87.6190
637.24	8.641E-03	0.092	0.010	88.5540
796.05	7.685E-03	0.105	0.012	89.4517
985.87	7.010E-03	0.118	0.013	90.2296
1244.03	6.117E-03	0.134	0.016	91.0001
1393.02	5.502E-03	0.143	0.008	91.3293
1596.18	5.428E-03	0.152	0.010	91.6659
1894.84	5.634E-03	0.169	0.017	92.1596
2297.50	5.430E-03	0.190	0.021	92.6832
2595.83	5.295E-03	0.205	0.016	93.0124
2996.65	5.288E-03	0.225	0.020	93.3789
3491.48	5.458E-03	0.251	0.026	93.7904
3991.31	5.344E-03	0.277	0.026	94.1569
4588.97	5.057E-03	0.307	0.030	94.5160
5382.96	5.364E-03	0.344	0.037	94.9050
6183.29	5.505E-03	0.389	0.045	95.3089
7078.95	5.017E-03	0.434	0.045	95.6605
8195.28	5.095E-03	0.485	0.051	96.0046

TABULAR DATA (continued)

PORESIZER 9320 VX.XX PAGE 3

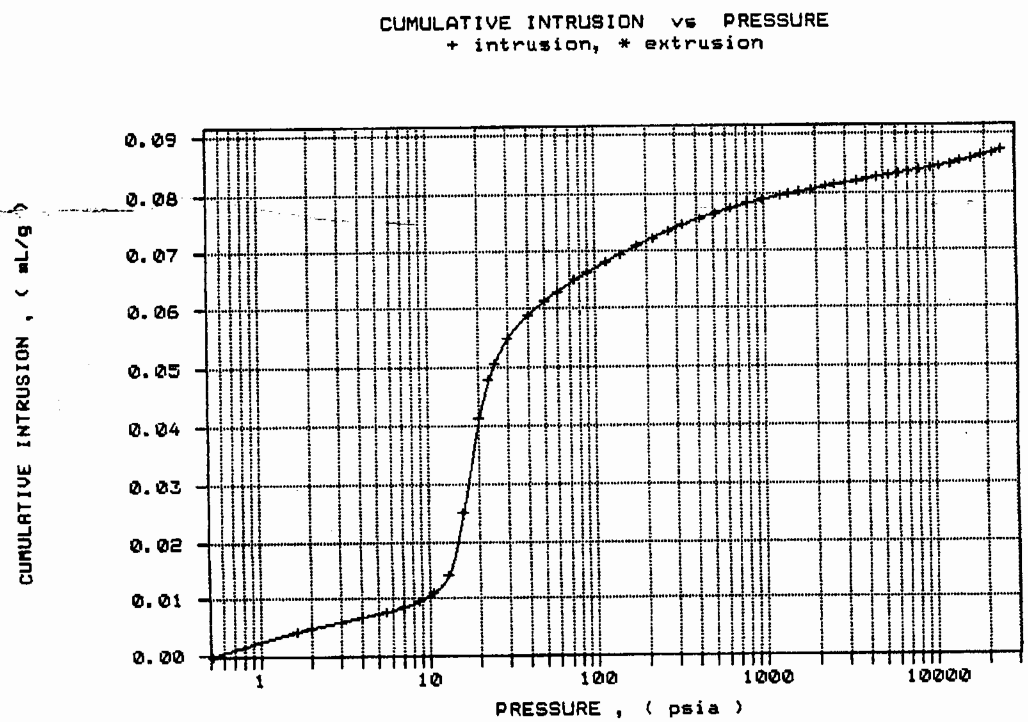
SAMPLE DIRECTORY/NUMBER: DATA1 /9
OPERATOR: PBT LP 16:15:19 09/18/89
SAMPLE ID: Reference Sandstone HP 17:27:03 09/18/89
SUBMITTER: MATERIALS ANALYSIS LAB REP 08:33:58 10/15/91

PRESSURE psia	LOG DIFF. VOL dV/dlogR mL/g	CUMULATIVE PORE AREA sq-m/g	INCREMENTAL PORE AREA sq-m/g	% OF TOTAL INTRUSION VOLUME
9491.44	5.601E-03	0.552	0.067	96.4010
10784.61	5.732E-03	0.624	0.072	96.7676
12479.77	6.130E-03	0.716	0.092	97.1790
14379.10	6.840E-03	0.836	0.120	97.6428
16579.26	7.635E-03	0.985	0.149	98.1440
18971.75	8.527E-03	1.172	0.187	98.6901
21966.75	9.617E-03	1.428	0.256	99.3409
24963.74	1.018E-02	1.722	0.294	99.9917
25000.00	1.018E-02	1.726	0.004	100.0000

CUMULATIVE INTRUSION vs PRESSURE

```
PORESIZER 9320  VX.XX                                           PAGE  4

SAMPLE DIRECTORY/NUMBER: DATA1    /9
OPERATOR: PBT                                  LP   16:15:19 09/18/89
SAMPLE ID: Reference Sandstone                 HP   17:27:03 09/18/89
SUBMITTER: MATERIALS ANALYSIS LAB              REP  08:33:58 10/15/91
```



CHAPTER 6

ENTERING AND MAINTAINING ANALYSIS INFORMATION

- Main Function Menu
- Entering and Maintaining Sample Information
- Entering and Maintaining Pressure Table Information
- Entering and Maintaining Rate Sequence Tables
- Entering and Maintaining Report Information
- Entering and Maintaining Tabular Data Sets
- Terminal Emulation Mode
- Performing Low Pressure Runs
- Performing High Pressure Runs
- Controlling and Monitoring Low and High Pressure Runs

ENTERING AND MAINTAINING ANALYSIS INFORMATION

MAIN FUNCTION MENU

The functions in the Analysis Program are accessed from the Main Function Menu, which is shown below. This chapter describes all the functions listed on this menu except Utilities Menu. Chapter 7 describes the functions listed on the Utilities Menu.

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/13/89 TIME:10:06:08
LP:    14.70 psia IDLE                                HP:     14.7 psia IDLE
PORT        1         2                               PORT      1
SAMPLE                                                SAMPLE
INTVOL    0.000     0.000    mL/g                     INTVOL    0.000    mL/g
% STEM                                                % STEM

                           MAIN FUNCTION MENU

 F1  - Help

 F3  - Sample Information Menu          F4  - Pressure Table Menu

 F5  - Rate Sequence Table Menu         F6  - Report/Transmission Menu

 F7  - Start Low Pressure Run           F8  - Start High Pressure Run

 F9  - Run Control/Status Menu          F10 - Utilities Menu

Press Desired Key
```

FUNCTION	DESCRIPTION
F1 - Help	Displays the HELP screens.
F3 - Sample Information Menu	Displays the menu that enables you to add, change, delete and print sample information and to display or print a directory of previously-entered samples.
F4 - Pressure Table Menu	Displays the menu that enables you to add, change, delete and print pressure tables and to display or print a directory of previously-entered pressure tables.
F5 - Rate Sequence Table Menu	Displays the menu that enables you to add, change, delete and print rate sequence tables and to display or print a directory of previously-entered rate sequence tables.

FUNCTION	DESCRIPTION
F6 – **Report/ Transmission Menu**	Enables you to send reports to a printer, plotter, screen, drive A:, drive C:, over a serial line, and to cancel previously-requested reports. Enables you to display menus that allow you to add, change, delete and print report options sets and tabular data sets. Also enables you to enter terminal emulation mode.
F7 – **Start Low Pressure Run**	Enables you to start low pressure runs.
F8 – **Start High Pressure Run**	Enables you to start a high pressure run.
F9 – **Run Control/ Status Menu**	Displays the menu that enables you to control and view the status of both low and high pressure runs.
F10 – **Utilities Menu**	Displays the menu that enables you to perform system maintenance functions such as backing up diskettes and setting system and communications options.

ENTERING AND MAINTAINING SAMPLE INFORMATION

Before a sample can be automatically analyzed, it must have an information file assigned to it. The file consists of information groups which, collectively, identify the sample, guide the analysis and specify the report options. This section describes the functions used to enter and maintain sample information.

3. SAMPLE INFORMATION MENU

```
PORESIZER 9320 VX.XX                 DIR: DATA1          DATE:07/13/89 TIME:10:07:32
LP:      14.70 psia IDLE                                 HP:      14.7 psia IDLE
PORT       1        2                                    PORT       1
SAMPLE                                                   SAMPLE
INTVOL     0.000    0.000    mL/g                        INTVOL     0.000    mL/g
% STEM                                                   % STEM

                            3. SAMPLE INFORMATION MENU

  F1  - Help                                 F2  - Main Function Menu

  F3  - Add Sample Information               F4  - Change Sample Information

  F5  - Delete Sample Information            F6  - Print Sample Information

  F7  - Directory of Sample Information


Press Desired Key
```

FUNCTION	DESCRIPTION
F1 - **Help**	Displays the HELP screens.
F2 - **Main Function Menu**	Displays the Main Function Menu.
F3 - **Add Sample Information**	Enables you to add sample information.
F4 - **Change Sample Information**	Enables you to change sample information.
F5 - **Delete Sample Information**	Enables you to delete sample information.

FUNCTION	DESCRIPTION
F6 – **Print Sample Information**	Enables you to print sample information.
F7 – **Directory of Sample Information**	Enables you to print or display a directory of sample information.

3.3p1 ADD SAMPLE INFORMATION

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/13/89 TIME:09:13:53
LP:     14.70 psia IDLE                               HP:      14.7 psia IDLE
PORT       1        2                                 PORT       1
SAMPLE                                                SAMPLE
INTVOL    0.000    0.000    mL/g                      INTVOL    0.000    mL/g
% STEM                                                % STEM

                      3.3p1 ADD SAMPLE INFORMATION

Sample number:     1
Sample ID:  REFERENCE MATERIAL
Submitter ID:
Operator ID:  JANE BARRETT

Pntr number:  05-0673                     Pntr constant:  10.790  µL/pF
Pntr wt:     1.0000  g                    Max head press:  4.680   psi
Pntr vol:   1.0000  mL                    Stem vol:  0.4120  mL

Advancing contact <:  130.000  deg        Receding contact <:  130.000  deg
Hg surf ten:  485.000  dynes/cm           Hg density:  13.5335  g/mL
Samp wt:     1.5000  g
                                                                 [PgDn]
```

FIELD	DESCRIPTION
Sample number:	*Range:* *1 to 400* Defaults to the lowest available number in the current sample directory. If you do not use the default value, the number you enter must not already exist in the current sample directory.
Sample ID:	*Alphanumeric* *Length:* 40
Submitter ID:	*Alphanumeric* *Length:* 40
Operator ID:	*Alphanumeric* *Length:* 20

FIELD	DESCRIPTION
Pntr number:	*Penetrometer number* *Alphanumeric* *Length: 10* The penetrometer number is etched in the bulb of the penetrometer.
Pntr constant:	*Penetrometer constant* *Range: 10.000 to 50.000 μL/pF* Refer to Table 4-1 for the correct penetrometer constant.
Pntr wt:	*Penetrometer weight* *Range: 1.0000 to 1000.0000 g*
Max head press:	*Maximum head pressure* *Range: 3.000 to 5.000 psi or 0.021 to 0.034 MPa* Refer to Table 4-1 for the correct maximum head pressure.
Pntr vol:	*Penetrometer volume* *Range: 1.0000 to 30.0000 mL* This field is optional. It is required only when measuring density.
Stem vol:	*Penetrometer stem volume* *Range: 0.2000 to 10.0000 mL* Refer to Table 4-1 for the correct stem volume.
Advancing contact:	*Advancing contact angle* *Range: 90.100 to 179.900 degrees*
Receding contact:	*Receding contact angle* *Range: 90.100 to 179.900 degrees*
Hg surf ten:	*Mercury surface tension* *Range: 400.000 - 600.000 dynes/cm*
Hg density:	*Mercury density* *Range: 13.0000 to 14.0000 g/mL*
Sample weight:	*Range: 0.0010 to 1000.0000 g*

Use the arrow keys to move from field to field. Enter the desired value in each field and press [↵], or just press [↵] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
[PgDn]	Go to page 2, Report Options.
[F2]	Save the information you entered and return to the Sample Information Menu.
[Esc]	Discard the information you entered and return to the Sample Information Menu.

3.3p2 ADD SAMPLE - REPORT OPTIONS

```
PORESIZER 9320 VX.XX                 DIR: DATA1        DATE:07/13/89 TIME:10:28:15
LP:     14.70 psia IDLE                                HP:     14.7 psia IDLE
PORT         1          2                              PORT         1
SAMPLE                                                 SAMPLE
INTVOL      0.000      0.000    mL/g                   INTVOL      0.000    mL/g
% STEM                                                 % STEM

                    3.3p2 ADD SAMPLE  -  REPORT OPTIONS                [PgUp]

Report set number:    0
Report set ID:
Report negative intrusion?    no
                      Report Heading and Summary Page

Report title:

Summary page?  yes

Calculation range specified as?  pressure

Calculation range from      0.000   to   31000.000   psia
                                                                       [PgDn]
```

FIELD	DESCRIPTION
Report set number:	*Range:* *1 to 50* After you enter the report options set number, the values stored in the set are displayed. You can change the values for this sample only; the report options set will not be affected. If you do not enter a report options set number, the default values will be used for reports unless you change them individually.
Report set ID:	The identification of the report options set is displayed.
Report negative intrusion?	*Toggle* *Choices:* *yes or no*
Report title:	*Alphanumeric* *Length:* *50* When reports are printed, the title is automatically centered.

FIELD	DESCRIPTION
Summary page?	*Toggle* *Choices: yes or no*
Calculation range specified as?	*Toggle* *Choices: pressure or diameter (radius)*
Calculation range from to:	**Pressure** *Range: 0.000 to 61000.000 psia* *0.000 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*

Use the arrow keys to move from field to field. Enter the desired value in each field and press [Enter], or just press [Enter] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
[PgUp]	Go to page 1, Add Sample Information.
[PgDn]	Go to page 3, Tabular Data.
[F2]	Save the information you entered and return to the Sample Information Menu.
[Esc]	Discard the information you entered and return to the Sample Information Menu.

3.3p3 ADD SAMPLE - TABULAR DATA

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/13/89 TIME:10:29:49
LP:     14.70 psia IDLE                               HP:      14.7 psia IDLE
PORT      1         2                                 PORT       1
SAMPLE                                                SAMPLE
INTVOL    0.000     0.000    mL/g                     INTVOL    0.000    mL/g
% STEM                                                % STEM

                         3.3p3 ADD SAMPLE  -  TABULAR DATA                 [PgUp]

Tabular data page?  yes

Tabular data defined by?  Collected points


Column 1?  Pressure
Column 2?  Pore diameter
Column 3?  Mean diameter
Column 4?  Cumulative intrusion volume
Column 5?  Incremental intrusion volume
Column 6?  Differential intrusion volume

                                                                           [PgDn]
```

FIELD	DESCRIPTION
Tabular data page?	*Toggle* *Choices: yes or no*
Tabular data defined by?	*Toggle* *Choices: collected points or tabular data set* If you choose tabular data set, the following prompt is displayed.
Tabular data set number:	*Range: 1 to 50 (must be an existing number)* This field is displayed only if you selected **tabular data set** at the previous prompt. After you enter an existing tabular data set number, the tabular data set ID is displayed.

FIELD	DESCRIPTION
Columns 1 through 6	*Toggle* *(All remaining fields are toggle fields with the same choices)* Enter values for columns 1 - 6. *Choices:* *none* *pressure* *pore diameter (radius)* *mean diameter (radius)* *cumulative intrusion volume* *incremental intrusion volume* *differential intrusion volume* *log differential intrusion volume* *cumulative pore area* *incremental pore area* *percent of total intrusion volume*

Use the arrow keys to move from field to field. Enter the desired value in each field and press [↵], or just press [↵] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
[PgUp]	Go to page 2, Report Options.
[PgDn]	Go to page 4, Graph Page 1.
[F2]	Save the information you entered and return to the Sample Information Menu.
[Esc]	Discard the information you entered and return to the Sample Information Menu.

3.3p4 ADD SAMPLE - GRAPH PAGE1

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/13/89 TIME:10:30:44
LP:     14.70 psia IDLE                               HP:      14.7 psia IDLE
PORT        1          2                              PORT        1
SAMPLE                                                SAMPLE
INTVOL      0.000      0.000    mL/g                  INTVOL      0.000    mL/g
% STEM                                                % STEM

                    3.3p4 ADD SAMPLE  -  GRAPH PAGE1                        [PgUp]

Graph?  yes

X-axis choice?  diameter                  X-axis scale?  Log

X-axis range from     300.000   to      0.006   μm

Y-axis choice?  Cumulative intrusion volume

Y-axis range from       0.000   to      0.000   mL/g

Type of data to plot?  Collected data and continuous curve

Intrusion data to plot?  All              Extrusion data to plot?  All
                                                                            [PgDn]
```

FIELD	DESCRIPTION
Graph?	*Toggle* *Choices:* *yes or no* If you choose no, the following fields are not displayed and no further graph pages may be specified.
X-axis choice?	*Toggle* *Choices:.* *pressure or diameter (radius)*
X-axis scale?	*Toggle* *Choices:* *log or normal*

FIELD	DESCRIPTION
X-axis range from to:	<u>**Pressure**</u> *Range:* *0.100 to 61000.000 psia* *0.001 to 420.580 MPa* <u>**Diameter**</u> *Range:* *0.001 to 5000.000 µm* *5 to 50000000 Å* <u>**Radius**</u> *Range:* *0.000 to 2500.000 µm* *3 to 25000000 Å* Values may be entered in ascending or descending order.
Y-axis choice?	*Toggle* *Choices:* *cumulative intrusion volume* *incremental intrusion volume* *differential intrusion volume* *log differential intrusion volume* *cumulative pore area* *incremental pore area*
Y-axis range from to:	If Y-axis choice is cumulative or incremental intrusion volume: *Range:* *-100.000 to 100.000 mL/g* If Y-axis choice is log differential intrusion volume, cumulative or incremental pore area: *Range:* *-10000.000 to 10000.000 mL/g or sq-m/g* If Y-axis choice is differential intrusion volume: *Range:* *-20000.000 to 20000.000 mL/g-µm if radius and µm* *-10000.000 to 10000.000 mL/g-µm if diameter and µm* *-1.000 to 1.000 mL/g-Å if diameter and Å* *-2.000 to 2.000 mL/g-Å if radius and Å* 0 = autoscale to 105% of the maximum value in the data. Values may be entered in ascending or descending order.

FIELD	DESCRIPTION
Type of data to plot?	*Toggle* *Choices: collected points* *continuous curve* *collected points and continuous curve*
Intrusion data to plot?	*Toggle* *Choices: none, first, all* Displays only if you chose **cumulative intrusion volume** for Y-axis choice.
Extrusion data to plot?	*Toggle* *Choices: none, first, all* Displays only if you chose **cumulative intrusion volume** for Y-axis choice.

Use the arrow keys to move from field to field. Enter the desired value in each field and press [Enter], or just press [Enter] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
[PgUp]	Go to page 3, Tabular Data.
[PgDn]	Go to page 5, Graph Page 2 if you answered **yes** to the graph prompt. Save the information you entered and return to the Sample Information Menu if you answered **no** to the graph prompt.
[F2]	Save the information you entered and return to the Sample Information Menu.
[Esc]	Discard the information you entered and return to the Sample Information Menu.

3.3p5 ADD SAMPLE - GRAPH PAGE2

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/13/89 TIME:10:31:27
LP:      14.70 psia IDLE                              HP:      14.7 psia IDLE
PORT        1        2                                PORT        1
SAMPLE                                                SAMPLE
INTVOL    0.000    0.000   mL/g                       INTVOL    0.000    mL/g
% STEM                                                % STEM

                  3.3p5 ADD SAMPLE  -  GRAPH PAGE2                     [PgUp]

Graph?  yes

X-axis choice?  diameter              X-axis scale?  Log

X-axis range from    300.000   to     0.006    µm

Y-axis choice?  Incremental intrusion volume

Y-axis range from      0.000   to     0.000    mL/g

Type of data to plot?  Collected data and continuous curve

                                                                       [PgDn]
```

Refer to screen 3.3p4 Add Sample - Graph Page 1 for field descriptions.

Use the arrow keys to move from field to field. Enter the desired value in each field and press [↵], or just press [↵] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
[PgUp]	Go to page 4, Graph Page 1.
[PgDn]	Go to page 6, Graph Page 3 if you answered **yes** to the graph prompt. Save the information you entered and return to the Sample Information Menu if you answered **no** to the graph prompt.
[F2]	Save the information you entered and return to the Sample Information Menu.
[Esc]	Discard the information you entered and return to the Sample Information Menu.

3.3p6 ADD SAMPLE - GRAPH PAGE3

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/13/89 TIME:10:32:08
LP:    14.70 psia IDLE                                HP:    14.7 psia IDLE
PORT       1          2                               PORT       1
SAMPLE                                                SAMPLE
INTVOL     0.000      0.000    mL/g                   INTVOL     0.000    mL/g
% STEM                                                % STEM

                    3.3p6 ADD SAMPLE  -  GRAPH PAGE3                        [PgUp]
Graph?  yes
X-axis choice?  diameter                   X-axis scale?  Log
X-axis range from    300.000  to      0.006    µm
Y-axis choice?  Differential intrusion volume
Y-axis range from      0.000  to      0.000    mL/g-µm
Type of data to plot?  Collected data and continuous curve

                                                                            [PgDn]
```

Refer to screen 3.3p4 Add Sample - Graph Page 1 for field descriptions.

Use the arrow keys to move from field to field. Enter the desired value in each field and press ↵, or just press ↵ to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
PgUp	Go to page 5, Graph Page 2.
PgDn	Go to page 7, Graph Page 4 if you answered **yes** to the graph prompt. Save the information you entered and return to the Sample Information Menu if you answered **no** to the graph prompt.
F2	Save the information you entered and return to the Sample Information Menu.
Esc	Discard the information you entered and return to the Sample Information Menu.

3.3p7 ADD SAMPLE - GRAPH PAGE4

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/13/89 TIME:10:32:47
LP:     14.70 psia IDLE                               HP:     14.7 psia IDLE
PORT       1        2                                 PORT       1
SAMPLE                                                SAMPLE
INTVOL    0.000    0.000    mL/g                      INTVOL    0.000    mL/g
% STEM                                                % STEM

                 3.3p7 ADD SAMPLE  -  GRAPH PAGE4                          [PgUp]

Graph?  yes

X-axis choice?  diameter                   X-axis scale?  Log

X-axis range from    300.000   to     0.006   µm

Y-axis choice?  Log differential intrusion volume

Y-axis range from      0.000   to     0.000   mL/g

Type of data to plot?  Collected data and continuous curve

                                                                           [PgDn]
```

Refer to screen 3.3p4 Add Sample - Graph Page 1 for field descriptions.

Use the arrow keys to move from field to field. Enter the desired value in each field and press ⏎, or just press ⏎ to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
PgUp	Go to page 6, Graph Page 3.
PgDn	Go to page 8, Graph Page 5 if you answered **yes** to the graph prompt.
	Save the information you entered and return to the Sample Information Menu if you answered **no** to the graph prompt.
F2	Save the information you entered and return to the Sample Information Menu.
Esc	Discard the information you entered and return to the Sample Information Menu.

3.3p8 ADD SAMPLE - GRAPH PAGE5

```
PORESIZER 9320 VX.XX              DIR: DATA1            DATE:07/13/89 TIME:10:33:30
LP:     14.70 psia IDLE                                 HP:      14.7 psia IDLE
PORT        1         2                                 PORT          1
SAMPLE                                                  SAMPLE
INTVOL      0.000     0.000    mL/g                     INTVOL     0.000    mL/g
% STEM                                                  % STEM

                  3.3p8 ADD SAMPLE  -  GRAPH PAGE5                          [PgUp]

Graph?  yes

X-axis choice?  diameter                 X-axis scale?  Log

X-axis range from     300.000  to      0.006    µm

Y-axis choice?  Cumulative pore area

Y-axis range from       0.000  to      0.000    sq-m/g

Type of data to plot?  Collected data and continuous curve
```

Refer to screen 3.3p4 Add Sample - Graph Page 1 for field descriptions.

Use the arrow keys to move from field to field. Enter the desired value in each field and press [↵] or just press [↵] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
[PgUp]	Go to page 7, Graph Page 4.
[F2]	Save the information you entered and return to the Sample Information Menu.
[Esc]	Discard the information you entered and return to the Sample Information Menu.

3.4p1 THROUGH 3.4p8 CHANGE SAMPLE INFORMATION

Refer to screens 3.3p1 through 3.3p8 for sample screens and field descriptions for the eight pages of sample information.

NOTE

The following prompt appears on screen 3.4p1 after a low pressure run.

Samp wt + Pntr wt + Hg wt:

Enter this weight if density calculations are to be made. The range for this field is 1.0000 to 1000.0000 g.

NOTE

You cannot change a sample that is currently active.

To change information, enter the number of the sample you wish to change and press [Enter]. The information for the selected sample will be displayed. Use the arrow keys to move to the desired fields and enter the changes. Use [PgDn] and [PgUp] to move between pages.

3.5 DELETE SAMPLE INFORMATION

Refer to screen 3.3p1 for a sample screen and field descriptions.

NOTE

You cannot delete a sample that is currently active.

To delete information, enter the number of the sample you wish to delete and press [↵]. The information for the selected sample will be displayed. Press [PgDn] to delete the information.

3.6 PRINT SAMPLE INFORMATION

Refer to screen 3.3p1 for a sample screen and field descriptions. To print information, enter the number of the sample you wish to print and press [↵]. The information for the selected sample will be displayed. Press [PgDn] to print the information.

3.7 DIRECTORY OF SAMPLE INFORMATION

```
PORESIZER 9320 VX.XX            DIR: DATA1          DATE:07/13/89 TIME:11:59:29
LP:    14.70 psia IDLE                              HP:     14.7 psia IDLE
PORT       1        2                               PORT       1
SAMPLE                                              SAMPLE
INTVOL     0.000    0.000   mL/g                    INTVOL     0.000    mL/g
% STEM                                              % STEM

                  3.7 DIRECTORY OF SAMPLE INFORMATION

  Since Date:    1 /  1 / 88

  Directory of ?   all samples

  List directory on ?   screen




Press [PgDn] to produce directory, or [Esc] for no action
```

FIELD	DESCRIPTION	
Since date:	*Format:*	*month/day/year*
Directory of?	*Toggle Choices:*	*all samples* *samples with no runs* *samples now running LP* *samples with LP complete* *samples now running HP* *samples with HP complete*
List directory on?	*Toggle Choices:*	*screen or printer*

Use the arrow keys to move from field to field. Enter the desired value in each field and press [↵], or just press [↵] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
[PgDn]	Print or display the directory. The directory contains the following information for each sample information file: the sample file number, date and time the file was created or edited last, the sample file ID, and the status of the sample file.
[Esc]	Discard the information you entered and return to the Sample Information Menu.

ENTERING AND MAINTAINING PRESSURE TABLE INFORMATION

4. PRESSURE TABLE MENU

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/13/89 TIME:12:00:13
LP:     14.70 psia IDLE                               HP:      14.7 psia IDLE
PORT        1        2                                PORT        1
SAMPLE                                                SAMPLE
INTVOL    0.000    0.000   mL/g                       INTVOL    0.000    mL/g
% STEM                                                % STEM

                         4. PRESSURE TABLE MENU

F1  - Help                               F2  - Main Function Menu

F3  - Add Pressure Table                 F4  - Change Pressure Table

F5  - Delete Pressure Table              F6  - Print Pressure Table

F7  - Directory of Pressure Tables


Press Desired Key
```

FUNCTION	DESCRIPTION
F1 – **Help**	Displays the HELP screens.
F2 – **Main Function Menu**	Displays the Main Function Menu.
F3 – **Add Pressure Table**	Enables you to add pressure tables.
F4 – **Change Pressure Table**	Enables you to change pressure tables.
F5 – **Delete Pressure Table**	Enables you to delete pressure tables.

FUNCTION	DESCRIPTION
F6 – Print Pressure Table	Enables you to print pressure tables.
F7 – Directory of Pressure Tables	Enables you to print or display a directory of pressure tables.

4.3 ADD PRESSURE TABLE

```
PORESIZER 9320 VX.XX            DIR: DATA1          DATE:07/13/89 TIME:12:15:01
LP:    14.70 psia IDLE                              HP:     14.7 psia IDLE
PORT       1        2                               PORT       1
SAMPLE                                              SAMPLE
INTVOL    0.000    0.000   mL/g                     INTVOL    0.000    mL/g
% STEM                                              % STEM

                         4.3 ADD PRESSURE TABLE

Pressure table number:   2  in psia
Pressure table ID:  SILICA
  1)      100.000
  2)      300.000
  3)      500.000
  4)      700.000
  5)      900.000
  6)     1100.000
  7)     1350.000
  8)     1600.000
  9)     1800.000
 10)     1950.000
```

FIELD	DESCRIPTION
Pressure table number:	*Range:* *1 to 50* Enter a pressure table number from 1 to 50 that does not already exist.
Pressure table ID:	*Alphanumeric* *Length:* *24*
Pressure table values Range:	*10.000 to 30000.000 psia* *0.069 to 206.843 MPa* The table may contain up to 250 pressure points and must contain at least one point.

Use the arrow keys to move from field to field. Enter the desired value in each field and press ↵, or just press ↵ to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
Ins	Insert a point in the pressure table.
Del	Delete a point in the pressure table.
F2	Save the information you entered and return to the Pressure Table Menu.
Esc	Discard the information you entered and return to the Pressure Table Menu.

4.4 CHANGE PRESSURE TABLE

Refer to screen 4.3 Add Pressure Table for a sample screen and field descriptions. To change information, enter the number of the pressure table you wish to change and press [↵]. The information for the selected pressure table will be displayed. Use the arrow keys to move to the desired fields and enter the changes.

4.5 DELETE PRESSURE TABLE

Refer to screen 4.3 Add Pressure Table for a sample screen and field descriptions. To delete information, enter the number of the existing pressure table you wish to delete and press [↵]. The pressure table will be displayed. Press [PgDn] to delete the table.

4.6 PRINT PRESSURE TABLE

Refer to screen 4.3 Add Pressure Table for a sample screen and field descriptions. To print a pressure table, enter the number of the existing pressure table you wish to print and press [↵]. The selected pressure table will be displayed. Press [PgDn] to print the table.

4.7 DIRECTORY OF PRESSURE TABLES

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/13/89 TIME:12:18:38
LP:     14.70 psia IDLE                               HP:     14.7 psia IDLE
PORT        1         2                               PORT        1
SAMPLE                                                SAMPLE
INTVOL     0.000     0.000    mL/g                    INTVOL     0.000    mL/g
% STEM                                                % STEM

                    4.7 DIRECTORY OF PRESSURE TABLES

Since date:   1 /  1 / 89

List directory on?  screen




Press [PgDn] to produce directory, or [Esc] for no action
```

FIELD	DESCRIPTION
Since date:	*Format:* *month/day/year*
List directory on?	*Toggle* *Choices:* *screen or printer*

Use the arrow keys to move from field to field. Enter the desired value in each field and press ↵, or just press ↵ to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
PgDn	Print or display the directory. The directory contains the following information for each pressure table: the pressure table number, date and time the table was created or edited last, and the pressure table ID.
Esc	Discard the information you entered and return to the Pressure Table Menu.

ENTERING AND MAINTAINING RATE SEQUENCE TABLES

5. RATE SEQUENCE TABLE MENU

```
PORESIZER 9320 VX.XX              DIR: DATA1            DATE:07/13/89 TIME:12:52:08
LP:     14.70 psia IDLE                                 HP:     14.7 psia IDLE
PORT         1        2                                 PORT         1
SAMPLE                                                  SAMPLE
INTVOL    0.000    0.000   mL/g                         INTVOL    0.000    mL/g
% STEM                                                  % STEM

                         5. RATE SEQUENCE TABLE MENU

  F1  - Help                             F2  - Main Function Menu

  F3  - Add Rate Sequence Table          F4  - Change Rate Sequence Table

  F5  - Delete Rate Sequence Table       F6  - Print Rate Sequence Table

  F7  - Directory of Rate Sequence Tables


Press Desired Key
```

FUNCTION	DESCRIPTION
F1 – Help	Displays the HELP screens.
F2 – Main Function Menu	Displays the Main Function Menu.
F3 – Add Rate Sequence Table	Enables you to add rate sequence tables.
F4 – Change Rate Sequence Table	Enables you to change rate sequence tables.
F5 – Delete Rate Sequence Table	Enables you to delete rate sequence tables.

FUNCTION	DESCRIPTION
F6 – Print Rate Sequence Table	Enables you to print rate sequence tables.
F7 – Directory of Rate Sequence Tables	Enables you to print or display a directory of rate sequence tables.

5.3 ADD RATE SEQUENCE TABLE

A rate sequence table enables you to collect data during an automatic non-equilibrating high pressure run in Scanning Mode. It works in conjunction with a pressure table. The pressure table contains the pressure points at which data are collected. The rate sequence table determines the range and rate of pressure generation. Each entry consists of a pressure generation rate and a pressure boundary at which the rate will change to the next rate in the table. Data collection continues until the final pressure boundary in the table is reached.

The pressure table and the rate sequence table must complement each other. The PoreSizer will follow the pressurization schedule that is given in the rate sequence table. If your pressure table contains a point outside of the rate table pressures, then that point will not be taken and data collection will stop.

```
PORESIZER-9320 VX.XX              DIR: DATA1           DATE:07/14/89 TIME:08:23:09
LP:      14.70 psia IDLE                               HP:      14.7 psia IDLE
PORT      1         2                                  PORT      1
SAMPLE                                                 SAMPLE
INTVOL    0.000     0.000    mL/g                      INTVOL    0.000    mL/g
% STEM                                                 % STEM

                     5.3 ADD RATE SEQUENCE TABLE

Rate sequence table number:   1  in psia
Rate sequence table ID:  RATE TABLE 1

  1)     2000.000       Low
  2)     3000.000       Medium low
  3)     4000.000       Medium low
  4)     5000.000       Medium low
  5)     6000.000       Medium low
  6)     7000.000       Medium low
  7)     8000.000       Medium high
  8)     9000.000       Medium high
  9)    10000.000       Medium high
```

FIELD	DESCRIPTION
Rate sequence table number:	*Range:* *1 to 50* Enter a rate sequence table number from 1 to 50 that does not already exist.
Rate Sequence table ID:	*Alphanumeric* *Length:* *24*

FIELD	DESCRIPTION
Pressure	*Range:* *10.000 to 30000.000 psia* *0.069 to 206.843 MPa* Enter the pressure boundaries at which the rate will change to the next rate in the table.
Rate?	*Toggle Choices:* *low, medium low, medium high, high* High is equivalent to the current setting of the RATE control knob during the run. The table may contain up to 100 entries and must contain at least one.

Use the arrow keys to move from field to field. Enter the desired value in each field and press [↵], or just press [↵] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
[Ins]	Insert a point in the rate sequence table.
[Del]	Delete a point in the rate sequence table.
[F2]	Save the information you entered and return to the Rate Sequence Table Menu.
[Esc]	Discard the information you entered and return to the Rate Sequence Table Menu.

5.4 CHANGE RATE SEQUENCE TABLE

Refer to screen 5.3 Add Rate Sequence Table for a sample screen and field descriptions. To change information, enter the number of the rate sequence table you wish to change and press [↵]. The information for the selected rate sequence table will be displayed. Use the arrow keys to move to the desired fields and enter the changes.

5.5 DELETE RATE SEQUENCE TABLE

Refer to screen 5.3 Add Rate Sequence Table for a sample screen and field descriptions. To delete information, enter the number of the rate sequence table you wish to delete and press [↵]. The table will be displayed. Press [PgDn] to delete the table.

5.6 PRINT RATE SEQUENCE TABLE

Refer to screen 5.3 Add Rate Sequence Table for a sample screen and field descriptions. To print a rate sequence table, enter the number of the rate sequence table you wish to print and press [↵]. The selected rate sequence table will be displayed. Press [PgDn] to print the table.

5.7 DIRECTORY OF RATE SEQUENCE TABLES

```
PORESIZER 9320 VX.XX              DIR: DATA1        DATE:07/13/89 TIME:12:53:29
LP:    14.70 psia IDLE                              HP:     14.7 psia IDLE
PORT       1        2                               PORT       1
SAMPLE                                              SAMPLE
INTVOL     0.000    0.000    mL/g                   INTVOL     0.000    mL/g
% STEM                                              % STEM

                    5.7 DIRECTORY OF RATE SEQUENCE TABLES

Since date:    1 /  1 / 88

List directory on?  screen




Press [PgDn] to produce directory, or [Esc] for no action
```

FIELD	DESCRIPTION
Since date:	*Format:* *month/day/year*
List directory on?	*Toggle* *Choices:* *screen or printer*

Use the arrow keys to move from field to field. Enter the desired value in each field and press [↵], or just press [↵] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
[PgDn]	Print or display the directory. The directory contains the following information for each rate sequence table: the rate sequence table number, date and time the table was created or edited last, and the rate sequence table ID.
[Esc]	Discard the information you entered and return to the Rate Sequence Table Menu.

ENTERING AND MAINTAINING REPORT INFORMATION

6. REPORT/TRANSMISSION MENU

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/13/89 TIME:12:54:22
LP:     14.70 psia IDLE                               HP:     14.7 psia IDLE
PORT        1         2                               PORT        1
SAMPLE                                                SAMPLE
INTVOL     0.000     0.000    mL/g                    INTVOL     0.000    mL/g
% STEM                                                % STEM

                         6. REPORT/TRANSMISSION MENU

  F1  - Help                             F2  - Main Function Menu

  F3  - Start Report                     F4  - Cancel Reports

  F5  - Report Options Set Menu          F6  - Tabular Data Set Menu

  F7  - Terminal Emulation


Press Desired Key
```

FUNCTION	DESCRIPTION
F1 – Help	Displays the HELP screens.
F2 – Main Function Menu	Displays the Main Function Menu.
F3 – Start Report	Enables you to start transmitting a report to the printer, plotter, screen, serial line, drive A: or drive C:.
F4 – Cancel Reports	Enables you to cancel reports that are in process.
F5 – Report Options Set Menu	Displays the menu that enables you to add, change, delete and print report options sets and to display or print a directory of report options sets.

FUNCTION	DESCRIPTION
F6 – **Tabular Data Set Menu**	Displays the menu that enables you to add, change, delete and print tabular data sets and to display or print a directory of tabular data sets.
F7 – **Terminal Emulation**	Enables you to run the control module in terminal emulation mode.

NOTE

The instructions for screen 6.3 Start Report and 6.4 Cancel Reports are included in Chapter 5, Printing Reports.

6.5 REPORT OPTIONS SET MENU

```
PORESIZER 9320 VX.XX              DIR: DATA1           DATE:07/13/89 TIME:13:39:44
LP:    14.70 psia IDLE                                 HP:     14.7 psia IDLE
PORT        1        2                                 PORT        1
SAMPLE                                                 SAMPLE
INTVOL    0.000    0.000    mL/g                       INTVOL    0.000    mL/g
% STEM                                                 % STEM

                            6.5 REPORT OPTIONS SET MENU

  F1  - Help                               F2  - Report/Transmission Menu

  F3  - Add Report Options Set             F4  - Change Report Options Set

  F5  - Delete Report Options Set          F6  - Print Report Options Set

  F7  - Directory of Report Options Sets


Press Desired Key
```

FUNCTION	DESCRIPTION
F1 – Help	Displays the HELP screens.
F2 – Report/ Transmission Menu	Displays the Report/Transmission Menu.
F3 – Add Report Options Set	Enables you to add report options sets.
F4 – Change Report Options Set	Enables you to change report options sets.
F5 – Delete Report Options Set	Enables you to delete report options sets.
F6 – Print Report Options Set	Enables you to print report options sets.
F7 – Directory of Report Options Sets	Enables you to print or display a directory of report options sets.

6.5.3p1 ADD REPORT OPTIONS SET

```
PORESIZER 9320 VX.XX            DIR: DATA1          DATE:07/13/89 TIME:13:41:43
LP:     14.70 psia IDLE                             HP:      14.7 psia IDLE
PORT      1          2                              PORT       1
SAMPLE                                              SAMPLE
INTVOL    0.000      0.000    mL/g                  INTVOL     0.000    mL/g
% STEM                                              % STEM

                         6.5.3p1 ADD REPORT OPTIONS SET

Report set number:    1
Report set ID:
Report negative intrusion?     no

                         Report Heading and Summary Page

Report title:

Summary page?  yes

Calculation range specified as?   pressure

Calculation range from       0.000   to   31000.000    psia
                                                                        [PgDn]
```

FIELD	DESCRIPTION
Report set number:	*Range:* *1 to 50* Enter a report options set number from 1 to 50 that does not already exist.
Report set ID:	*Alphanumeric* *Length:* *24*
Report negative intrusion?	*Toggle* *Choices:* *yes or no*
Report title:	*Alphanumeric* *Length:* *50* When reports are printed, the title is automatically centered.
Summary page?	*Toggle* *Choices:* *yes or no*

FIELD	DESCRIPTION
Calculation range specified as?	*Toggle* *Choices:* *pressure or diameter (radius)*
Calculation range from to:	**Pressure** *Range:* *0.000 to 61000.000 psia* *0.000 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*

Use the arrow keys to move from field to field. Enter the desired value in each field and press [Enter], or just press [Enter] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
[PgDn]	Go to page 2, Tabular Data.
[F2]	Save the information you entered and return to the Report Options Set Menu.
[Esc]	Discard the information you entered and return to the Report Options Set Menu.

6.5.3p2 ADD REPORT OPTIONS - TABULAR DATA

```
PORESIZER 9320 VX.XX              DIR: DATA1               DATE:08/13/89 TIME:16:59:09
LP:     14.70 psia IDLE                                    HP:      14.7 psia IDLE
PORT        1          2                                   PORT        1
SAMPLE                                                     SAMPLE
INTVOL     0.000      0.000     mL/g                       INTVOL     0.000     mL/g
% STEM                                                     % STEM

                      6.5.3p2 ADD REPORT OPTIONS  -  TABULAR DATA          [PgUp]

Tabular data page?  yes

Tabular data defined by?  Collected points


Column 1?  Pressure
Column 2?  Pore diameter
Column 3?  Mean diameter
Column 4?  Cumulative intrusion volume
Column 5?  Incremental intrusion volume
Column 6?  Differential intrusion volume
                                                                           [PgDn]
```

FIELD	DESCRIPTION
Tabular data page?	*Toggle* *Choices: yes or no*
Tabular data defined by?	*Toggle* *Choices: collected points or tabular data set* If you choose tabular data set, the following prompt is displayed.
Tabular data set number:	*Range: 1 to 50 (must be an existing number)* This field is displayed only if you selected tabular data set at the previous prompt. After you enter an existing tabular data set number, the tabular data set ID is displayed.
Tabular data set ID:	The identification of the tabular data set is displayed.

FIELD	DESCRIPTION
Columns 1 through 6	*Toggle* *(All remaining fields are toggle fields with the same choices)* Select values for columns 1 through 6. *Choices:* *none* *pressure* *pore diameter (radius)* *mean diameter (radius)* *cumulative intrusion volume* *incremental intrusion volume* *differential intrusion volume* *log differential intrusion volume* *cumulative pore area* *incremental pore area* *percent of total intrusion volume*

Use the arrow keys to move from field to field. Enter the desired value in each field and press [Enter], or just press [Enter] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
[PgUp]	Go to page 1, Report Options Set.
[PgDn]	Go to page 3, Graph Page 1.
[F2]	Save the information you entered and return to the Report Options Set Menu.
[Esc]	Discard the information you entered and return to the Report Options Set Menu.

6.5.3p3 ADD REPORT OPTIONS - GRAPH PAGE1

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:08/13/89 TIME:17:00:03
LP:     14.70 psia IDLE                               HP:      14.7 psia IDLE
PORT        1         2                               PORT        1
SAMPLE                                                SAMPLE
INTVOL      0.000     0.000    mL/g                   INTVOL      0.000    mL/g
% STEM                                                % STEM

                   6.5.3p3 ADD REPORT OPTIONS  -  GRAPH PAGE1          [PgUp]

Graph?  yes

X-axis choice?  diameter                X-axis scale?  Log

X-axis range from    300.000  to      0.006    µm

Y-axis choice?  Cumulative intrusion volume

Y-axis range from      0.000  to      0.000    mL/g

Type of data to plot?  Collected data and continuous curve

Intrusion data to plot?  All            Extrusion data to plot?  All
                                                                       [PgDn]
```

FIELD	DESCRIPTION
Graph?	*Toggle* *Choices: yes or no* If you choose no, the following fields are not displayed and no further graph pages may be specified.
X-axis choice?	*Toggle* *Choices: pressure or diameter (radius)*
X-axis scale?	*Toggle* *Choices: log or normal*

FIELD	DESCRIPTION
X-axis range from to:	<u>**Pressure**</u> *Range: 0.100 to 61000.000 psia* *0.001 to 420.580 MPa* <u>**Diameter**</u> *Range: 0.001 to 5000.000 µm* *5 to 500000000 Å* <u>**Radius**</u> *Range: 0.000 to 2500.000 µm* *3 to 25000000 Å* Values may be entered in ascending or descending order.
Y-axis choice?	*Toggle* *Choices: cumulative intrusion volume* *incremental intrusion volume* *differential intrusion volume* *log differential intrusion volume* *cumulative pore area* *incremental pore area*
Y-axis range from to:	If Y-axis choice is cumulative or incremental intrusion volume: *Range: -1000.000 to 100.000 mL/g* If Y-axis choice is log differential intrusion volume, cumulative or incremental pore area: *Range: -10000.000 to 10000.000 mL/g or sq-m/g* If Y-axis choice is differential intrusion volume: *Range: -20000.000 to 20000.000 mL/g-µm if radius and µm* *-10000.000 to 10000.000 mL/g-µm if diameter and µm* *-1.000 to 1.000 mL/g-Å if diameter and Å* *-2.000 to 2.000 mL/g-Å if radius and Å* *0 = autoscale to 105% of the maximum value in the data.* Values may be entered in ascending or descending order.

FIELD	DESCRIPTION
Type of data to plot?	*Toggle* *Choices:* *collected points* *continuous curve* *collected points and continuous curve*
Intrusion data to plot?	*Toggle* *Choices:* *none, first, all* This field is displayed only if you chose **cumulative intrusion volume** for Y-axis choice.
Extrusion data to plot?	*Toggle* *Choices:* *none, first, all* This field is displayed only if you chose **cumulative intrusion volume** for Y-axis choice.

Use the arrow keys to move from field to field. Enter the desired value in each field and press [↵], or just press [↵] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
[PgUp]	Go to page 3, Tabular Data.
[PgDn]	Go to page 4, Graph Page 2 if you answered **yes** to the graph prompt. Save the information you entered and return to the Report Options Menu if you answered **no** to the graph prompt.
[F2]	Save the information you entered and return to the Report Options Menu.
[Esc]	Discard the information you entered and return to the Report Options Menu.

6.5.3p4 ADD REPORT OPTIONS - GRAPH PAGE2

```
PORESIZER 9320 VX.XX            DIR: DATA1              DATE:08/13/89 TIME:17:01:02
LP:    14.70 psia IDLE                                  HP:     14.7 psia IDLE
PORT       1          2                                 PORT        1
SAMPLE                                                  SAMPLE
INTVOL     0.000      0.000    mL/g                     INTVOL      0.000     mL/g
% STEM                                                  % STEM

              6.5.3p4 ADD REPORT OPTIONS  -  GRAPH PAGE2               [PgUp]

Graph?  yes
X-axis choice?  diameter                    X-axis scale?  Log
X-axis range from    300.000  to     0.006   µm
Y-axis choice?  Incremental intrusion volume
Y-axis range from      0.000  to     0.000   mL/g
Type of data to plot?  Collected data and continuous curve

                                                                        [PgDn]
```

Refer to screen 6.5.3p3 Add Report Options - Graph Page 1 for field descriptions.

Use the arrow keys to move from field to field. Enter the desired value in each field and press [Enter], or just press [Enter] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
PgUp	Go to page 3, Graph Page 1.
PgDn	Go to page 5, Graph Page 3 if you answered **yes** to the graph prompt. Save the information you entered and return to the Report Options Menu if you answered **no** to the graph prompt.
F2	Save the information you entered and return to the Report Options Menu.
Esc	Discard the information you entered and return to the Report Options Menu.

6.5.3p5 ADD REPORT OPTIONS - GRAPH PAGE3

```
PORESIZER 9320 VX.XX                DIR: DATA1          DATE:08/13/89 TIME:17:02:03
LP:     14.70 psia IDLE                                 HP:     14.7 psia IDLE
PORT         1          2                               PORT       1
SAMPLE                                                  SAMPLE
INTVOL     0.000      0.000    mL/g                     INTVOL     0.000    mL/g
% STEM                                                  % STEM

                 6.5.3p5 ADD REPORT OPTIONS  -  GRAPH PAGE3                [PgUp]
Graph?  yes

X-axis choice?  diameter                       X-axis scale?  Log

X-axis range from     300.000   to      0.006     µm

Y-axis choice?  Differential intrusion volume

Y-axis range from        0.000   to      0.000     mL/g-µm

Type of data to plot?  Collected data and continuous curve

                                                                           [PgDn]
```

Refer to screen 6.5.3p3 Add Report Options - Graph Page 1 for field descriptions.

Use the arrow keys to move from field to field. Enter the desired value in each field and press [↵], or just press [↵] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
PgUp	Go to page 4, Graph Page 2.
PgDn	Go to page 6, Graph Page 4 if you answered **yes** to the graph prompt.
	Save the information you entered and return to the Report Options Menu if you answered **no** to the graph prompt.
F2	Save the information you entered and return to the Report Options Menu.
Esc	Discard the information you entered and return to the Report Options Menu.

6.5.3p6 ADD REPORT OPTIONS - GRAPH PAGE4

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:08/13/89 TIME:17:02:43
LP:   14.70 psia IDLE                                 HP:    14.7 psia IDLE
PORT       1         2                                PORT       1
SAMPLE                                                SAMPLE
INTVOL    0.000     0.000    mL/g                     INTVOL    0.000    mL/g
% STEM                                                % STEM

                  6.5.3p6 ADD REPORT OPTIONS  -  GRAPH PAGE4           [PgUp]

Graph?  yes

X-axis choice?  diameter                  X-axis scale?  Log

X-axis range from    300.000   to      0.006    µm

Y-axis choice?  Log differential intrusion volume

Y-axis range from      0.000   to      0.000    mL/g

Type of data to plot?  Collected data and continuous curve

                                                                       [PgDn]
```

Refer to screen 6.5.3p3 Add Report Options - Graph Page 1 for field descriptions.

Use the arrow keys to move from field to field. Enter the desired value in each field and press [↵], or just press [↵] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
PgUp	Go to page 5, Graph Page 3.
PgDn	Go to page 7, Graph Page 5 if you answered **yes** to the graph prompt. Save the information you entered and return to the Report Options Menu if you answered **no** to the graph prompt.
F2	Save the information you entered and return to the Report Options Menu.
Esc	Discard the information you entered and return to the Report Options Menu.

6.5.3p7 ADD REPORT OPTIONS - GRAPH PAGE5

```
PORESIZER 9320 VX.XX              DIR: DATA1           DATE:08/13/89 TIME:17:03:39
LP:     14.70 psia IDLE                                HP:      14.7 psia IDLE
PORT       1         2                                 PORT        1
SAMPLE                                                 SAMPLE
INTVOL     0.000     0.000    mL/g                     INTVOL     0.000    mL/g
% STEM                                                 % STEM

                    6.5.3p7 ADD REPORT OPTIONS  -  GRAPH PAGE5          [PgUp]

Graph?  yes

X-axis choice?  diameter               X-axis scale?  Log

X-axis range from    300.000  to      0.006   µm

Y-axis choice?  Cumulative pore area

Y-axis range from      0.000  to      0.000   sq-m/g

Type of data to plot?  Collected data and continuous curve
```

Refer to screen 6.5.3p3 Add Report Options - Graph Page 1 for field descriptions.

Use the arrow keys to move from field to field. Enter the desired value in each field and press ⏎, or just press ⏎ to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
PgUp	Go to page 6, Graph Page 4.
F2	Save the information you entered and return to the Report Options Menu.
Esc	Discard the information you entered and return to the Report Options Menu.

6.5.4p1 THROUGH 6.5.4P7 CHANGE REPORT OPTIONS SET

Refer to screens 6.5.3p1 through 6.5.3p7 for sample screens and field descriptions for the seven pages of report options.

To change information, enter the number of the report options set you wish to change and press [↵]. The information for the selected report options set will be displayed. Use the arrow keys to move to the desired fields and enter the changes.

6.5.5 DELETE REPORT OPTIONS SET

Refer to screen 6.5.3p1 for a sample screen and field descriptions.

To delete information, enter the number of the report options set you wish to delete and press [↵]. The information for the selected report options set will be displayed. Press [PgDn] to delete the information.

6.5.6 PRINT REPORT OPTIONS SET

Refer to screen 6.5.3p1 for a sample screen and field descriptions. To print information, enter the number of the report options set you wish to print and press [↵]. The information for the selected report options set will be displayed. Press [PgDn] to print the information.

6.5.7 DIRECTORY OF REPORT OPTIONS SETS

```
PORESIZER 9320 VX.XX                 DIR: DATA1           DATE:07/14/89 TIME:07:31:29
LP:     14.70 psia IDLE                                   HP:      14.7 psia IDLE
PORT        1          2                                  PORT         1
SAMPLE                                                    SAMPLE
INTVOL     0.000      0.000     mL/g                      INTVOL      0.000    mL/g
% STEM                                                    % STEM

                     6.5.7 DIRECTORY OF REPORT OPTIONS SETS

Since date:    1 /  1 / 89

List directory on?  screen



Press [PgDn] to produce directory, or [Esc] for no action
```

FIELD	DESCRIPTION
Since date:	*Format:* *month/day/year*
List directory on?	*Toggle* *Choices:* *screen or printer*

Use the arrow keys to move from field to field. Enter the desired value in each field and press [↵], or just press [↵] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
[PgDn]	Print or display the directory. The directory contains the following information for each report options set: the report options set number, date and time the file was created or edited last, and the report options set ID.
[Esc]	Discard the information you entered and return to the Report Options Set Menu.

ENTERING AND MAINTAINING TABULAR DATA SETS

6.6 TABULAR DATA SET MENU

```
PORESIZER 9320 VX.XX            DIR: DATA1          DATE:07/14/89 TIME:06:16:53
LP:     14.70 psia IDLE                             HP:     14.7 psia IDLE
PORT     1          2                               PORT      1
SAMPLE                                              SAMPLE
INTVOL   0.000     0.000    mL/g                    INTVOL   0.000    mL/g
% STEM                                              % STEM

                        6.6 TABULAR DATA SET MENU

  F1  - Help                              F2  - Report Menu

  F3  - Add Tabular Data Set              F4  - Change Tabular Data Set

  F5  - Delete Tabular Data Set           F6  - Print Tabular Data Set

  F7  - Directory of Tabular Data Sets


Press Desired Key
```

FUNCTION	DESCRIPTION
F1 - **Help**	Displays the HELP screens.
F2 - **Report Menu**	Displays the Report/Transmission Menu.
F3 - **Add Tabular Data Set**	Enables you to add tabular data sets.
F4 - **Change Tabular Data Set**	Enables you to change tabular data sets.
F5 - **Delete Tabular Data Set**	Enables you to delete tabular data sets.

FUNCTION	DESCRIPTION
F6 - **Print Tabular Data Set**	Enables you to print tabular data sets.
F7 - **Directory of Tabular Data Sets**	Enables you to print or display a directory of tabular data sets.

6.6.3 ADD TABULAR DATA SET

```
PORESIZER 9320 VX.XX             DIR: DATA1          DATE:07/14/89 TIME:06:19:29
LP:     14.70 psia IDLE                              HP:      14.7 psia IDLE
PORT         1         2                             PORT        1
SAMPLE                                               SAMPLE
INTVOL     0.000     0.000    mL/g                   INTVOL     0.000    mL/g
% STEM                                               % STEM

                              6.6.3 ADD TABULAR DATA SET

Tabular data set number:     1
Tabular data set ID:  DATA SET 1
Data type?  pressure  in psia
           1)      1.000
           2)      5.000
           3)     10.000
           4)     20.000
           5)     30.000
           6)     40.000
           7)     50.000
           8)     60.000
           9)     70.000
```

FIELD	DESCRIPTION
Tabular data set number:	*Range:* *1 to 50* Enter a tabular data set number from 1 to 50 that does not already exist.
Tabular data set ID:	*Alphanumeric* *Length:* *24*
Data type?	*Toggle* *Choices:* *pressure or diameter (radius)*

FIELD	DESCRIPTION
Data values	<u>**Pressure**</u> *Range: 0.100 to 61000.000 psia* *0.001 to 420.580 MPa* <u>**Diameter**</u> *Range: 0.0005 to 5000.0000 μm* *5 to 50000000 Å* <u>**Radius**</u> *Range: 0.0003 to 2500.0000 μm* *3 to 25000000 Å* You can enter up to 250 data values. The minimum entry is one.

Use the arrow keys to move from field to field. Enter the desired value in each field and press [Enter], or just press [Enter] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
[Ins]	Insert a point in the table.
[Del]	Delete a point in the table.
[F2]	Save the information you entered and return to the Tabular Data Set Menu.
[Esc]	Discard the information you entered and return to the Tabular Data Set Menu.

6.6.4 CHANGE TABULAR DATA SET

Refer to screen 6.6.3 Add Tabular Data Set for a sample screen and field descriptions.

To change information, enter the number of the tabular data set you wish to change and press [↵]. The information for the selected tabular data set will be displayed. Use the arrow keys to move to the desired fields and enter the changes.

6.6.5 DELETE TABULAR DATA SET

Refer to screen 6.6.3 Add Tabular Data Set for a sample screen and field descriptions.

To delete information, enter the number of the tabular data set you wish to delete and press [↵]. The information for the selected tabular data set will be displayed. Press [PgDn] to delete the set.

6.6.6 PRINT TABULAR DATA SET

Refer to screen 6.6.3 Add Tabular Data Set for a sample screen and field descriptions. To print information, enter the number of the tabular data set you wish to print and press [↵]. The selected tabular data set will be displayed. Press [PgDn] to print the set.

6.6.7 DIRECTORY OF TABULAR DATA SETS

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/14/89 TIME:06:20:21
LP:     14.70 psia IDLE                               HP:      14.7 psia IDLE
PORT       1          2                               PORT       1
SAMPLE                                                SAMPLE
INTVOL    0.000      0.000     mL/g                   INTVOL    0.000    mL/g
% STEM                                                % STEM

                      6.6.7 DIRECTORY OF TABULAR DATA SETS

Since date:    1 /  1 / 89

List directory on?  screen




Press [PgDn] to produce directory, or [Esc] for no action
```

FIELD	DESCRIPTION
Since date:	*Format: month/day/year*
List directory on?	*Toggle* *Choices: screen or printer*

Use the arrow keys to move from field to field. Enter the desired value in each field and press [↵], or just press [↵] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
[PgDn]	Print or display the directory. The directory contains the following information for each tabular data set: the tabular data set number, date and time the file was created or edited last, and the tabular data set ID.
[Esc]	Discard the information you entered and return to the Tabular Data Set Menu.

TERMINAL EMULATION MODE

Terminal emulation is used to establish communications with remote data handling equipment prior to transmitting data by way of the Start Report screen. Transmission parameters must match those of the receiving hardware (refer to screen 10.9 Communications Options).

Data are transmitted by way of an RS-232C communication link. Characters typed or received in terminal mode are displayed near the bottom of the screen (characters typed are displayed only if local echo was enabled on screen 10.9). Linefeeds received and lines greater than 80 columns in length cause an upward scrolling. Fifteen lines are displayed. The Status Display remains visible.

Data may be transmitted while runs are in progress.

6.7 TERMINAL EMULATION

```
PORESIZER 9320 VX.XX                 DIR: DATA1          DATE:07/14/89 TIME:06:20:59
LP:      14.70 psia  IDLE                                HP:      14.7 psia  IDLE
PORT       1         2                                   PORT       1
SAMPLE                                                   SAMPLE
INTVOL     0.000     0.000    mL/g                       INTVOL     0.000    mL/g
% STEM                                                   % STEM

                          6.7 TERMINAL EMULATION







Press [F2] to exit.
```

Press This Key	To
F2	Exit from terminal mode.

PERFORMING LOW PRESSURE RUNS

The Start Low Pressure Run screen contains six pages. The first page prompts you to enter the sample number for each port and to indicate whether reports are to be printed automatically after the run. The remaining pages contain operational instructions.

7p1 START LOW PRESSURE RUN

```
PORESIZER 9320 VX.XX              DIR: DATA1            DATE:07/14/89 TIME:06:22:53
LP:     14.70 psia IDLE                                 HP:      14.7 psia IDLE
PORT        1         2                                 PORT        1
SAMPLE                                                  SAMPLE
INTVOL     0.000     0.000    mL/g                      INTVOL     0.000    mL/g
% STEM                                                  % STEM

                          7p1 START LOW PRESSURE RUN

                   PORT1         PORT2

Sample number:     1             2

Start report after run?  yes

Report destination?  printer



Press [PgDn] to start run, or [Esc] for no action.
```

FIELD	DESCRIPTION
Sample number:	*Range: 1 to 400* Enter a sample number for each port on which a sample is to be run.
Start report after run?	*Toggle* *Choices: yes or no*

FIELD	DESCRIPTION
Report destination?	*Toggle* *Choices: serial line, printer, printer/plotter, drive a:, drive c:* If you choose drive a: or drive c:, the report is written to a file named: (directory name).(sample number) For example, for the directory DATA1, sample number 1, the name would be: **DATA1.001**.

Use the arrow keys to move from field to field. Enter the desired value in each field and press [↵], or just press [↵] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
[PgDn]	Start the low pressure run.
[Esc]	Discard the information you entered and return to the Main Function Menu.

7p2 START LOW PRESSURE RUN

Two penetrometers or one penetrometer and one blank rod must be installed in the low pressure ports (refer to **Installing Penetrometers in Low Pressure Ports** in Chapter 4) before continuing.

```
PORESIZER 9320 VX.XX            DIR: DATA1          DATE:07/14/89 TIME:06:23:37
LP:     14.70 psia MANU PREP                        HP:      14.7 psia IDLE
PORT     1        2                                 PORT     1
SAMPLE   31       32                                SAMPLE
INTVOL                  mL/g                        INTVOL     0.000   mL/g
% STEM                                              % STEM

                         7p2 START LOW PRESSURE RUN

     1)  Install assembled penetrometers in both ports or a penetrometer
         in one port and a dummy rod in the other.

     2)  Press EVACUATE SAMPLE SLOW button.

     3)  Turn on power switch for vacuum pump.


Press [PgDn] to continue run, or [Esc] to cancel run.
```

Follow the instructions displayed on the screen.

Press This Key	To
PgDn	Continue the low pressure run.
Esc	Cancel the low pressure run and return to the Main Function Menu.

7p3 START LOW PRESSURE RUN

```
PORESIZER 9320 VX.XX               DIR: DATA1           DATE:07/14/89 TIME:06:30:06
LP:       258 umHg  MANU PREP                            HP:      14.7 psia IDLE
PORT       1        2                                    PORT      1
SAMPLE    31       32                                    SAMPLE
INTVOL                       mL/g                        INTVOL    0.000    mL/g
% STEM                                                   % STEM

                         7p3 START LOW PRESSURE RUN

    4)  When pressure drops to 1.000 psia (see Manual if running a powder),
        release EVACUATE SAMPLE SLOW button and press EVACUATE SAMPLE MED
        button.
                                 CAUTION
        Beware of sample fluidization in powdered samples. Fine powders
        may require pumping to 0.500 psia before going to MED. Failure to
        do so may result in powder contaminating system.

    5)  When pressure drops to 250 µmHg, release EVACUATE SAMPLE MED button
        and press EVACUATE SAMPLE FAST button.

    6)  Install capacitance detectors over penetrometer assemblies.

Press [PgDn] to continue run, or [Esc] to cancel run.
```

Follow the instructions displayed on the screen.

CAUTION

High surface area samples such as carbon black, fine silica, controlled pore glass, etc. require gentler evacuation. Such materials will easily fluidize and could be drawn into the plumbing and valves. To avoid sample fluidization, the sample must be allowed to attain a lower pressure, about 0.5 psia, under slow evacuation. Press the EVACUATE SAMPLE MED button partially in until you hear the valve open; do not latch the button in. Watch the sample. If it begins to fluidize, release the EVACUATE SAMPLE MED button and allow slow evacuation to continue for a few more minutes. When safe medium evacuation may continue, press the µmHg PRESSURE TRANSDUCER and the EVACUATE SAMPLE MED buttons. Release the EVACUATE SAMPLE SLOW button.

Press This Key	To
PgDn	Continue the low pressure run.
Esc	Cancel the low pressure run and return to the Main Function Menu.

7p4 START LOW PRESSURE RUN

```
PORESIZER 9320 VX.XX          DIR: DATA1          DATE:08/13/89 TIME:17:05:26
LP:        50 umHg  MANU PREP                     HP:      14.7 psia IDLE
PORT       1        2                             PORT      1
SAMPLE     31       32                            SAMPLE
INTVOL              0.000    mL/g                 INTVOL    0.000    mL/g
% STEM                                            % STEM

                      7p4 START LOW PRESSURE RUN

    7)  When pressure drops to 50 µmHg (or has equilibrated at some lower
        pressure), lightly tap the MERCURY FILL button. If the pressure
        increases, allow longer evacuation; otherwise, press the MERCURY
        FILL button. Hold the MERCURY FILL button down until the MERCURY UP
        light comes on. If the MERCURY UP light goes out, press the MERCURY
        FILL button until the light comes on again.

                                  CAUTION
                 Release the button as soon as the light comes on.

    8)  Release the EVACUATE SAMPLE FAST button.

Press [PgDn] to continue run, or [Esc] to cancel run.
```

Follow the instructions displayed on the screen.

Press This Key	To
PgDn	Continue the low pressure run.
Esc	Cancel the low pressure run and return to the Main Function Menu.

7p5 START LOW PRESSURE RUN

```
PORESIZER 9320 VX.XX            DIR: DATA1          DATE:08/13/89 TIME:17:06:31
LP:       1.50 psia MANU PREP                       HP:      14.7 psia IDLE
PORT      1         2                               PORT     1
SAMPLE    31        32                              SAMPLE
INTVOL              0.000   mL/g                    INTVOL       0.000    mL/g
% STEM                                              % STEM

                         7p5 START LOW PRESSURE RUN

      9) Press and hold down the ADJUST LOW PRESSURE button until desired
         filling pressure is reached (usually 0.500 to 1.500 psia )

     10) Press the EVACUATE RESERVOIR button and wait 20 to 30 seconds or
         until the sound from the vacuum pump returns to normal.

         CAUTION: Do not release the EVACUATE RESERVOIR button.

     Take base low pressure and intrusion readings as follows:

     11) Press and hold down the MERCURY DRAIN button until the MERCURY
         DOWN light comes on.

Press [PgDn] to continue run, or [Esc] to cancel run.
```

Follow the instructions displayed on the screen.

CAUTION

Do not release the EVACUATE RESERVOIR button until the MERCURY DOWN indicator comes on. Failure to allow enough time to pull a complete vacuum on the system could cause mercury to back up and the warning buzzer to sound.

Press This Key	To
PgDn	Continue the low pressure run.
Esc	Cancel the low pressure run and return to the Main Function Menu.

7p6 START LOW PRESSURE RUN

```
PORESIZER 9320 VX.XX                    DIR: DATA1             DATE:08/13/89 TIME:03:51:10
LP:       1.58  psia MANU PREP                                 HP:       14.7 psia IDLE
PORT        1          2                                       PORT        1
SAMPLE     31         32                                       SAMPLE
INTVOL                         mL/g                            INTVOL      0.000     mL/g
% STEM                                                         % STEM

                          7p6 START LOW PRESSURE RUN

    12) Release the EVACUATE RESERVOIR button and the MERCURY DRAIN
        button.

        NOTE:  After recording base low pressure and intrusion readings,
               press F9 for Run Control/Status Menu, and then press F3
               for Low Pressure Run Control in order to record low
               pressure data points.

    13) Press [PgDn] to record base low pressure and intrusion readings.


Press [Esc] to cancel run.
```

Follow the instructions displayed on the screen.

Press This Key	To
PgDn	Take readings. The following message is displayed: **RECORDING BASE LOW PRESSURE AND INTRUSION READINGS** The system then returns to the Main Function Menu. **NOTE** **When the system returns to the Main Function Menu press F9, F3 to display the Low Pressure Run Control screen and record low pressure data points.**
Esc	Cancel the low pressure run and display screen 7p7.

7p7 START LOW PRESSURE RUN

```
PORESIZER 9320 VX.XX                DIR: DATA1          DATE:08/13/89 TIME:17:09:24
LP:    14.70 psia TERM GENR                             HP:     14.7 psia IDLE
PORT       1        2                                   PORT         1
SAMPLE     31       32                                  SAMPLE
INTVOL              0.000   mL/g                        INTVOL     0.000   mL/g
% STEM                                                  % STEM

                       7p7 START LOW PRESSURE RUN

                 System is BELOW atmospheric pressure

   1)  Return the system to atmospheric pressure by lightly tapping the
       ADJUST LOW PRESSURE button until a pressure between
       14.2 - 15.2 psia is reached.

   2)  The low pressure run is now complete. Remove and reweigh the
       penetrometer(s)

Press [PgDn] to continue.
```

NOTE

This screen is displayed only if you pressed Esc on the previous page.

Lightly tap the ADJUST LOW PRESSURE button until the specified pressure is reached. Then press PgDn .The system returns to the Main Function Menu.

PERFORMING HIGH PRESSURE RUNS

The Start High Pressure Run screen contains one page for automatic runs and four pages for manual runs.

8p1 START HIGH PRESSURE RUN (Automatic and Manual Runs)

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/14/89 TIME:06:45:49
LP:      14.70 psia IDLE                              HP:      14.7 psia IDLE
PORT        1        2                                PORT        1
SAMPLE     31       32                                SAMPLE
INTVOL     0.000    0.000    mL/g                     INTVOL     0.000     mL/g
% STEM                                                % STEM

                         8p1 START HIGH PRESSURE RUN

Sample number:     1                      Pressure table number:  1

Run type?  automatic                      Run method?  equilibrated

Start report after run?  yes              Equilibration time:      10 seconds

Report destination?  printer              Maximum intrusion volume:     0.000  mL/g

Atmospheric pressure:  14.7000  psia      Last collected point:  14.7000     psia

Sampwt+Pntrwt+Hgwt:     1.0000  g
```

FIELD	DESCRIPTION
Automatic and Manual Runs	
Sample number:	*Range:* *1 to 400* Enter a sample number that exists in the current directory and that has completed a low pressure run.
Run type?	*Toggle* *Choices:* *automatic or manual*
Start report after run?	*Toggle* *Choices:* *yes or no*

FIELD	DESCRIPTION
Report destination?	*Toggle* *Choices: serial line, printer, printer/plotter, drive a:, drive c:* If you choose drive a: or drive c:, the report is written to a file named: (directory name).(sample number) For example, for the directory DATA1, sample number 1, the name would be: **DATA1.001**.
Atmospheric pressure:	*Range: 10.0000 to 20.0000 psia* *0.0689 to 0.1379 MPa*
Sampwt+Pntrwt+Hgwt:	*Range: 1.0000 to 1000.0000 g* If you entered the sample + penetrometer + mercury weight after the low pressure run, the value you entered is displayed. If you did not enter the weight and you wish to measure density, enter it here. The following fields are displayed only if you chose automatic for run type.
<u>**Automatic Runs Only**</u>	
Pressure table number:	*Range: 1 to 50 (must be an existing pressure table number)* **NOTE** **A pressure table MUST be entered for automatic runs.**
Run method?	*Toggle* *Choices: equilibrated or rate sequence*
Equilibration time:	*Range: 0 - 10000 seconds* Displays only when you chose **equilibrated** for run method.
Rate sequence table number:	*Range: 1 to 50 (must be an existing rate sequence table)* Displays only when you chose **rate sequence** for run method.

FIELD	DESCRIPTION
Maximum intrusion volume:	*Range:* *0.000 to 100.000 mL/g* An additional data point will be collected whenever this volume has been intruded since the last collected point.
Last collected point:	The last point collected during the sample's previous run is displayed.

Use the arrow keys to move from field to field. Enter the desired value in each field and press [Enter], or just press [Enter] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
[PgDn]	Start the high pressure run. ▪ If you chose automatic for run type, the following message is displayed: **Close the vent valve.** Close the vent valve on the high pressure chamber. The run begins and the system returns to the Main Function Menu. You may press [F9], [F5] to monitor or control the run. ▪ If you chose manual for run type, page 2 is displayed.
[Esc]	Cancel the run and return to the Main Function Menu.

8p2 START HIGH PRESSURE RUN (MANUAL RUN ONLY)

The penetrometer must be installed in the high pressure chamber (refer to **Installing the Penetrometer in the High Pressure Chamber** in Chapter 4) and the vent valve must be open before continuing.

```
PORESIZER 9320 VX.XX              DIR: DATA1              DATE:07/14/89 TIME:06:49:06
LP:     14.70 psia IDLE                                   HP:      15.8  psia MANU PREP
PORT        1          2                                  PORT       1
SAMPLE                                                    SAMPLE     1
INTVOL    0.000      0.000    mL/g                        INTVOL     0.000    mL/g
% STEM                                                    % STEM     0

                         8p2 START HIGH PRESSURE RUN

          To record the high pressure transducer offset, vent the

              high pressure chamber to atmospheric pressure.



Press [PgDn] to record offset, or [Esc] to cancel run.
```

Vent the high pressure chamber to atmospheric pressure, then press PgDn.

Press This Key	To
PgDn	Record the offset. The following message is displayed. **RECORDING HIGH PRESSURE TRANSDUCER OFFSET** After recording the offset, page 3 is displayed.
Esc	Cancel the run and return to the Main Function Menu.

8p3 START HIGH PRESSURE RUN (MANUAL RUN ONLY)

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/14/89 TIME:06:49:39
LP:    14.70 psia IDLE                                HP:     15.0  psia MANU PREP
PORT       1          2                               PORT      1
SAMPLE                                                SAMPLE    1
INTVOL     0.000      0.000    mL/g                   INTVOL    0.000    mL/g
% STEM                                                % STEM    0

                        8p3 START HIGH PRESSURE RUN

                  Make sure the vent valve is closed before

             starting pressurization of the high pressure system.



Press [PgDn] to continue, or [Esc] to cancel run.
```

Close the vent valve on the high pressure chamber, then press [PgDn].

Press This Key	To
[PgDn]	Continue the high pressure run.
[Esc]	Cancel the run and return to the Main Function Menu.

8p4 START HIGH PRESSURE RUN (MANUAL RUN ONLY)

```
PORESIZER 9320 VX.XX            DIR: DATA1          DATE:07/14/89 TIME:06:50:13
LP:      14.70 psia IDLE                            HP:        25.8 psia MANU PREP
PORT       1         2                              PORT         1
SAMPLE                                              SAMPLE       1
INTVOL     0.000     0.000     mL/g                 INTVOL       0.000    mL/g
% STEM                                              % STEM       0

                         8p4 START HIGH PRESSURE RUN

   The high pressure intrusion base reading will be recorded next. Raise
   the pressure in the high pressure system to    25.386 psia. The accuracy
   of the matching of the low and high pressure intrusion curves depends
   on how close the instrument is to the desired pressure.

       NOTE:  After recording the base intrusion reading,
              press F9 for Run Control/Status Menu, and then press F4
              for Manual High Pressure Run Control in order to record
              high pressure data points.


Press [PgDn] to record base reading, or [Esc] to cancel run.
```

NOTE

In the instruction on the above screen: "Raise the pressure in the high pressure system to 14.700 psia," 14.700 will be replaced with the last point collected for this sample.

Raise the pressure in the high pressure chamber, using the PRESSURE STEP or SCAN switch, to the pressure shown on the screen.

Press This Key	To
PgDn	Record base reading and return to the Main Function Menu. **NOTE** **When the system returns to the Main Function Menu, press F9, F4 to display the High Pressure Run Control screen and record high pressure data points.**
Esc	Cancel the run and return to the Main Function Menu.

CONTROLLING AND MONITORING LOW AND HIGH PRESSURE RUNS

9. RUN CONTROL/STATUS MENU

```
PORESIZER 9320 VX.XX              DIR: DATA1        DATE:07/14/89 TIME:06:51:08
LP:      14.70 psia IDLE                            HP:      14.7 psia MANU COLL
PORT        1         2                             PORT        1
SAMPLE                                              SAMPLE      1
INTVOL     0.000     0.000    mL/g                  INTVOL     0.000    mL/g
% STEM                                              % STEM     0

                          9. RUN CONTROL/STATUS MENU

 F1  - Help                               F2  - Main Function Menu

 F3  - Low Pressure Run Control           F4  - Manual High Pressure Run Control

 F5  - Auto High Pressure Run Control/Status



Press Desired Key
```

FUNCTION	DESCRIPTION
F1 – **Help**	Displays the HELP screens.
F2 – **Main Function Menu**	Displays the Main Function Menu.
F3 – **Low Pressure Run Control**	Enables you to record data points and terminate low pressure runs.
F4 – **Manual High Pressure Run Control**	Enables you to record data points and terminate manual high pressure runs.
F5 – **Auto High Pressure Run Control/Status**	Displays the status of the current high pressure run. Also enables you to skip a run step and suspend, resume, or cancel high pressure runs.

9.3 LOW PRESSURE RUN CONTROL

The Low Pressure Run Control screen can be accessed only when a low pressure run is in progress.

```
PORESIZER 9320 VX.XX            DIR: DATA1          DATE:07/13/89 TIME:09:20:43
LP:      3.34  psia MANU COLL                       HP:     14.7 psia IDLE
PORT     1         2                                PORT     1
SAMPLE   31        32                               SAMPLE
INTVOL   0.003     0.000    mL/g                    INTVOL   0.000    mL/g
% STEM   0                                          % STEM

                     9.3 LOW PRESSURE RUN CONTROL

                Low Pressure Sample Directory: DATA1
Point number  Pressure( psia )  Left Intrusion( mL/g )  Right Intrusion( mL/g )
      1)          1.50               0.000                     0.000
                  2.17               0.001                     0.000
                  3.34               0.003                     0.000

    F2 - Run Control/Status Menu     F3 - Record Data     F4 - Finish Run
```

Observe the low pressure shown in the status display portion of the screen. Record data points as follows:

1. Press the ADJUST LOW PRESSURE button on the front panel of the PoreSizer while observing the pressure reading in the status display.

2. Release the ADJUST LOW PRESSURE button when you wish to record a data point. Then press F3 to record the point.

3. Repeat Steps 1 and 2 for each pressure point you wish to record.

4. Press F4 when you have finished recording data points.

FIELD	DESCRIPTION
Low Pressure Sample Directory:	The sample directory containing the current samples is displayed.
Point number	The system assigns the point number.
Pressure (psia or MPa)	Data points are displayed as they are recorded. **NOTE** **The total number of data points collected for both low and high pressure runs cannot exceed 250. Therefore, the number of points collected here will reduce the maximum number of high pressure points that can be collected.** You may record up to 250 data points for the low and high pressure runs.
Left Intrusion (mL/g)	The intrusion volume of the sample in the left port is displayed.
Right Intrusion (mL/g)	The intrusion volume of the sample in the right port is displayed.

Use the arrow keys to move from field to field. Enter the desired value in each field and press [Enter], or just press [Enter] to accept the default value. When you have finished entering information, choose one of the following options:

Press This Key	To
[F2]	Suspend the run and return to the Run Control/Status Menu.
[F3]	Record a data point.
[F4]	End the low pressure run. When you press [F4], the following message is displayed: **Press [PgDn] to finish run or [Esc] for no action.** Choose one of the following: ▪ Press [PgDn] if you wish to end the run. Screen 9.3.4 will be displayed. ▪ Press [Esc] to continue the run.

Press This Key	To
F4	If the last data point collected in the low pressure run is below atmospheric pressure and the maximum head pressure, the following message is displayed: **Caution: High pressure run may not be able to match last recorded pressure.** Choose one of the following: ▪ Press Esc to continue collecting data. ▪ Press PgDn to end the run. If you end the run, intrusion data will be excluded from the analysis results.

9.3.4 LOW PRESSURE RUN CONTROL

One of two screens will be displayed depending on whether the system is above or below atmospheric pressure.

<u>Above Atmospheric Pressure</u>

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/14/89 TIME:07:05:09
LP:    14.70 psia TERM EVAC                            HP:     14.7 psia IDLE
PORT        1        2                                 PORT      1
SAMPLE      2                                          SAMPLE
INTVOL      0.887    0.881    mL/g                     INTVOL    0.000    mL/g
% STEM      0                                          % STEM

                        9.3.4 LOW PRESSURE RUN CONTROL

                    System is ABOVE atmospheric pressure

    1)  Return the system to atmospheric pressure by lightly tapping the
        EVACUATE SAMPLE SLOW button until a pressure between
        14.2 - 15.2 psia is reached.

    2)  The low pressure run is now complete. Remove and reweigh the
        penetrometer(s)


Press [PgDn] to continue.
```

Lightly tap the EVACUATE SAMPLE SLOW button until a pressure between atmospheric pressure ± 0.5 psia is displayed on the screen. Then remove and reweigh the penetrometers.

Press This Key	To
PgDn	Return to the Run Control/Status Menu.

Below Atmospheric Pressure

```
PORESIZER 9320 VX.XX            DIR: DATA1          DATE:07/17/89 TIME:08:26:57
LP:     14.70 psia TERM GENR                        HP:      14.7 psia IDLE
PORT        1         2                             PORT        1
SAMPLE      5                                       SAMPLE
INTVOL    0.000     0.000    mL/g                   INTVOL    0.000    mL/g
% STEM      0                                       % STEM

                      9.3.4 LOW PRESSURE RUN CONTROL

                  System is BELOW atmospheric pressure

    1)  Return the system to atmospheric pressure by lightly tapping the
        ADJUST LOW PRESSURE button until a pressure between
        14.2 - 15.2 psia is reached.

    2)  The low pressure run is now complete. Remove and reweigh the
        penetrometer(s)

Press [PgDn] to continue.
```

Lightly tap the ADJUST LOW PRESSURE button until a pressure between atmospheric pressure ± 0.5 psia is displayed on the screen. Then remove and reweigh the penetrometers.

Press This Key	To
PgDn	Return to the Run Control/Status Menu.

9.4 MANUAL HIGH PRESSURE RUN CONTROL

The Manual High Pressure Run Control screen can be accessed only when a manual high pressure run is in progress.

```
PORESIZER 9320 VX.XX              DIR: DATA1              DATE:07/13/89 TIME:09:55:17
LP:     14.70 psia IDLE                                    HP:      181.8 psia MANU COLL
PORT       1          2                                    PORT       1
SAMPLE                                                     SAMPLE     2
INTVOL     0.000      0.000    mL/g                        INTVOL     0.885      mL/g
% STEM                                                     % STEM     0

                       9.4 MANUAL HIGH PRESSURE RUN CONTROL

                      High Pressure Sample Directory: DATA1
       Point number          Pressure( psia )          Intrusion Volume( mL/g )
         MATCH                     30.3                          0.000
          (21)                    184.8                          0.885


   F2 - Run Control/Status Menu      F3 - Record Data      F4 - Finish Run
```

Observe the high pressure shown in the status display portion of the screen. Record data points as follows:

1. Press the PRESSURE STEP or SCAN switch on the front panel of the PoreSizer while observing the pressure reading in the status display.
2. Release the PRESSURE STEP or SCAN switch when you wish to record a data point. Then press F3 to record the point.
3. Repeat Steps 1 and 2 for each pressure point you wish to record.
4. Press F4 when you have finished recording data points.

FIELD	DESCRIPTION
High Pressure Sample Directory	The sample directory containing the current sample is displayed.
Point number	Displays the number of points taken for this sample during this and any previous runs.
Pressure (psia or MPa)	Data points are displayed as they are recorded. You may record up to 250 data points (low pressure and high pressure combined).
Intrusion Volume (mL/g)	The intrusion volume of the sample is displayed.

Press This Key	To
F2	Suspend the run and return to the Run Control/Status Menu. **CAUTION** **Return the SCAN switch to the center (OFF) position before suspending a run. Even though the run will be suspended, pressure will continue to increase.**
F3	Record a data point.
F4	End the high pressure run. When you press F4, the following message is displayed: **Press [PgDn] to finish run or [Esc] for no action.** Choose one of the following: ▪ Press PgDn if you wish to end the run. Screen 9.4.4 will be displayed. ▪ Press Esc to continue the run.

9.4.4 MANUAL HIGH PRESSURE RUN CONTROL

```
PORESIZER 9320 VX.XX          DIR: DATA1          DATE:07/14/89 TIME:07:07:20
LP:    14.70 psia IDLE                            HP:      25.0 psia TERM DEPR
PORT       1         2                            PORT       1
SAMPLE                                            SAMPLE     2
INTVOL     0.000     0.000   mL/g                 INTVOL    -0.002     mL/g
% STEM                                            % STEM     0

              9.4.4 MANUAL HIGH PRESSURE RUN CONTROL

     Return the high pressure system to the INTENSIFIER LOW limit by
     pressing the SCAN DOWN switch until the INTENSIFIER LOW light
     comes on.

     The high pressure run is now complete.

Press [PgDn] to continue.
```

Press the SCAN switch down until the INTENSIFIER LOW indicator comes on.

Press This Key	To
PgDn	Return to the Run Control/Status Menu.

9.5 AUTO HIGH PRESSURE RUN CONTROL/STATUS

```
PORESIZER 9320 VX.XX              DIR: DATA1         DATE:07/14/89 TIME:07:11:03
LP:    14.70 psia IDLE                               HP:       230.3 psia INTR GENR
PORT        1        2                               PORT        1
SAMPLE                                               SAMPLE      3
INTVOL    0.000    0.000   mL/g                      INTVOL    0.032       mL/g
% STEM                                               % STEM      2

                 9.5 AUTO HIGH PRESSURE RUN CONTROL/STATUS

                 High Pressure Sample Directory: DATA1

High Pressure Points Taken:    2         High Pressure Points Asked:  10


  F2  - Run/Control Status Menu          F3  - Skip High Pressure Run Step

  F4  - Suspend High Pressure Run        F5  - Cancel High Pressure Run
```

FIELD	DESCRIPTION
High Pressure Sample Directory	The sample directory containing the current sample is displayed.
High Pressure Points Taken:	The number of pressure points taken so far is displayed.
High Pressure Points Asked:	The number of pressure points specified in the pressure table for this run is displayed.
HP: (status message)	A one-line message denoting the current status of the high pressure run is displayed.

NOTE

If you end a high pressure run before the run is complete, use either the SCAN or STEP button to return the system to atmospheric pressure before removing the penetrometer from the high pressure chamber.

Press This Key	To
[F2]	Return to the Run Control/Status Menu.
[F3]	Skip a step. **NOTE** **For a rate sequence run, this key is active during the termination phase only.** The following message is displayed: **Press [PgDn] to skip a step or [Esc] for no action.** Choose one of the following: ▪ Press [PgDn] if you wish to skip a step. ▪ Press [Esc] to continue the run. If you press [F3] while a point is being equilibrated, the point will be recorded but will not be equilibrated for the remaining time.
[F4]	Suspend or resume the high pressure run. The following message is displayed: **Press [PgDn] to suspend (resume) run or [Esc] for no action.** Choose one of the following: ▪ Press [PgDn] if you wish to suspend or resume a run. ▪ Press [Esc] to continue the run.
[F5]	End the high pressure run. When you press [F5], the following message is displayed: **Press [PgDn] to cancel run or [Esc]** for no action. Choose one of the following: ▪ Press [PgDn] if you wish to end the run. ▪ Press [Esc] to continue the run.

CHAPTER 7

USING THE ANALYSIS PROGRAM UTILITIES

- Maintaining System Files
- Entering and Maintaining Sample Directories
- Entering and Maintaining System and Communications Parameters
- Exiting the Analysis Program

USING THE ANALYSIS PROGRAM UTILITIES

MAINTAINING SYSTEM FILES

This chapter describes the screens used to enter and maintain system operating parameters and sample directories, and to copy, back up and restore system data. The screens are organized by screen number.

10. UTILITIES MENU

The Utilities Menu contains functions that enable you to enter and maintain system operating parameters and sample directories, and copy and back up system data.

```
PORESIZER 9320 VX.XX            DIR: DATA1          DATE:07/14/89 TIME:07:12:10
LP:     14.70 psia IDLE                             HP:     14.7 psia INTR GENR
PORT      1        2                                PORT      1
SAMPLE                                              SAMPLE    3
INTVOL    0.000    0.000    mL/g                    INTVOL    0.000    mL/g
% STEM                                              % STEM    0

                        10. UTILITIES MENU

  F1  - Help                                F2  - Main Function Menu

  F3  - Format Disk                         F4  - Copy Sample Information File

  F5  - Back Up Utility                     F6  - Restore Utility

  F7  - Sample Directory Menu               F8  - System Options

  F9  - Communications Options              F10 - Exit to DOS
Press Desired Key
```

FUNCTION	DESCRIPTION
F1 – Help	Displays the HELP screens.
F2 – Main Function Menu	Displays the Main Function Menu.
F3 – Format Disk	Formats diskettes.

FUNCTION	DESCRIPTION
F4 - **Copy Sample Information File**	Copies a sample information file.
F5 - **Back up Utility**	Enables you to back up parameter sets or sample directories to diskette.
F6 - **Restore Utility**	Enables you to restore files that were backed up to diskette.
F7 - **Sample Directory Menu**	Displays screens that enable you to add, update, select and list sample directories.
F8 - **System Options**	Enables you to specify system operational parameters.
F9 - **Communications Options**	Enables you to enter and maintain communications parameters.
F10 - **Exit to DOS**	Enables you to exit the Analysis Program and return to DOS.

10.3 FORMAT DISK

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/14/89 TIME:07:18:24
LP:    14.70 psia IDLE                                HP:     14.7 psia IDLE
PORT      1        2                                  PORT       1
SAMPLE                                                SAMPLE
INTVOL    0.000    0.000   mL/g                       INTVOL     0.000    mL/g
% STEM                                                % STEM

                             10.3 FORMAT DISK

Make sure Drive A is open.



Press [PgDn] to begin format, or [Esc] for no action.
```

CAUTION

Formatting erases all the files on the diskette. Use a new diskette to reduce the possibility of losing data, but a previously-used diskette may be reformatted if the data on the used diskette is no longer needed.

Press This Key	To
PgDn	Format the diskette. Make sure drive A is open, then press PgDn. The system exits the Analysis Program and displays instructions for formatting the diskette. Follow the instructions displayed on the screen. Make sure you do not insert the diskette until instructed to do so. When formatting is complete, the system returns to the Format Disk screen. Press F2 to return to the Utilities Menu.
Esc	Return to the Utilities Menu without formatting the diskette.

10.4 COPY SAMPLE INFORMATION FILE

The COPY utility is used to copy a sample information file. You may specify the directory name and file name to which the file will be copied. The BACKUP utility (described later in this chapter) is used to copy **all** the files in a specified directory from the hard disk to diskette.

```
PORESIZER 9320 VX.XX              DIR: DATA1            DATE:07/14/89 TIME:07:19:12
LP:     14.70 psia IDLE                                 HP:      14.7 psia IDLE
PORT        1          2                                PORT        1
SAMPLE                                                  SAMPLE
INTVOL     0.000      0.000    mL/g                     INTVOL     0.000     mL/g
% STEM                                                  % STEM

                     10.4 COPY SAMPLE INFORMATION FILE

Copy file from?  current sample directory

Copy file to?  sample directory

Target directory name:  DATA1

Source sample file number:     1

Target sample file number:     4



Press [PgDn] to copy file, or [Esc] for no action.
```

NOTE

If copying to a diskette, the diskette should already be formatted.

FIELD	DESCRIPTION
Copy file from?	*Toggle* *Choices:* *current sample directory or drive A*
Copy file to?	*Toggle* *Choices:* *sample directory or drive A*

FIELD	DESCRIPTION
Target directory name:	*Alphanumeric* *Length: 8* Enter the name of the directory to which the file will be copied. The directory must be an existing sample directory. Displayed only if you selected copy file to sample directory.
Source sample file number:	*Range: 1 to 400* Enter the sample file number of the file to be copied.
Target sample file number:	*Range: 1 to 400* The default is the next available sample file number. Enter the sample file number of the file that will receive the data.

Press This Key	To
PgDn	Copy the sample information file. When the copy process is complete, press F2 to return to the Utilities Menu.
Esc	Return to the Utilities Menu without copying the sample information file.

10.5 BACKUP UTILITY

The **Backup** utility function is no longer provided within this application. Different versions of DOS include various backup utilities that operate differently.

To perform this function, exit to DOS and use the backup utility included with your version of DOS, or use a third party backup utility.

10.6 RESTORE UTILITY

The **Restore** utility function is no longer provided within this application. Different versions of DOS include various backup and restore utilities that operate differently.

To perform this function, exit to DOS and use the backup or restore utility included with your version of DOS, or use a third party backup/restore utility.

ENTERING AND MAINTAINING SAMPLE DIRECTORIES

Up to 200 sample directories can be created. The Sample Directory Menu enables you to add, select, update, and list sample directories.

10.7 SAMPLE DIRECTORY MENU

```
PORESIZER 9320 VX.XX          DIR: DATA1          DATE:07/14/89 TIME:07:25:19
LP:    14.70 psia IDLE                            HP:     14.7 psia IDLE
PORT        1         2                           PORT        1
SAMPLE                                            SAMPLE
INTVOL    0.000     0.000    mL/g                 INTVOL    0.000    mL/g
% STEM                                            % STEM

                        10.7 SAMPLE DIRECTORY MENU

  F1  - Help                             F2  - Utilities Menu

  F3  - Add Sample Directory             F4  - Select Sample Directory

  F5  - Update Sample Directory          F6  - List Sample Directories



Press Desired Key
```

FUNCTION	DESCRIPTION
F1 – Help	Displays the HELP screens.
F2 – Utilities Menu	Displays the Utilities Menu.
F3 – Add Sample Directory	Enables you to add a new sample directory.
F4 – Select Sample Directory	Enables you to select a directory to be used for processing.
F5 – Update Sample Directory	Reconstructs the list of sample files in a sample directory.
F6 – List Sample Directories	Displays a list of sample directories.

10.7.3 ADD SAMPLE DIRECTORY

```
PORESIZER 9320 VX.XX          DIR: DATA1          DATE:07/14/89 TIME:08:35:00
LP:    14.70 psia IDLE                            HP:      14.7 psia IDLE
PORT        1         2                           PORT         1
SAMPLE                                            SAMPLE
INTVOL      0.000     0.000    mL/g               INTVOL     0.000    mL/g
% STEM                                            % STEM

                    10.7.3 ADD SAMPLE DIRECTORY

Sample directory name:  DATA2




Press [PgDn] to add directory, or [Esc] for no action.
```

FIELD	DESCRIPTION
Sample directory name:	*Alphanumeric* *Length:* 8

Press This Key	To
PgDn	Create a new sample directory and return to the Sample Directory Menu.
Esc	Return to the Sample Directory Menu without creating a new directory.

10.7.4 SELECT SAMPLE DIRECTORY

The screen displayed is the same as the screen displayed for Add Sample Directory. Refer to **10.7.3 Add Sample Directory** for an illustration of the screen and description of the fields. To select the directory, enter the directory name and press PgDn. All subsequent references to a sample number refer to sample files stored in this directory. The directory name will appear on the status display.

10.7.5 UPDATE SAMPLE DIRECTORY

The screen displayed is the same as the screen displayed for Add Sample Directory. Refer to **10.7.3 Add Sample Directory** for an illustration of the screen and description of the fields.

This function reconstructs the list of sample files currently contained in the directory. This is necessary if sample files have been copied or deleted under DOS. It is not required after any file handling functions accessed from a PoreSizer 9320 menu.

NOTE

The update may take several minutes to complete. While the update is in progress, the following message is displayed:

Please wait...updating sample directory.

10.7.6 LIST SAMPLE DIRECTORIES

```
PORESIZER 9320 VX.XX              DIR: DATA1          DATE:07/14/89 TIME:07:27:19
LP:     14.70 psia IDLE                               HP:       14.7 psia IDLE
PORT        1         2                               PORT        1
SAMPLE                                                SAMPLE
INTVOL    0.000     0.000    mL/g                     INTVOL     0.000     mL/g
% STEM                                                % STEM

                          10.7.6 LIST SAMPLE DIRECTORIES

List directories on?  screen




Press [PgDn] to produce list, or [Esc] for no action.
```

FIELD	DESCRIPTION
List directories on?	*Toggle* *Choices:* *screen or printer*

Press This Key	To
PgDn	Print or display a list of sample directories. Press any key to return to the Sample Directory Menu.
Esc	Discard the information you entered and return to the Sample Directory Menu. **NOTE** **If you press** Esc, any reports being transmitted to the printer or any other device will be canceled.

ENTERING AND MAINTAINING SYSTEM AND COMMUNICATIONS PARAMETERS

10.8 SYSTEM OPTIONS

```
PORESIZER 9320 VX.XX              DIR: DATA1           DATE:07/14/89 TIME:07:29:03
LP:     14.70 psia IDLE                                HP:     14.7 psia IDLE
PORT      1        2                                   PORT      1
SAMPLE                                                 SAMPLE
INTVOL    0.000    0.000   mL/g                        INTVOL    0.000    mL/g
% STEM                                                 % STEM

                              10.8 SYSTEM OPTIONS

  Pressure units?  psia                      Date:   7 / 14 / 89

  Size type?  diameter                       Time:   7 : 27 : 55

  Size units?  micrometers                   Atmospheric pressure:  14.700   psia

  Type of plotter?  manual feed              Show grid?  no

  Type of printer?  IBM graphics
```

FIELD	DESCRIPTION
Pressure units?	*Toggle* *Choices: psia or MPa*
Date:	*Format: month/day/year*
Size type?	*Toggle* *Choices: diameter or radius*
Time:	*Format: hour/minutes/seconds (24-hour format)*
Size units?	*Toggle* *Choices: micrometers or angstroms*
Atmospheric pressure:	*Range: 10 - 20 psia* *0.069 to 0.138 MPa*

FIELD	DESCRIPTION
Type of plotter?	*Toggle* *Choices:* *automatic feed or manual feed*
Show grid?	*Toggle* *Choices:* *yes or no*
Type of printer?	*Toggle* *Choices:* *IBM graphics or Epson LQ*

Press This Key	To
F2	Save the information you entered and return to the Utilities Menu.
Esc	Discard the information you entered and return to the Utilities Menu.

10.9 COMMUNICATIONS OPTIONS

These options determine the transmission characteristics for the serial line used in Terminal Emulation and for report transmission to the serial line. When this line is to be attached to another computer or data recording device, these options should be set to match the requirements of the other system.

```
PORESIZER 9320 VX.XX             DIR: DATA1          DATE:07/14/89 TIME:07:29:46
LP:     14.70 psia  IDLE                             HP:       14.7 psia  IDLE
PORT        1         2                              PORT        1
SAMPLE                                               SAMPLE
INTVOL      0.000     0.000    mL/g                  INTVOL      0.000    mL/g
% STEM                                               % STEM

                        10.9 COMMUNICATIONS OPTIONS

 Transmission mode?  burst                   Baud rate?  9600

 Local echo?  enabled                        Number of data bits?  8

 End of transmission character:  26          Number of stop bits?  1

 Handshake character:   6                    Parity?  none

 X-on, X-off?  enabled

 Send linefeeds?  no
```

FIELD	DESCRIPTION
Transmission mode?	*Toggle* *Choices: burst or handshake* Burst mode means transmit report data all in one burst, stopping only when XOFF is received (if enabled) or based on hardwire protocol. Handshake mode means stop transmission after each carriage return and wait to receive a handshake character in response before transmitting more data. The ASCII code for the handshake character is specified below.
Baud rate?	*Toggle* *Choices: 9600, 110, 150, 300, 600, 1200, 2400, 4800*

FIELD	DESCRIPTION
Local echo?	*Toggle* *Choices:* *enabled or disabled*
Number of data bits?	*Toggle* *Choices:* *8 or 7*
End of transmission character:	*Range:* *0 to 255* The specified ASCII character code will be transmitted at the end of each report to indicate end of transmission.
Number of stop bits?	*Toggle* *Choices:* *1 or 2*
Handshake character:	*Range:* *0 to 255* This ASCII code is used only in handshake transmission mode (see above).
Parity?	*Toggle* *Choices:* *none, odd, even*
X-on, X-off?	*Toggle* *Choices:* *enabled or disabled*
Send linefeeds?	*Toggle* *Choices* *yes or no*

Press This Key	To
F2	Save the information you entered and return to the Utilities Menu.
Esc	Discard the information you entered and return to the Utilities Menu.

EXITING THE ANALYSIS PROGRAM

10.10 EXIT TO DOS

```
PORESIZER 9320 VX.XX              DIR: DATA1           DATE:07/14/89 TIME:07:30:26
LP:     14.70 psia IDLE                                HP:       14.7 psia IDLE
PORT      1        2                                   PORT        1
SAMPLE                                                 SAMPLE
INTVOL    0.000    0.000    mL/g                       INTVOL    0.000    mL/g
% STEM                                                 % STEM

                               10.10 EXIT TO DOS

Do you want to automatically return to this program after exiting DOS?  yes



Make sure Drive A is open.

Press [PgDn] to exit to DOS, or [Esc] for no action.
```

The following prompt is displayed:

Do you want to automatically return to this program after exiting DOS?

Answer yes or no to this prompt. Make sure that Drive A is open, then press PgDn to exit to DOS or Esc for no action.

NOTE

You cannot exit to DOS while an automatic operation or reports are in progress without first canceling the operation or reports.

CHAPTER 8

TROUBLESHOOTING AND MAINTENANCE

- Troubleshooting
- Preventive Maintenance

TROUBLESHOOTING AND MAINTENANCE

The PoreSizer 9320 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 video monitor screen and their solutions are provided in Table 8-1. Operational problems that are indicated on the video monitor screen and their solutions are provided in **Chapter 10, Error Messages.**

Table 8-1. Common Operational Problems

What Happened	Why	What To Do
Difficulty attaining adequate vacuum conditions during low pressure analyses.	1. Vacuum pump power is off.	1. Turn on power to the vacuum pump.
	2. Vacuum hose not properly connected.	2. Be sure connections both to the vacuum pump and the external trap are lubricated with a good grade of vacuum grease and that external clamps are tight. Check the fitting between the trap and the PoreSizer.
	3. The vacuum hose is contaminated or has non-evident cracks.	3. Check the vacuum hose, replace if necessary.

Table 8-1. Common Operational Problems (continued)

What Happened	Why	What To Do
Difficulty attaining adequate vacuum conditions during low pressure analyses. (continued)	4. Proper sealing not achieved on penetrometers.	4. 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 of 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.
	5. Scratches or other imperfections in either the lip of the penetrometer bulb or the penetrometer cap.	5. Polish the lip of the bulb and the cap using 600-grit emory 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.
	6. Leaky valves.	6. Check the valves for leaks. Refer to **Checking the Valves for Leaks** later in this chapter.
	7. Moisture has accumulated in the system.	7. Remove the accumulated moisture (refer to **Removing Moisture from the System** later in this chapter).
Mercury warning buzzer sounds.	Mercury has been improperly drawn into the trap.	a. Press and latch the EVACUATE RESERVOIR button.
		b. Use the ADJUST LOW PRESSURE button to raise the pressure to (or slightly above) atmospheric pressure.
		c. Press and hold down the MERCURY DRAIN button until the MERCURY DOWN indicator comes on.

Table 8-1. Common Operational Problems (continued)

What Happened	Why	What To Do
Mercury warning buzzer sounds. (continued)	Mercury has been improperly drawn into the trap. (continued)	d. Release the EVACUATE RESERVOIR and MERCURY DRAIN buttons.
		e. Open a low pressure port to allow air to enter the system while draining the mercury.
		f. Open the TRAP DRAIN valve on the rear panel and drain the mercury into an appropriate container.
The RESERVOIR LEVEL LOW indicator does not turn off after mercury has been added to the reservoir.	Excessively contaminated residual mercury, possibly from sample carryover or inevitable oxidation products, has accumulated in the reservoir.	Draw out the mercury from the reservoir and add fresh mercury. (Refer to **Replacing the Mercury in the Reservoir** later in this chapter.)
High pressure system failed to retain a reasonably constant pressure.	1. Leakage around the high pressure chamber cap.	1. Replace the O-rings and backup rings on the high pressure chamber cap. Refer to **Replacing O-Rings and Backup Ring in High Pressure Chamber Cap** later in this chapter.
	2. Rupture disk damaged due to excessive pressure. This occurs only when the pressure in the high pressure chamber exceeds 32,500 psi. When this occurs, the unit will be inoperable and you will notice oil being released from the HYDRAULIC PRESSURE RELIEF valve on the rear panel of the PoreSizer.	2. Replace the rupture disk as follows: a. Remove the red cover from the HYDRAULIC PRESSURE RELIEF valve on the rear panel of the PoreSizer. b. Remove the retaining nut. c. Remove the damaged disk and insert a new disk. d. Replace the retaining nut. e. Replace the cover.

Table 8-1. Common Operational Problems (continued)

What Happened	Why	What To Do
Indicator lights on the front panel do not come on.	Fuse is blown.	Replace the fuse (refer to **Selecting the Power Input** in Chapter 2 for instructions on installing fuses).

PREVENTIVE MAINTENANCE

Table 8-2 lists the maintenance procedures which should be performed to keep the PoreSizer 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 container	Check daily, service as needed
Maintaining high pressure fluid level	Check before performing high pressure run, service as needed
Maintaining vacuum pump fluid level	Check monthly, service as needed
Greasing low pressure ports	Check monthly, service as needed
Replacing O-rings and backup ring in high pressure closure component	Check monthly, service as needed
Cleaning the diskette drive	Service monthly
Replacing the mercury in the reservoir	Replace every three months
Cleaning the vacuum trap	Clean every six months
Changing vacuum pump fluid	As needed
Checking the valves for leaks	As needed
Cleaning valves	As needed
Removing moisture from the system	As needed
Replacing the banana plug	Check every six months, 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. The **Reservoir Level Low** indicator on the upper front panel comes on when the level of mercury drops below the minimum level. The volume of mercury remaining may not be sufficient to fill the penetrometers. To avoid delays, topping-out the reservoir at the beginning of each day is recommended.

1. Lift out the mercury reservoir plug.

2. Observe the level of mercury. It must not reach the lower opening of the neck of the reservoir filling hole. Fill the reservoir to within 0.5 to 1.0 inch of that level.

CAUTION

Do not overfill. Doing so may damage the vacuum system.

3. Replace the plug by pressing it down firmly.

DRAINING SPILLED MERCURY CONTAINER

A container for collecting mercury swept into the drain hole is located just behind the mercury reservoir. A drain fitting, labeled WASTE DRAIN, is located on the rear panel of the PoreSizer. At the beginning of each day, open the drain valve and allow any accumulated mercury to drain into a proper receptacle.

NOTE

Approximately one-eighth inch (0.3 cm) of oil should remain in the container to inhibit the escape of mercury vapors.

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 5-cc penetrometer is installed. Add high pressure fluid as needed.

MAINTAINING VACUUM PUMP FLUID LEVEL

A vacuum pump is sold as an accessory for the PoreSizer. The oil level in the pump should be checked monthly as follows:

1. After the pump has been running at least five minutes, observe the oil level indicator on the pump. The oil level should remain between the two indicator lines.

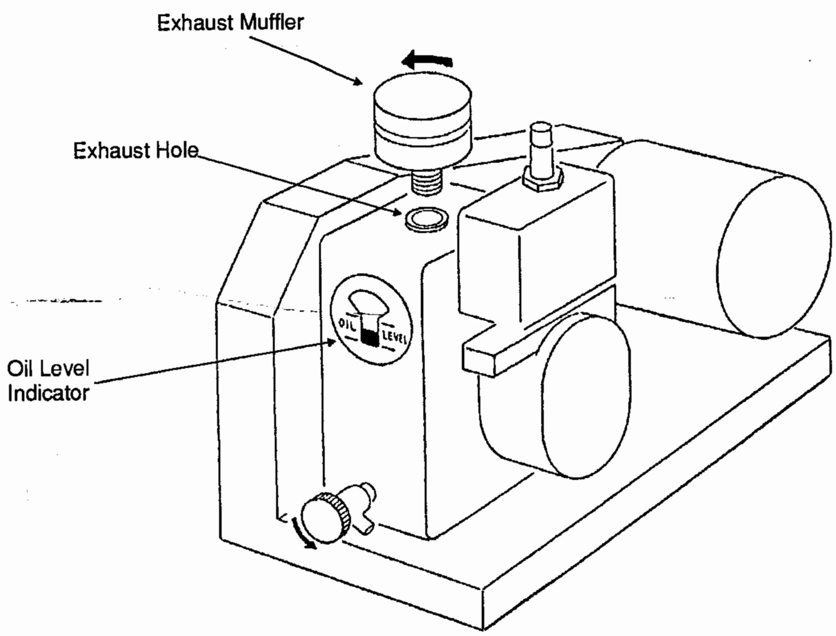


Figure 8-1. Oil Level Indicator

2. If oil is needed, remove the exhaust muffler and add oil through the exhaust hole until the proper level is reached. Replace the exhaust muffler.

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. Do not remove the knobs; internal components may be disoriented.

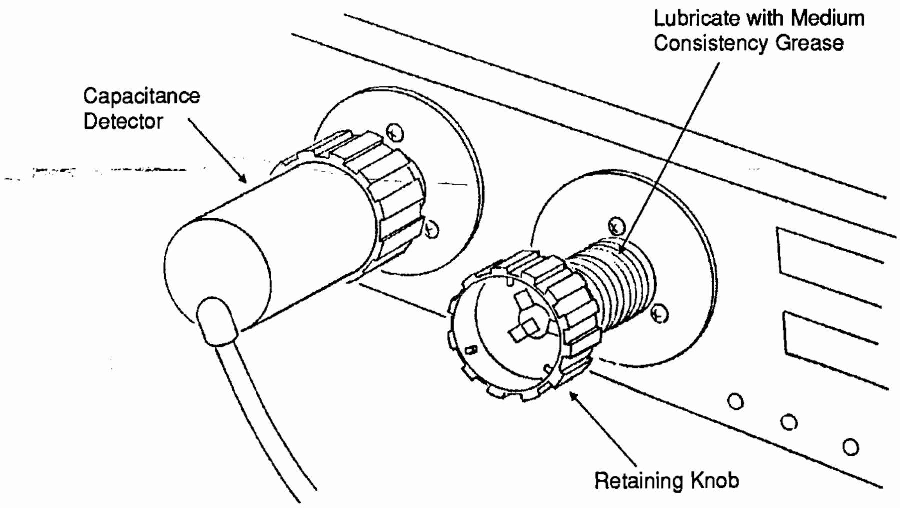


Figure 8-2. Greasing the Low Pressure Ports

3. Lubricate 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 O-RINGS AND BACKUP RING IN HIGH PRESSURE CHAMBER CAP

The chamber cap is sealed into the high pressure chamber by means of an O-ring 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 small slivers coming from the O-ring or pinched areas on the O-ring. 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 O-rings and backup ring. The rings are sufficiently pliable for stretching on and off. Pushing a sharp pointed object beside the rings may help in removal.

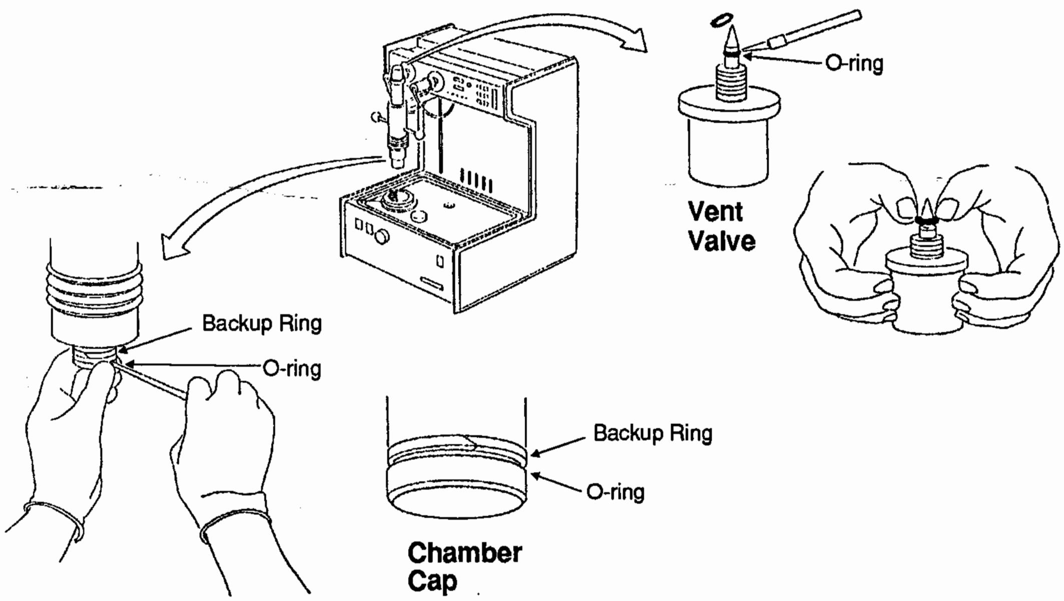


Figure 8-3. Replacing O-Rings and Backup Ring

2. Replace the rings on the lower portion of the chamber cap with the backup ring uppermost and with the slight cup of the backup ring pointing downward.

3. Replace the O-ring in the vent valve as shown in Figure 8-3.

CLEANING THE DISKETTE DRIVE

The diskette drive (Drive A) should be cleaned every month to avoid read/write errors. A diskette drive head cleaning kit, which contains two cleaning diskettes and cleaning solution, is supplied with the control module. Follow the instructions provided with the kit.

REPLACING THE MERCURY IN THE RESERVOIR

Mercury exposed to base elements and then the atmosphere gradually collects oxidation products. These rise to the surface in the mercury reservoir and are not ordinarily a part of the mercury that contacts sample because this mercury is drawn from the bottom. Nevertheless, such products accumulate, some of them stick to the reservoir walls and some build up on contacts in the reservoir. This part of the system should be cleaned to ensure reliable operation after a period of storage and non-use, and it should be cleaned and filled with fresh mercury at about 3-month intervals to prevent the creation of contaminated mercury.

1. Remove the mercury reservoir cap.

2. Raise the high pressure chamber cap by turning the lever arms counterclockwise.

3. Remove the tray.

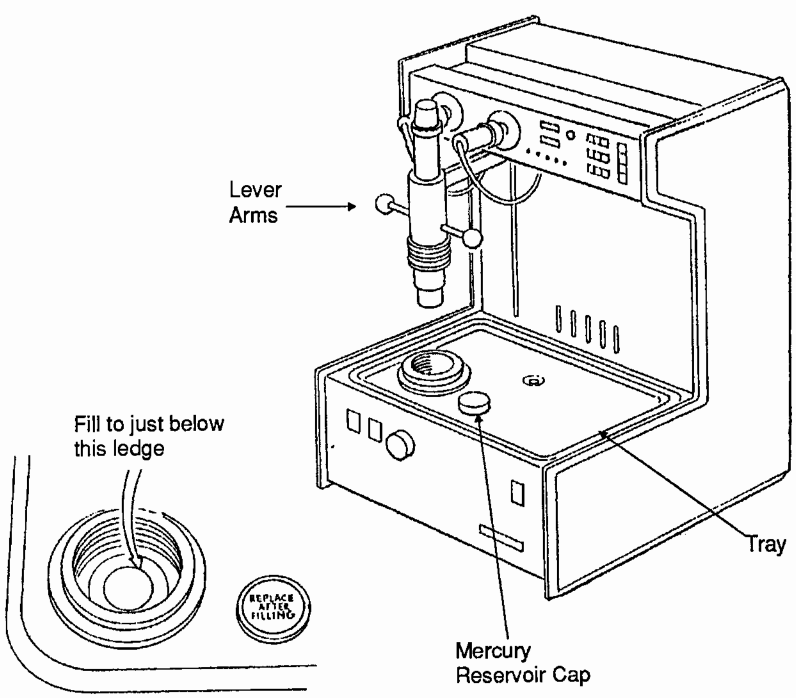


Figure 8-4. Replacing the Mercury in the Reservoir

4. Withdraw mercury from the reservoir using a syringe.

5. Clean the inside of the reservoir using IPA.

6. Replace the tray.

7. When dry, add fresh mercury. The mercury must not reach the lower opening of the neck of the reservoir filling hole. Fill the reservoir to within 0.5 to 1.0 inch of that level.

CAUTION

Do not overfill. Doing so may damage the vacuum system.

8. Replace the mercury reservoir cap by pressing it down firmly.

CLEANING THE VACUUM TRAP

The vacuum trap installed externally between the PoreSizer and the vacuum pump should be drained and cleaned at about 6-month intervals as follows.

1. Detach the trap from both the instrument and the pump.

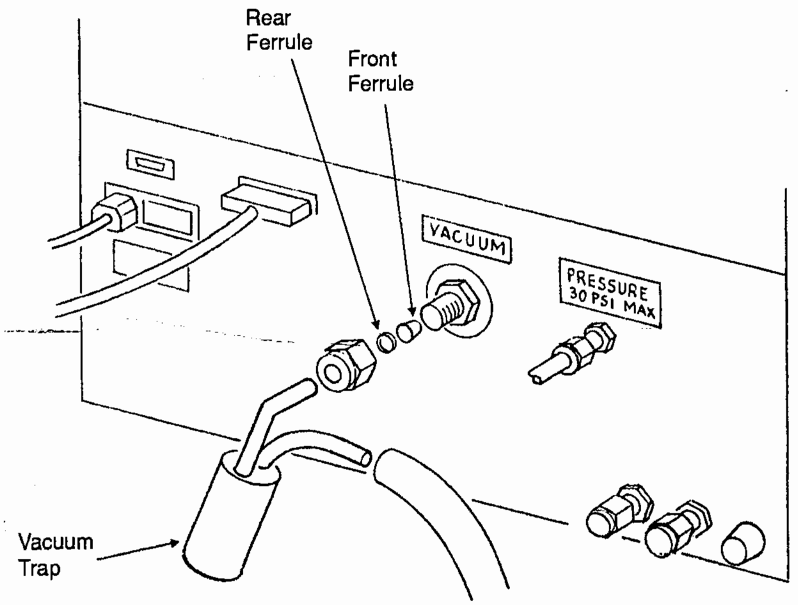


Figure 8-5. Cleaning the Vacuum Trap

2. Invert the trap to permit any accumulated matter to drain from the shorter arm.
3. Wash the trap with isopropyl alcohol and dry by passing air through it before reinstalling.
4. When dry, attach the trap to the instrument and the pump.

NOTE

The front ferrule (refer to Figure 8-5) should be replaced each time the trap is disconnected from the PoreSizer.

CHANGING VACUUM PUMP FLUID

The most common cause of loss in efficiency in a vacuum pump is contamination of the fluid. Contamination is caused by condensation of vapors and by foreign particles. The pump should be completely drained, flushed, and filled with fresh fluid when contamination occurs.

1. Turn the pump off.

2. Place a container under the drain valve to hold the expelled oil. Open the drain valve and remove the exhaust muffler; most of the oil will drain out.

3. Turn on the pump and let it run for one minute, then turn it off.

4. Remove the intake hose (red rubber hose) and insert a funnel in its place.

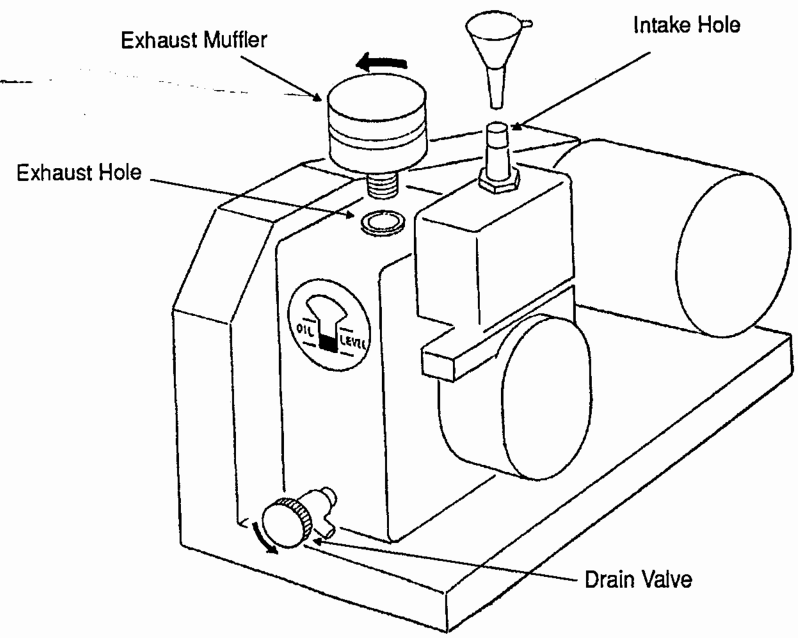


Figure 8-6. Replacing Vacuum Pump Fluid

5. Cover the exhaust hole with a paper towel so oil does not splash out.

6. Turn the pump on.

7. Add about 1/2 cup oil at a time into the intake hole (keep drain valve open).

8. Put a little pressure on the exhaust by placing your thumb over the exhaust hole to pull the oil through the system.

9. Repeat Steps 7 and 8 until the oil coming out is clean.

10. Turn the pump off.
11. Reconnect the intake hose.
12. Add clean oil through the exhaust hole until the level is between the indicator lines on the pump.
13. Replace the exhaust cap and hand-tighten.
14. Turn on the pump and recheck the oil level after five minutes. Add more oil if necessary.

CHECKING THE VALVES FOR LEAKS

Leakage by valves handling mercury can be detected by one of two methods. The first is most commonly used because each of the valves is isolated. The second method can be employed by pressurizing the sample preparation portion of the system just as in performing an analysis. Instructions follow for each method.

Method One

NOTE

An assistant is needed to observe the mercury deaerator during this procedure.

WARNING

Turn off power to the unit before removing the rear panel. Failure to do so could result in personal injury.

1. Place the Power ON/OFF switch on the front panel in the OFF position.
2. Unscrew the seven retaining screws on the rear panel and remove the panel.
3. Place the Power ON/OFF switch on the front panel in the ON position.
4. Place blank rods in the low pressure ports and tighten the retaining knobs until firm.
5. Evacuate the manifold as follows:
   a. Press the EVACUATE SAMPLE SLOW button.
   b. When the indicated pressure drops below 1 psia, release the EVACUATE SAMPLE SLOW button and press the EVACUATE SAMPLE MED button.
   c. When the indicated pressure drops to 250 μmHg, release the EVACUATE SAMPLE MED button and press the EVACUATE SAMPLE FAST button.
   d. Allow evacuation to continue until the vacuum pressure is below 50 μmHg.
6. Momentarily press the MERCURY FILL button while the assistant observes the deaerator (refer to Figure 8-7). Immediately release the MERCURY FILL button when mercury starts spurting into the deaerator.

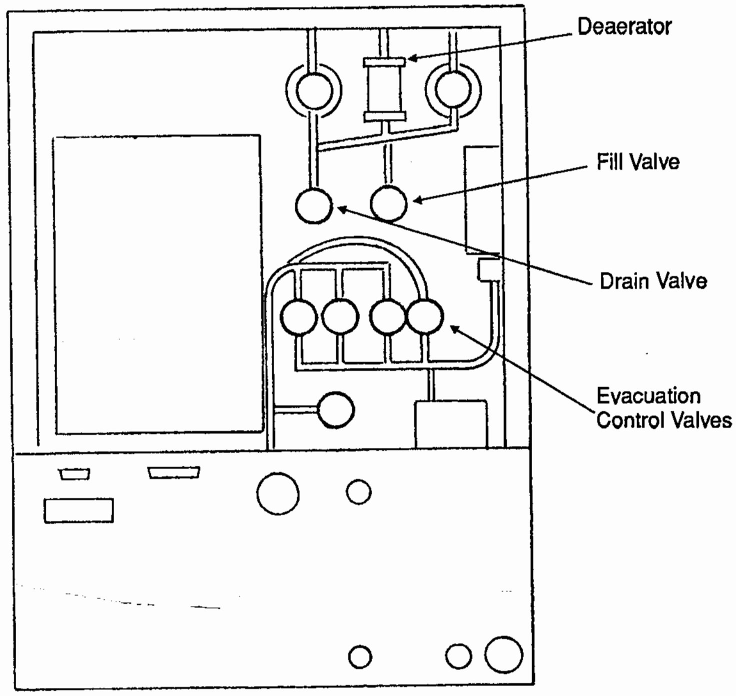


Figure 8-7. Low Pressure and Mercury Handling Manifolds

7. Observe the deaerator. Mercury should not continue to leak from the fill tube. If mercury continues to leak, the FILL valve must be cleaned.

8. Momentarily press the MERCURY DRAIN button. Immediately release the MERCURY DRAIN button when mercury starts flowing up into the deaerator. Observe the deaerator. If mercury continues to flow into it, then the DRAIN valve must be cleaned.

9. After checking the valves, mercury must be drained back into the reservoir to prevent mercury contamination.

   a. Press the ADJUST LOW PRESSURE button.

   b. When 14 psia is reached, release the button.

   c. Press the EVACUATE RESERVOIR button and wait 20-30 seconds.

   d. Press the MERCURY DRAIN button until the MERCURY DOWN indicator turns on, then release both buttons.

   e. Press the ADJUST LOW PRESSURE button until 25 psia is reached, then release it.

   f. Press the MERCURY FILL button for 10-20 seconds, then release it.

10. If leaks were found, the valves must be cleaned (refer to **Cleaning Valves** later in this chapter).

Method Two

1. Raise the pressure to 25-30 psia.
2. Remove the sealing plug from the mercury reservoir.
3. Valve leakage is indicated by gas bubbles seen escaping through the mercury surface. Correction requires cleaning the valves.

CLEANING VALVES

WARNING
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. If the rear panel has not been removed, remove it by unscrewing the seven retaining screws.
3. The fill and drain valves are the upper two (refer to Figure 8-7). Remove the 11/16-in. nut retaining the valve actuating coil from each and pull the coils off.
4. Remove the plunger housing from each using the special spanner nut from the accessory kit.
5. Hold a container below the valves to capture any retained mercury.
6. Clean the plunger and housing with IPA, 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 four lower solenoid valves, the evacuation control valves, are much less likely to collect debris. They can be cleaned in the same 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 preferred accumulation point is the restricting frits that control flow rates. The best way to remove moisture is to evacuate the PoreSizer 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.

Moisture accumulation can be avoided in the future by including a better drying system on the incoming air and by evacuating the manifold during those periods that the PoreSizer is not in use: overnight and during the weekends.

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.

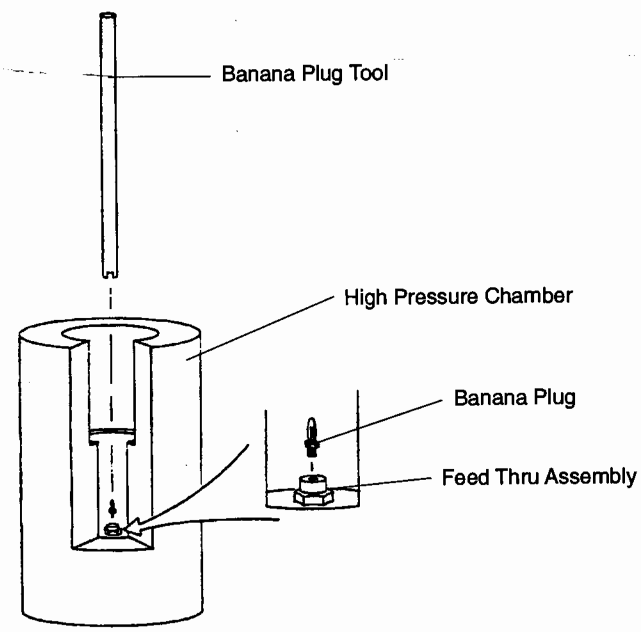


Figure 8-8. Replacing the Banana Plug in the High Pressure Chamber

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 low pressure capacitance detector 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.

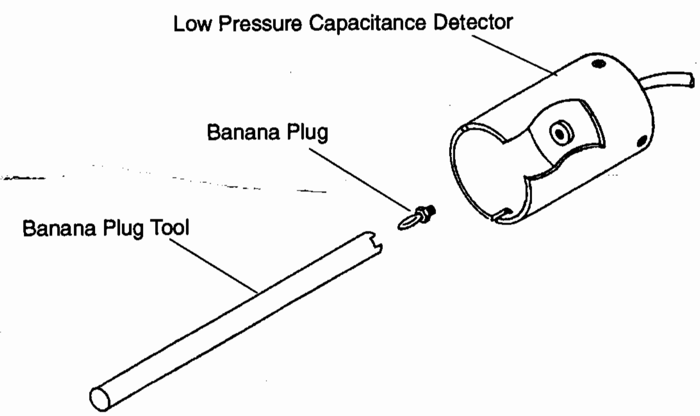


Figure 8-9. Replacing the Banana Plug in the Low Pressure Capacitance Detector

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

CHAPTER 9

ORDERING INFORMATION

ORDERING INFORMATION

Part Number	Item and Description
	9320 PoreSizer and Accessories
932-00000-00	PoreSizer 9320, for pore structure studies from 360 to 0.006 µm pore opening diameter. Includes software and interface cable for automatic high-pressure operation via accessory control module. Requires vacuum pump for operation. Universal electrical input.
Call for current model and part number	Control Module, controls analyses, reduces data, and generates reports; includes color monitor, dot-matrix printer, and cables (100/120 V, 50/60 Hz).
Call for current model and part number	Control Module, controls analyses, reduces data, and generates reports; includes color monitor, dot-matrix printer, and cables (230 V, 50/60 Hz)
062-00000-11	Vacuum Pump and hoses, direct drive, two-stage, with vented exhaust (115 V, 50/60 Hz)
062-00000-23	Vacuum Pump and hoses, direct drive, two-stage, with vented exhaust (230 V, 50/60 Hz)
920-62606-00	Vacuum Gauge Tube.
004-25105-00	Ferrule, front, Teflon, for 3/8-in. OD tubing
004-25103-00	Ferrule, front, Teflon, for 1/4-in. OD tubing
004-25131-00	Ferrule, front, Teflon, for 1/8-in. OD tubing
004-25104-00	Ferrule, rear, nylon, for 1/4-in. OD tubing
004-25106-00	Ferrule, rear, nylon, for 3/8-in. OD tubing
004-25132-00	Ferrule, rear, nylon, for 1/8-in. OD tubing
932-42801-00	Operator's Manual
932-33600-00	Extended operation supplies. Includes fluids, gaskets, plungers, etc.
932-33601-00	Spare Parts Kit. Primarily for those customers in remote locations or where spare parts are to be stocked on site. Includes printed circuit boards, plungers, etc.
	General Accessories for Mercury Porosimetry
150-10000-00	Contact Anglometer 1501, for wetting (contact) angle between liquid and flat surface or packed powder bed
004-16004-01	Mercury, 5 lbs
004-16007-00	Vacuum Grease, Apiezon-H, 25 g; operating range is 15 to 250°C

Part Number	Item and Description
920-16001-00	High Pressure Fluid, 1 Liter, a proprietary blend of oils
004-16003-01	Vacuum Pump Oil, 1 liter
008-25001-00	Syringe, 60 cc, used to withdraw high-pressure fluid when cleaning chambers
930-54601-00	Cleaning Brush, 0.4-cc stems, used to aid in the cleaning of the penetrometer
930-54602-00	Cleaning Brush, 1.1-cc stems, used to aid in the cleaning of the penetrometer
930-54603-00	Cleaning Brush, 1.8-cc stems, used to aid in the cleaning of the penetrometer
930-17801-00	Penetrometer Simulator, 82 pF
930-17801-01	Penetrometer Simulator, 150 pF
930-17801-05	Penetrometer Simulator, 15 pF
004-16822-00	Reference Material, average pore diameter ≅ 0.1 µm, pore volume ≅ 0.5 cm^3/g; 15 g
900-42705-00	Plotting Sheet, Cumulative Pore Volume Distribution, pad of 100
930-25849-00	Banana Plug
922-09808-00	Tool, banana plug
	Penetrometer Assemblies for Solid Samples
920-61713-00	Penetrometer, solids; Sample Volume - 3 cc, Intrusion Volume - 0.39 cc
920-61715-00	Penetrometer, solids; Sample Volume - 3 cc, Intrusion Volume - 1.1 cc
920-61707-00	Penetrometer, solids; Sample Volume - 5 cc, Intrusion Volume - 0.38 cc
920-61709-00	Penetrometer, solids; Sample Volume - 5 cc, Intrusion Volume - 1.1 cc
920-61711-00	Penetrometer, solids; Sample Volume - 5 cc, Intrusion Volume - 1.8 cc
920-61701-00	Penetrometer, solids; Sample Volume - 15 cc, Intrusion Volume - 0.38 cc
920-61703-00	Penetrometer, solids; Sample Volume - 15 cc, Intrusion Volume - 1.1 cc
920-61705-00	Penetrometer, solids; Sample Volume - 15 cc, Intrusion Volume - 1.8 cc
920-61724-00	Penetrometer, solids; Sample Volume - 15 cc, Intrusion Volume - 3.1 cc
920-61725-00	Penetrometer, solids; Sample Volume - 15 cc, Intrusion Volume - 4.0 cc
	Penetrometer Assemblies for Powder Samples
920-61714-00	Penetrometer, powder; Sample Volume - 3 cc, Intrusion Volume - 0.39 cc
920-61716-00	Penetrometer, powder; Sample Volume - 3 cc, Intrusion Volume - 1.1 cc
920-61708-00	Penetrometer, powder; Sample Volume - 5 cc, Intrusion Volume - 0.38 cc
920-61710-00	Penetrometer, powder; Sample Volume - 5 cc, Intrusion Volume - 1.1 cc
920-61712-00	Penetrometer, powder; Sample Volume - 5 cc, Intrusion Volume - 1.8 cc

Part Number	Item and Description
920-61702-00	Penetrometer, powder; Sample Volume - 15 cc, Intrusion Volume - 0.38 cc
920-61704-00	Penetrometer, powder; Sample Volume - 15 cc, Intrusion Volume - 1.1 cc
920-61706-00	Penetrometer, powder; Sample Volume - 15 cc, Intrusion Volume - 1.8 cc
	Other Penetrometer Parts
920-25871-00	Seal (cap), replacement, for 3- and 5-cc sample penetrometers.
920-25872-00	Insulator, replacement, for 3- and 5-cc sample volume penetrometers.
920-25827-00	Retaining collar, replacement, for 3-cc sample volume penetrometers.
920-25828-00	Nylon Spacer, for 3-cc sample volume penetrometer assemblies.
920-25825-00	Retaining collar, replacement, for 5- and 15-cc sample volume penetrometers.
920-25826-02	Insulator, replacement, for 15-cc sample volume penetrometers.
920-25826-01	Seal (cap), replacement, for 15-cc sample volume penetrometers.
920-25829-00	Spring, replacement, for all penetrometers.

CHAPTER 10

ERROR MESSAGES

- Status Display Error Messages
- Data Entry Error Messages
- Fatal Error Messages

ERROR MESSAGES

There are three types of error messages that may be displayed on the video monitor: status display error messages, data entry error messages, and fatal error messages. Status display error messages appear in the status display portion of the screen. Data entry messages appear on line 25 of the screen. Fatal error messages appear on line one of the screen.

STATUS DISPLAY ERROR MESSAGES

The following error messages may appear on line seven of the status display. When there is more than one message, each message will be alternately displayed for about five seconds.

One or more causes and corrective actions are given for each error message. The corrective action for some of the error messages involves assigning attributes to a file using the DOS command ATTRIB. Refer to the DOS manual for detailed instructions on the ATTRIB command.

There are four types of status display error messages:

- Messages that start with HP are high pressure operation error messages. These messages can be cleared by entering the Start High Pressure Run screen or either of the High Pressure Control screens.
- Messages that start with LP are low pressure operation error messages. These messages can be cleared by entering the Start Low Pressure Run screen or the Low Pressure Run Control screen.
- Messages that start with RP are report generation error messages. These messages can be cleared by entering the Start Report screen.
- Messages that do not pertain to a PoreSizer operation (such as printer or plotter errors) are displayed on line seven of the status display in reverse video (not flashing). These messages can be cleared by entering the Start Report screen.

HP: Intensifier lower limit

Cause: You requested a pressure lower than pressure in the system when the run was started or atmospheric high pressure offset was computed when system was not at atmospheric pressure.

Action: Make sure the high pressure system is at atmospheric pressure, verify that the pressure and/or rate sequence table pressures are all above atmospheric pressure plus head pressure, then start the run again.

HP: Intensifier upper limit

Cause: Not enough oil is in the system.

Action: Add oil to the high pressure chamber.

Cause: There is a leak in the hydraulic system.

Action: Contact appropriate service personnel.

Cause: The vent valve is not sufficiently tightened.

Action: Tighten the vent valve and restart the high pressure run.

Cause: Too much air is in the system.

Action: Contact appropriate service personnel.

HP: Invalid target pressure requested

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

HP: Operator canceled run

Cause: The analysis was canceled by the operator.

Action: No action necessary, this is a status message.

HP: Run canceled; pressure table number is required.

Cause: You tried to start a high pressure analysis without specifying a pressure table.

Action: Enter a pressure table number on screen **8p1 Start High Pressure Run** before attempting to start a high pressure analysis.

HP: Run canceled; rate sequence table number is required.

Cause: You selected rate sequence for run method, then tried to start a high pressure analysis without specifying a rate sequence table.

Action: Enter a rate sequence table number on screen **8p1 Start High Pressure Run** before attempting to start a high pressure analysis.

HP: Unable to access (file name)

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

HP: Unable to close (file name)

Cause: Hard disk may be damaged.

Action Refer problem to appropriate service personnel.

HP: Unable to create (file name)

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

HP: Unable to open (file name)

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

Cause: You selected a file that may be tagged "read only." This is the result of interrupting the analysis program during file access.

Action: Exit the analysis program to DOS. Use the DOS command ATTRIB to determine if the file is tagged "read only" and to remove the tag.

HP: Unable to read (file name)

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

HP: Unable to write (file name)

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

Instrument interface I/O error

Cause: The cable connecting the PoreSizer to the control module is not properly installed or is defective.

Action: Check the cable. Make sure it is properly connected to both units.

Cause: The baud rate switch on the PoreSizer processor card is not set to 9600 baud.

Action: Set the baud rate switch on the PoreSizer processor card to 9600 baud.

Cause: PoreSizer power is off.

Action: Place the Power Switch in the ON (I) position.

Cause: The PoreSizer is malfunctioning.

Action: Refer the problem to the appropriate service personnel.

Cause: The control module is malfunctioning.

Action: Refer the problem to the appropriate service personnel. If you were performing an analysis when the problem occurred, you can turn off the PoreSizer and the control module, then turn the PoreSizer back on and operate the PoreSizer using the front panel controls.

LP: Unable to open (file name)

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

Cause: You selected a file that may be tagged "read only." This is the result of interrupting the analysis program during file access.

Action: Exit the analysis program to DOS. Use the DOS command ATTRIB to determine if the file is tagged "read only" and to remove the tag.

LP: Unable to write (file name)

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

Plotter failed initialization

Cause: Communications parameters between the control module and plotter do not match.

Action: Turn off power to the plotter and then turn it back on. If the problem still exists, check for proper installation of the plotter.

Cause: Serial interface cards not configured properly.

Action: Refer problem to appropriate service personnel.

Plotter not ready

Cause: Paper not loaded in plotter before a plot is requested.

Action: Load a sheet of paper into the plotter before requesting a plot.

Cause: Improper cable used to connect the plotter to the control module.

Action: Install correct cable (refer to **Connecting Cables** in Chapter 2).

Cause: Following cancellation of reports, the proper re-initialization indication was not received from the plotter.

Action: Turn off power to the plotter and then turn it back on.

Cause: Plotter interface cable is not connected properly or is defective.

Action: Check plotter interface cable connections; correct as necessary.

Cause: Plotter is malfunctioning.

Action: Refer problem to appropriate service personnel.

Cause: Plotter interface misconfigured.

Action: Properly configure plotter interface.

Printer error

Cause: Following cancellation of reports, the proper re-initialization indication was not received from the printer.

Action: Turn off power to the printer and then turn it back on.

Cause: Printer interface cable is not connected properly or is defective.

Action: Check printer interface cable connections; correct as necessary.

Cause: Printer is malfunctioning.

Action: Refer problem to the appropriate service personnel.

Printer not selected

Cause: The SELECT or ONLINE switch on the printer is disabled.

Action: Enable the SELECT or ONLINE switch on the printer. (Refer to printer manufacturer's manual for instructions.)

Printer out of paper

Cause: Printer is out of paper.

Action: Load paper into the printer. (Refer to printer manufacturer's manual for instructions.)

RP: No data for sample

Cause: Analysis for report sample is still in process, but not enough data have been collected for the report.

Action: Wait for the current analysis to collect enough data and then request the sample report.

Cause: Analysis for report sample was canceled by the operator before enough data were collected.

Action: Run the analysis and allow enough data to be collected for the report.

Cause: Analysis for report sample was canceled by the system before enough data were collected.

Action: Correct the condition which caused cancellation of the analysis and repeat the analysis before requesting the report.

Cause: Sample has not been analyzed yet.

Action: Run the analysis and allow enough data to be collected for the report.

RP: Unable to open (file name)

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

RP: Unable to read (file name)

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

DATA ENTRY ERROR MESSAGES

The following error messages are displayed on line 25 of the screen. These messages appear when you enter information incorrectly or when a requested action cannot be performed by the system. They will only remain on the screen for a few seconds and will be accompanied by a beep.

Can not find any directory entries

Cause: You requested a list of directory entries to be displayed on the screen and no entries were found.

Action: Abandon the request.

Can not find any directory entries for requested date

Cause: You requested a list of directory entries for a rate sequence table, pressure table, tabular data set or report options set on or after a specified date and no entries were found.

Action: Either change the specified date or abandon the request.

Duplicate sample numbers.

Cause: You entered the same sample file number to be run on both the left low pressure port and the right low pressure port.

Action: Change the sample file number specified for the left or right low pressure port.

Enter a sample file number.

Cause: You tried to copy a sample file but did not enter a sample file number.

Action: Enter the number of the sample file to be copied.

Enter a target file number.

Cause: You tried to copy a sample file but did not enter a target file number.

Action: Enter the number of the file to which the sample file will be copied.

Enter valid value to leave field.

Cause: You tried to leave a field that requires an entry without entering a valid value.

Action: Enter a valid value.

Error recording base intrusion reading.

Cause: A problem was encountered in the interface between the control module and the PoreSizer.

Action: Refer to the status display error message "Instrument interface I/O error".

Error recording base low pressure and intrusion readings.

Cause: A problem was encountered in the interface between the control module and the PoreSizer.

Action: Refer to the status display error message "Instrument interface I/O error".

Error recording high pressure transducer offset.

Cause: A problem was encountered in the interface between the control module and the PoreSizer.

Action: Refer to the status display error message "Instrument interface I/O error".

Error sample directory is full.

Cause: All of the available sample numbers (1-400) for the current directory are in use.

Action: Create a new sample directory and select it as the new current directory, or delete some samples from the current directory. (Refer to Chapter 7 for instructions.)

File already exists

Cause: You entered the file number of a file that already exists.

Action: Enter a file number that does not already exist.

File does not exist

Cause: You entered a file number for which no file currently exists.

Action: Enter a file number for which a file currently exists. (If you do not know what files currently exist, go to the appropriate Directory screen for a list of files.)

High pressure run in progress

Cause: You tried to access a Start Run screen while a high pressure run was in progress.

Action: Wait until the high pressure run has been completed before starting another run.

Invalid sample directory name.

Cause: You entered a sample directory name that is not a valid directory name.

Action: Enter a valid sample directory. (For a list of rules for naming directories, refer to the DOS manual.)

Low pressure run already completed for sample.

Cause: You requested a low pressure analysis on a sample that has already been run.

Action: Enter a sample that has not already been run.

Low pressure run in progress

Cause: You tried to start a run when one was already in process.

Action: Wait for the current run to finish before requesting another.

Maximum number of data points already recorded.

Cause: You tried to enter more than 250 data points.

Action: You may not enter any more data points. Press F4 to finish the run.

Maximum number of data points have been recorded.

Cause: The maximum number of data points in the pressure table have been recorded.

Action: No action; this is a status message.

No automatic high pressure run in progress.

Cause: You tried to skip a run step, suspend/resume a run, or cancel a run when no run was in progress.

Action: Abandon request.

No communication ports available

Cause: You tried to enter terminal emulation mode but have no serial ports available.

Action: Abandon request.

Cause: Appropriate serial interface cards are not installed or are installed incorrectly.

Action: Install appropriate serial interface cards. Refer to control module manufacturer's manual for instructions.

Cause: Serial interface cards are misconfigured.

Action: Check serial interface card configuration. (Contact Micromeritics Service Department for information if necessary.) Correct as necessary.

Cause: Serial interface cards are malfunctioning.

Action: Replace serial interface cards or contact appropriate service personnel.

No deletion allowed.

Cause: The minimum number of values has been reached for the table or column to which entries are currently being made.

Action: You may modify the entry but it cannot be removed.

No insertion allowed.

Cause: The maximum number of values has been reached for the table or column to which entries are currently being made.

Action: You may modify or delete the entry, but you may not add an entry.

No low pressure run completed for sample.

Cause: You tried to start a high pressure run on a sample that has not completed a low pressure run.

Action: Complete the low pressure run on the sample before starting a high pressure run.

No low pressure run in progress.

Cause: You tried to access the Low Pressure Run Control screen when there was no low pressure run in progress.

Action: Abandon request.

No manual high pressure run in progress.

Cause: You tried to access the Manual High Pressure Run Control screen when there was no manual high pressure run in progress.

Action: Abandon request.

No more characters allowed

Cause: You tried to enter more characters than the maximum number allowed for a particular field.

Action: Check the maximum length allowed (refer to Chapter 6); enter up to the maximum length allowed for the field.

No more digits allowed

Cause: You tried to enter more digits than the maximum number allowed for a particular field.

Action: Check the range of numbers allowed (refer to Chapter 6); enter up to the maximum length allowed for the field.

No more sample subdirectories are allowed.

Cause: You tried to add a sample directory when the maximum number (200) of sample directories had already been entered.

Action: Back up sample directories which are not used regularly on diskettes using the Back Up Utility (press F10, F5 from the Main Function Menu). Then exit the analysis program to DOS. Use DOS commands to delete the directories which were backed up to diskette. Return to the analysis program to add a new directory.

Only digits may be entered

Cause: You tried to enter letters or other characters in a field in which only numbers may be entered.

Action: Enter a valid number.

Only printable characters may be entered

Cause: You tried to enter a non-printable character.

Action: Enter a printable character.

Plotter failure.

Cause: Following cancellation of reports, the proper re-initialization indication was not received from the plotter.

Action: Turn off power to the plotter, then turn it back on.

Cause: Plotter interface cable is not connected properly or is defective.

Action: Check plotter interface cable connections; correct as necessary.

Cause: Plotter is malfunctioning.

Action: Refer problem to appropriate service personnel.

Pressure table not saved, at least one entry is required.

Cause: You tried to save a pressure table that did not have any entries.

Action: Make at least one entry in the table before saving the table.

Printer failure.

Cause: Following cancellation of reports, the proper re-initialization indication was not received from the printer.

Action: Turn off power to the printer, then turn it back on.

Cause: Printer interface cable is not connected properly or is defective.

Action: Check printer interface cable connections; correct as necessary.

Cause: You tried to cancel reports when the printer was not selected for report destination.

Action: No action necessary.

Cause: Printer is malfunctioning.

Action: Refer problem to appropriate service personnel.

Rate sequence table not saved, at least one entry is required.

Cause: You tried to save a rate sequence table that did not have any entries.

Action: Make at least one entry in the table before saving the table.

Run in progress for specified sample.

Cause: You tried to delete or change a sample information file for a sample that is currently being analyzed.

Action: Wait until the analysis is complete or cancel the analysis before attempting to change or delete the sample information file.

Sample directory already exists.

Cause: When trying to add a sample directory, you entered the name of a sample directory that already exists.

Action: Enter a sample directory name that does not already exist.

Sample directory does not exist.

Cause: You entered the name of a sample directory that does not exist.

Action: Enter the name of an existing sample directory. (To display a list of sample directories, press F10, F7, F6 from the Main Function Menu.)

Sample has already completed high pressure run.
Match-up will be at last collected point.

Cause: You started a high pressure run on a sample that has already been run.

Action: This is a status message. Press PgDn to start the high pressure run or Esc if you do not wish to start the run.

Selection not allowed during termination.

Cause: You tried to suspend or cancel an automatic high pressure run that was terminating.

Action: Abandon request; you cannot suspend or cancel a run that is terminating.

Skip not allowed for rate sequence runs.

Cause: You tried to skip a step during a rate sequence automatic high pressure run before the run was in the termination phase.

Action: You cannot skip a step until the run is in the termination phase; abandon the request or choose another option.

Tabular data set not saved, at least one entry is required.

Cause: You tried to save a tabular data set that did not have any entries.

Action: Make at least one entry in the tabular data set before saving it.

Transmission error: enter character again.

Cause: You tried to transmit over the serial line and a serial communication error occurred.

Action: Make sure the serial communication cable from the control module is properly connected to the remote terminal. Make sure the communications options are appropriately set for the control module and the remote terminal. Then try the transmission again.

Unable to close (file name)

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

Cause: Diskette may be damaged.

Action: Terminate the analysis program. Try cleaning the disk drive (drive A). If this does not eliminate the problem, use a backup copy of the file.

Unable to create (file name)

Cause: Not enough space left on disk.

Action: Copy files which are not used regularly from the hard disk to floppy diskette, and then delete them from the hard disk.

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

Unable to create spool file (file name):

Cause: Not enough space left on the disk.

Action: Copy files which are not used regularly from the hard disk to diskette, and then delete them from the hard disk.

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

Unable to delete (file name)

Cause: You selected a file that may be tagged "read only." This is the result of interrupting the analysis program during file access.

Action: Exit the analysis program to DOS. Use the DOS command ATTRIB to determine if the file is tagged "read only" and to remove the tag.

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

Unable to open (file name)

Cause: You selected a file that may be tagged "read only." This is the result of interrupting the analysis program during file access.

Action: Exit the analysis program to DOS. Use the DOS command ATTRIB to determine if the file is tagged "read only" and to remove the tag.

Cause: Not enough space left on the disk.

Action: Copy files which are not used regularly from the hard disk to diskette, and then delete them from the hard disk.

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

Cause: Diskette may be damaged.

Action: Terminate the analysis program. Try cleaning the disk drive (drive A). If this does not eliminate the problem, use a backup copy of the file.

Cause: Diskette has a write protect tab.

Action: Remove write protect tab from diskette or use a diskette without tab.

Unable to read (file name)

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

Cause: Diskette may be damaged.

Action: Terminate the analysis program. Try cleaning the disk drive (drive A). If this does not eliminate the problem, use a backup copy of the file.

Unable to record data [F3] to try again.

Cause: A problem was encountered in the interface between the control module and the PoreSizer.

Action: Press F3 to try again. If the problem still occurs, refer to the status display error message "Instrument interface I/O error".

Unable to seek within file: (file name)

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

Unable to update directory: (directory name)

Cause: You selected a file that may be tagged "read only." This is the result of interrupting the analysis program during file access.

Action: Exit the analysis program to DOS. Use the DOS command ATTRIB to determine if the file is tagged "read only" and to remove the tag.

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

Cause: Not enough space left on the disk.

Action: Copy files which are not used regularly from the hard disk to diskette, and then delete them from the hard disk.

Unable to update the autoexec file

Cause: The AUTOEXEC.BAT file may be tagged "read only". This may be the result of interrupting the analysis program during file access.

Action: Exit the analysis program to DOS. Use the DOS command ATTRIB to determine if the file is tagged "read only" and to remove the tag.

Cause: Not enough space left on the disk.

Action: Copy files which are not used regularly from the hard disk to diskette, and then delete them from the hard disk.

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

Unable to write (file name)

Cause: Not enough space left on the hard disk.

Action: Copy files which are not used regularly from the hard disk to floppy diskette, and then delete them from the hard disk.

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

Unable to write spool file: (file name):

Cause: Not enough space left on the disk.

Action: Copy files which are not used regularly from the hard disk to diskette, and then delete them from the hard disk.

Cause: Hard disk may be damaged.

Action: Refer problem to appropriate service personnel.

Wait for printed reports to finish.

Cause: You requested a report be displayed on the screen while a previously-requested report to the screen was being processed.

Action: Wait for the current report to finish, then initiate the request for another report.

Wait until all runs and reports are complete.

Cause: From the Utilities Menu, you tried to select either Format Disk, Back Up Utility, Restore Utility, or Exit to DOS while analyses or reports were still in progress.

Action: Wait until analyses and reports are complete or cancel analyses and reports.

Wait until all runs are complete.

Cause: You tried to update a sample directory that contains a sample currently being analyzed.

Action: Wait until the analysis is complete or cancel it.

FATAL ERROR MESSAGES

Fatal error messages, which are displayed on line one of the screen, cause a "lock up" of the software. A fatal error is often caused by a damaged hard disk. When a fatal error occurs, processing cannot continue until corrective action is taken. Call the appropriate service personnel when one of the following error messages is displayed:

ERROR LOADING DISK INTERRUPT VECTOR

ERROR LOADING EXEC...ABORTING

EXEC ERROR. FILE NOT LOADED

FATAL ERROR INITIALIZING VRTX

Hard disk parameter mismatch...system halted

Initialization failed...instrument interface malfunction

math coprocessor malfunction - system halted

math coprocessor not present - required for instrument operation

No Communication Port For Instrument

NON-MASKABLE INTERRUPT

PROGRAMMER ERROR - CALL TO DOS TRAPPED

*****STACK OVERFLOW*****

Unable to read disk parameters...initialization failed

APPENDIX A

ANALYSIS PROGRAM FORMS

micromeritics

Date: ______________________

Current Directory: ______________

PoreSizer 9320
Sample Data Sheet

Press F3 twice from the Main Function Menu to display the Add Sample Information screen.

3.3p1 Add Sample Information

	Port 1	Port 2	
Sample number			(1 to 400)
Sample ID			(40 char. max)
Submitter ID			(40 char. max)
~~Operator ID~~			(20 char. max)
Penetrometer number			(10 char. max)
Penetrometer constant			(10.790 µL/pF
Penetrometer weight			(1.0000 g)
Maximum head pressure			(4.680 psi)
Penetrometer volume			(1.0000 mL)
Stem volume			(0.4120 mL)
Advancing contact angle			(130.00 deg)
Receding contact angle			(130.00 deg)
Hg surface tension			(485.0 dynes/cm)
Hg density			(13.5335 g/mL)
Sample weight			(1.0000 g)
Sample + penetrometer weight			

Press PgDn .

3.3p2 Add Sample - Report Options

Report options set number ______________________ (1 to 50)

Press PgDn .

Shaded Value = Default

3.3p3 Add Sample - Tabular Data Set

Tabular data page	______	(yes or no)
Tabular data defined by	______	(collected points or tabular data set)
Tabular data set number	______	(1 to 50)

Press [PgDn] to add graph pages or press [F2] twice, then [F8] to display Start High Pressure Run screen.

8p1 Start High Pressure Run

	Run 1	Run 2	
Manual and Automatic Runs:			
Sample number	______	______	(1 to 400)
Run type	______	______	(automatic or manual)
Start report after run	______	______	(yes or no)
Report destination	______	______	(printer, serial line, printer/plotter, drive a:, drive c:)
Atmospheric pressure	______	______	(10.0 to 20.0 psia or 0.068 to 0.137 MPa)
Sample weight + penetrometer weight + mercury weight	______	______	(1.0000 to 1000.0000 g)
Automatic Runs only:			
Pressure table number	______	______	(1 to 50)
Run method	______	______	(equilibrated or rate sequence)
Rate sequence table number	______	______	(1 to 50)
Equilibration time	______	______	(0 to 10,000 seconds)
Maximum intrusion volume	______	______	(0.000 to 100.000 mL/g)

Shaded Value = Default

micromeritics®

Date: ______________________
Current Directory: ______________________

Press F6, F5, F3 from the Main Function Menu to display the Add Report Options Set screen.

PoreSizer 9320
Report Options WorkSheet

6.5.3p1 Add Report Options Set

Report options set number	(1 to 50)	______
Report options set ID	(24 characters max)	______
Report negative intrusion	(yes or no)	______
Report title	(50 characters max)	______
Summary page	(yes or no)	______
Calculation range specified as	(pressure or diameter (radius))	______
Calculation range		from ______ to ______

Press PgDn.

6.5.3p2 Add Report Options - Tabular Data Set

Tabular data page	(yes or no)	______
Tabular data defined by	(collected points or tabular data set)	______
Tabular data set number	(1 to 50)	______
Columns 1 through 6		______

Choices:

none	differential intrusion volume
pressure	log differential intrusion volume
pore diameter (radius)	cumulative pore area
mean diameter (radius)	incremental pore area
cumulative intrusion volume	percent of total intrusion volume
incremental intrusion volume	

Press PgDn.

6.5.3p3 Add Report Options - Graph Page 1

Graph	(yes or no)	______
X-axis choice	(pressure or diameter (radius))	______
X-axis scale	(log or normal)	______
X-axis range		from ______ to ______
Y-axis choice		______
Y-axis range		from ______ to ______
(0 = autoscale to 105% of the maximum value in the data)		
Type of data to plot	(collected points, continuous curve, collected points and continuous curve)	______
Intrusion data to plot	(all, none, first)	______
Extrusion data to plot	(all, none, first)	______

Press PgDn.

Shaded Value = Default

6.5.3p4 Add Report Options - Graph Page 2

Graph	(yes or no)	______
X-axis choice	(pressure or diameter (radius))	______
X-axis scale	(log or normal)	______
X-axis range		from______to ______
Y-axis choice		______
Y-axis range		from______to ______
(0 = autoscale to 105% of the maximum value in the data)		
Type of data to plot	(collected points, continuous curve, collected points and continuous curve)	______

Press PgDn.

6.5.3p5 Add Report Options - Graph Page 3

Graph	(yes or no)	______
X-axis choice	(pressure or diameter (radius))	______
X-axis scale	(log or normal)	______
X-axis range		from______to ______
Y-axis choice		______
Y-axis range		from______to ______
(0 = autoscale to 105% of the maximum value in the data)		
Type of data to plot	(collected points, continuous curve, collected points and continuous curve)	______

Press PgDn.

6.5.3p6 Add Report Options - Graph Page 4

Graph	(yes or no)	______
X-axis choice	(pressure or diameter (radius))	______
X-axis scale	(log or normal)	______
X-axis range		from______to ______
Y-axis choice		______
Y-axis range		from______to ______
(0 = autoscale to 105% of the maximum value in the data)		
Type of data to plot	(collected points, continuous curve, collected points and continuous curve)	______

Press PgDn.

6.5.3p7 Add Report Options - Graph Page 5

Graph	(yes or no)	______
X-axis choice	(pressure or diameter (radius))	______
X-axis scale	(log or normal)	______
X-axis range		from______to ______
Y-axis choice		______
Y-axis range		from______to ______
(0 = autoscale to 105% of the maximum value in the data)		
Type of data to plot	(collected points, continuous curve, collected points and continuous curve	______

Press PgDn.

Shaded Value = Default

PENETROMETER VOLUME CALIBRATION

Penetrometer: ______________________ Date: ______________________

Number: ______________________ 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: ____

____

____

DENSITY DETERMINATION BY MERCURY POROSIMETRY

Sample: ______________________________ Date: ______________________

______________________________ By: ______________________

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

Bulk Density Calculation:

5. Weight of penetrometer and sample . . . . ________ g
6. Weight of penetrometer, sample and mercury . . . . ________ g
7. Weight of sample (No. 5 minus No. 2) . . . . ________ g
8. Weight of mercury (No. 6 minus No. 5) . . . . ________ g
   Room temperature = _____°C
9. Volume of mercury (No. 8 divided by Density of Mercury*) . . . . ________ cc
10. Volume of sample (No. 4 minus No. 9) . . . . ________ cc
11. Bulk volume of sample (No. 10 divided by No. 7) . . . . ________ cc/g
12. Bulk density of sample (reciprocal of No. 11) . . . . ________ g/cc

Apparent Density Calculation:

13. Pore volume, mercury displaced in penetrometer stem . . . . ________ cc
    at a maximum pressure of . . . . ________ psia
14. Volume of sample, less pore volume (No. 10 minus No. 13) . . . . ________ cc
15. Specific volume of sample (No. 14 divided by No. 7) . . . . ________ cc/g
16. Apparent density of sample (reciprocal of No. 15) . . . . ________ g/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: ____

____

____

PORE AREA DISTRIBUTION DATA

Sample: ______________________________ Date: ______________________________

Sample Weight:__________ g Penetrometer No:________ By: ______________________________

Penetrometer Volume:__________ cc, Penetrometer Constant:__________ µL/pF

Mercury Surface Tension:__________ dynes/cm, Contact Angle:__________° Sheet____of____

PRESSURE ☐ psia or ☐ MPa		INTRUSION VOLUME		PORE SIZE	PORE AREA	
Corrected Pressure	Average Pressure	Cumulative (cc/g)	Incremental (cc/g)	☐ Diameter ☐ Radius (µm)	Incremental (m^2/g)	Cumulative (m^2/g)

PORE SIZE DISTRIBUTION DATA

Sample: ______________________________ Date: ______________________________

Sample Weight: __________ g Penetrometer No: ________ By: ______________________________

Penetrometer Volume: __________ cc, Penetrometer Constant: __________ µL/pF

Mercury Surface Tension: __________ dynes/cm, Contact Angle: __________ ° Sheet ____ of ____

PRESSURE ☐ psia or ☐ MPa			PORE SIZE ☐ Diameter ☐ Radius	INTRUSION			
Mercury Head Correction	Pressure Reading	Corrected Pressure	(µm)	Intrusion Reading (pF)	Cumulative Intrusion (ΣΔpF)	Cumulative Pore Volume (cc)	Cumulative Pore Volume (cc/g)

micromeritics®

micromeritics

RETURN MATERIAL AUTHORIZATION FORM

MICROMERITICS or its authorized representative must be contacted for approval before any goods are returned. One copy of this form must accompany all material returned for any reason. Forward a second copy directly to person authorizing return.

Name of Sender ______________________________

Company ______________________________

Address ______________________________

City ______________________ State ____________ Zip ____________

Telephone Number ______________________

Purchase Order Number ______________________ Date ______________________
(To cover shipping and repair charges)

Model Number ______________________ Serial Number ______________________

Date Purchased ______________________

Reason for Return (and any special instructions) ______________________

____

____

____

____

____

Return Authorized by ______________________ Number ____________

One Micromeritics Drive • Norcross, GA 30093-1877 • Telex: 682-7018

Domestic Sales — (404) 662-3633
International Sales — (404) 662-3660
Domestic Repair Service — (404) 662-3666
Customer Service — (404) 662-3636

Form No. 008-42078-00
28 Oct 1988

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:

$$D = -(1/P)\, 4\gamma \cos \varphi$$

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 I 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: $P = 1.20 \times 10^{-3}$ Torr. 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.

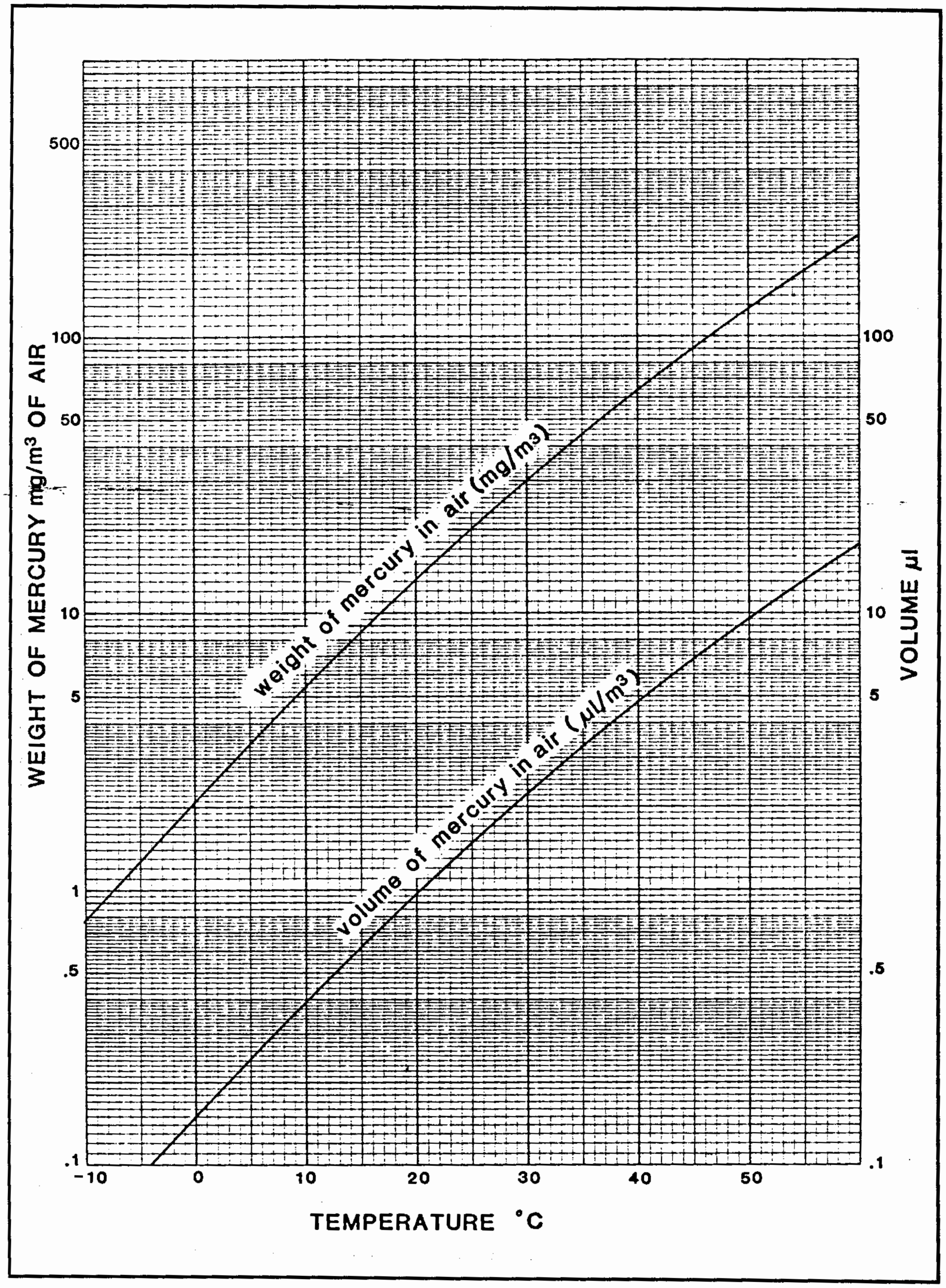


Figure C-1. Equilibrium Concentration of Mercury at 25°

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

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.

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. These instruments are also designed with a ventilation system that pulls ambient air over the counter, through the instrument and out a four-inch duct at the rear. A built-in tray work area allows the operator to easily wipe exposed mercury to a drain where it drops into a reservoir and is covered with oil. Our product literature on porosimetry supplies detailed site recommendations to assure safe operation.

The old adage about cleanliness holds true with any operation where mercury is used. Mercury vapor levels well below the accepted safe level can easily be achieved through regular monitoring, diligent handling and proper clean-up practices.

---

* Hg-X is a mercury decontaminant of water soluble, metallic mercury-sulphide converted powder in combination with a chelating compound and dispersing agent. This solution can be obtained through Action Associates, 100 Thompson Street, Pittston, PA 18640. Other mercury clean-up compounds and kits are available on the market and may be as effective as the Hg-X solution.

APPENDIX D

MERCURY HEAD CORRECTION

MERCURY HEAD CORRECTION

The mercury head correction calculates the increment of pressure applied to the sample by the weight of the column of mercury (in the stem) above the sample when the penetrometer is first turned to a vertical position for placement in the high pressure chamber (see Table D-1 and Figure D-1). This pressure increment decreases as mercury is then intruded into the sample and retreats down the penetrometer stem. The maximum correction, P_o, therefore depends on the total length, h_o, of the penetrometer stem: 24 cm for the 3-cc penetrometers or 23 cm for the 5- and 15-cc penetrometers. The head correction for any data point is decreased from the maximum value by the ratio of the current head, h, to the maximum head, h_o, which is expressed by

$$P_c = P_o \frac{h}{h_o} \tag{1}$$

The height, h, cannot be observed directly, but it can be calculated from the total intruded volume for the point in question.

The maximum head pressure, P_o, is given by

$$P_o = 0.1934\, h_o \text{ (psia)} \tag{2}$$

where h_o is either 23 cm (for 5- and 15-cc penetrometers) or 24 cm (for the 3-cc penetrometer) and the constant is a conversion factor which converts pressure in cm of Hg to pressure in psia. Also, the total stem volume of the penetrometer is given by

$$V_o = \frac{\pi}{4} d^2\, h_o \text{ (cc)} \tag{3}$$

where d is the inside diameter of the particular penetrometer stem (0.15, 0.25, 0.32, 0.425 or 0.481 cm). Further, the intruded volume, V, is related to the decrease in head by

$$V = \frac{\pi}{4} d^2\, (h_o - h) \text{ (cc)} \tag{4}$$

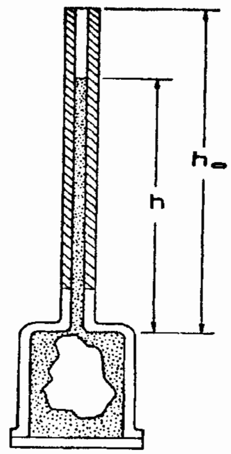


Figure D-1. Mercury Head Presssure Parameters

Combining equations (1), (3) and (4) gives the head correction

$$P_c = P_o - \frac{P_o}{V_o} V \text{ (psia)} \tag{5}$$

This head correction, P_c, can be calculated as a function of intrusion volume, V, by use of the constants in Table D-1 in conjunction with equation (5), or by specific tables in Table D-2. Figure D-2 shows the relationship graphically.

Table D-1. Terms in Head Correction Equation (No. 5)

Penetrometer Volume	Stem Diameter	Stem Length	P_o	V_o	P_o/V_o
3	0.150	24	4.64	0.41	11.27
3	0.250	24	4.64	1.19	3.90
5 and 15	0.150	23	4.45	0.39	11.35
5 and 15	0.250	23	4.45	1.13	3.94
5 and 15	0.320	23	4.45	1.84	2.42
15	0.425	23*	4.45	3.23	1.38
15	0.481	23*	4.45	4.14	1.08

*0.3 cm of the stem length is 0.15 cm diameter.

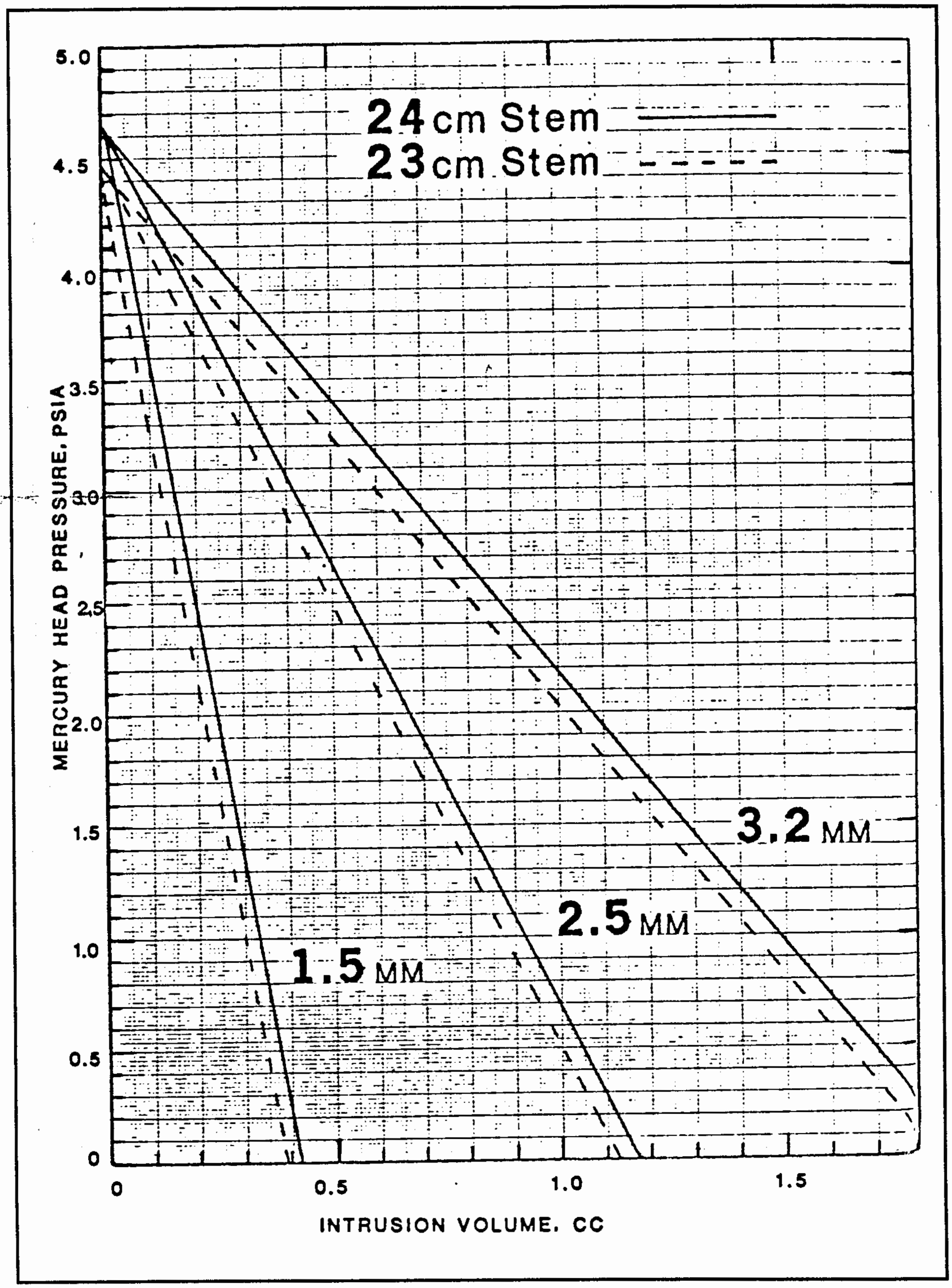


Figure D-2. Mercury Head Pressure Correction

Table D-2. Mercury Head Correction Lookup Tables

The following pages contain mercury head-pressure corrections for maximum measurable intrusion volumes of:

.366 cc

.387 cc

1.057 cc

1.116 cc

1.716 cc

3.007 cc

3.857 cc

Mercury Head-Pressure Corrections for .366 cc Volume

Total Capillary Length (H):	230 mm
Measurable Capillary (Stem) Length (I)	215 mm
Capillary Diameter (D):	1.473 mm
Maximum Measurable Intrusion Volume:	.366 cc

Intrusion Volume (cc)	% Stem Used	Head Pressure		
		(mmHg)	(psia)	(MPa)
.000	.0	230	4.45	.0307
.005	1.4	227	4.39	.0303
.010	2.7	224	4.33	.0299
.015	4.1	221	4.28	.0295
.020	5.5	218	4.22	.0291
.025	6.8	215	4.16	.0287
.030	8.2	212	4.11	.0283
.035	9.6	209	4.05	.0279
.040	10.9	206	3.99	.0275
.045	12.3	203	3.94	.0271
.050	13.7	200	3.88	.0267
.055	15.0	197	3.82	.0264
.060	16.4	194	3.77	.0260
.065	17.8	191	3.71	.0256
.070	19.1	188	3.65	.0252
.075	20.5	185	3.60	.0248
.080	21.9	183	3.54	.0244
.085	23.2	180	3.48	.0240
.090	24.6	177	3.43	.0236
.095	26.0	174	3.37	.0232
.100	27.3	171	3.31	.0228
.105	28.7	168	3.26	.0224
.110	30.1	165	3.20	.0220
.115	31.4	162	3.14	.0217
.120	32.8	159	3.08	.0213
.125	34.2	156	3.03	.0209
.130	35.5	153	2.97	.0205
.135	36.9	150	2.91	.0201
.140	38.3	147	2.86	.0197
.145	39.6	144	2.80	.0193
.150	41.0	141	2.74	.0189
.155	42.3	138	2.69	.0185
.160	43.7	136	2.63	.0181
.165	45.1	133	2.57	.0177
.170	46.4	130	2.52	.0173
.175	47.8	127	2.46	.0170

Intrusion Volume (cc)	% Stem Used	Head Pressure (mmHg)	Head Pressure (psia)	Head Pressure (MPa)
.180	49.2	124	2.40	.0166
.185	50.5	121	2.35	.0162
.190	51.9	118	2.29	.0158
.195	53.3	115	2.23	.0154
.200	54.6	112	2.18	.0150
.205	56.0	109	2.12	.0146
.210	57.4	106	2.06	.0142
.215	58.7	103	2.01	.0138
.220	60.1	100	1.95	.0134
.225	61.5	97	1.89	.0130
.230	62.8	94	1.84	.0126
.235	64.2	91	1.78	.0123
.240	65.6	89	1.72	.0119
.245	66.9	86	1.66	.0115
.250	68.3	83	1.61	.0111
.255	69.7	80	1.55	.0107
.260	71.0	77	1.49	.0103
.265	72.4	74	1.44	.0099
.270	73.8	71	1.38	.0095
.275	75.1	68	1.32	.0091
.280	76.5	65	1.27	.0087
.285	77.9	62	1.21	.0083
.290	79.2	59	1.15	.0080
.295	80.6	56	1.10	.0076
.300	82.0	53	1.04	.0072
.305	83.3	50	.98	.0068
.310	84.7	47	.93	.0064
.315	86.1	44	.87	.0060
.320	87.4	42	.81	.0056
.325	88.8	39	.76	.0052
.330	90.2	36	.70	.0048
.335	91.5	33	.64	.0044
.340	92.9	30	.59	.0040
.345	94.3	27	.53	.0036
.350	95.6	24	.47	.0033
.355	97.0	21	.42	.0029
.360	98.4	18	.36	.0025
.365	99.7	15	.30	.0021
.366	100.0	15	.29	.0020

Mercury Head-Pressure Corrections for .387 cc Volume

Total Capillary Length (H):	242 mm
Measurable Capillary (Stem) Length (I):	227 mm
Capillary Diameter (D):	1.473 mm
Maximum Measurable Intrusion Volume:	.387 cc

Intrusion Volume (cc)	% Stem Used	Head Pressure		
		(mmHg)	(psia)	(MPa)
.000	.0	242	4.68	.0323
.004	1.0	239	4.63	.0319
.008	2.1	237	4.59	.0316
.012	3.1	234	4.54	.0313
.016	4.1	232	4.50	.0310
.020	5.2	230	4.45	.0307
.024	6.2	227	4.41	.0304
.028	7.2	225	4.36	.0301
.032	8.3	223	4.32	.0298
.036	9.3	220	4.27	.0294
.040	10.3	218	4.23	.0291
.044	11.4	216	4.18	.0288
.048	12.4	213	4.14	.0285
.052	13.4	211	4.09	.0282
.056	14.5	209	4.05	.0279
.060	15.5	206	4.00	.0276
.064	16.5	204	3.95	.0273
.068	17.6	202	3.91	.0269
.072	18.6	199	3.86	.0266
.076	19.6	197	3.82	.0263
.080	20.7	195	3.77	.0260
.084	21.7	192	3.73	.0257
.088	22.7	190	3.68	.0254
.092	23.8	188	3.64	.0251
.096	24.8	185	3.59	.0248
.100	25.8	183	3.55	.0244
.104	26.9	180	3.50	.0241
.108	27.9	178	3.46	.0238
.112	28.9	176	3.41	.0235
.116	30.0	173	3.36	.0232
.120	31.0	171	3.32	.0229
.124	32.0	169	3.27	.0226
.128	33.1	166	3.23	.0223
.132	34.1	164	3.18	.0219
.136	35.1	162	3.14	.0216
.140	36.2	159	3.09	.0213

Intrusion Volume (cc)	% Stem Used	Head Pressure (mmHg)	Head Pressure (psia)	Head Pressure (MPa)
.144	37.2	157	3.05	.0210
.148	38.2	155	3.00	.0207
.152	39.3	152	2.96	.0204
.156	40.3	150	2.91	.0201
.160	41.3	148	2.87	.0197
.164	42.4	145	2.82	.0194
.168	43.4	143	2.77	.0191
.172	44.4	141	2.73	.0188
.176	45.5	138	2.68	.0185
.180	46.5	136	2.64	.0182
.184	47.5	134	2.59	.0179
.188	48.6	131	2.55	.0176
.192	49.6	129	2.50	.0172
.196	50.6	127	2.46	.0169
.200	51.7	124	2.41	.0166
.204	52.7	122	2.37	.0163
.208	53.7	119	2.32	.0160
.212	54.8	117	2.28	.0157
.216	55.8	115	2.23	.0154
.220	56.8	112	2.18	.0151
.224	57.9	110	2.14	.0147
.228	58.9	108	2.09	.0144
.232	59.9	105	2.05	.0141
.236	61.0	103	2.00	.0138
.240	62.0	101	1.96	.0135
.244	63.0	98	1.91	.0132
.248	64.1	96	1.87	.0129
.252	65.1	94	1.82	.0126
.256	66.1	91	1.78	.0122
.260	67.2	89	1.73	.0119
.264	68.2	87	1.69	.0116
.268	69.3	84	1.64	.0113
.272	70.3	82	1.59	.0110
.276	71.3	80	1.55	.0107
.280	72.4	77	1.50	.0104
.284	73.4	75	1.46	.0101
.288	74.4	73	1.41	.0097
.292	75.5	70	1.37	.0094
.296	76.5	68	1.32	.0091
.300	77.5	66	1.28	.0088
.304	78.6	63	1.23	.0085

Intrusion Volume (cc)	% Stem Used	Head Pressure (mmHg)	Head Pressure (psia)	Head Pressure (MPa)
.308	79.6	61	1.19	.0082
.312	80.6	58	1.14	.0079
.316	81.7	56	1.10	.0076
.320	82.7	54	1.05	.0072
.324	83.7	51	1.00	.0069
.328	84.8	49	.96	.0066
.332	85.8	47	.91	.0063
.336	86.8	44	.87	.0060
.340	87.9	42	.82	.0057
.344	88.9	40	.78	.0054
.348	89.9	37	.73	.0050
.352	91.0	35	.69	.0047
.356	92.0	33	.64	.0044
.360	93.0	30	.60	.0041
.364	94.1	28	.55	.0038
.368	95.1	26	.51	.0035
.372	96.1	23	.46	.0032
.376	97.2	21	.41	.0029
.380	98.2	19	.37	.0025
.384	99.2	16	.32	.0022
.387	100.0	15	.29	.0020

Mercury Head-Pressure Corrections for 1.057 cc Volume

Total Capillary Length (H):	230 mm
Measurable Capillary (Stem) Length (I):	215 mm
Capillary Diameter (D):	2.502 mm
Maximum Measurable Intrusion Volume:	1.057 cc

Intrusion Volume (cc)	% Stem Used	Head Pressure		
		(mmHg)	(psia)	(MPa)
.000	.0	230	4.45	.0307
.010	.9	227	4.41	.0304
.020	1.9	225	4.37	.0301
.030	2.8	223	4.33	.0298
.040	3.8	221	4.29	.0296
.050	4.7	219	4.25	.0293
.060	5.7	217	4.21	.0290
.070	6.6	215	4.17	.0288
.080	7.6	213	4.13	.0285
.090	8.5	211	4.09	.0282
.100	9.5	209	4.05	.0279
.110	10.4	207	4.02	.0277
.120	11.4	205	3.98	.0274
.130	12.3	203	3.94	.0271
.140	13.2	201	3.90	.0269
.150	14.2	199	3.86	.0266
.160	15.1	197	3.82	.0263
.170	16.1	195	3.78	.0260
.180	17.0	193	3.74	.0258
.190	18.0	191	3.70	.0255
.200	18.9	189	3.66	.0252
.210	19.9	187	3.62	.0250
.220	20.8	185	3.58	.0247
.230	21.8	183	3.54	.0244
.240	22.7	181	3.50	.0242
.250	23.7	179	3.46	.0239
.260	24.6	177	3.43	.0236
.270	25.5	175	3.39	.0233
.280	26.5	173	3.35	.0231
.290	27.4	171	3.31	.0228
.300	28.4	168	3.27	.0225
.310	29.3	166	3.23	.0223
.320	30.3	164	3.19	.0220
.330	31.2	162	3.15	.0217
.340	32.2	160	3.11	.0214
.350	33.1	158	3.07	.0212

Intrusion Volume (cc)	% Stem Used	Head Pressure		
		(mmHg)	(psia)	(MPa)
.360	34.1	156	3.03	.0209
.370	35.0	154	2.99	.0206
.380	36.0	152	2.95	.0204
.390	36.9	150	2.91	.0201
.400	37.8	148	2.87	.0198
.410	38.8	146	2.84	.0195
.420	39.7	144	2.80	.0193
.430	40.7	142	2.76	.0190
.440	41.6	140	2.72	.0187
.450	42.6	138	2.68	.0185
.460	43.5	136	2.64	.0182
.470	44.5	134	2.60	.0179
.480	45.4	132	2.56	.0176
.490	46.4	130	2.52	.0174
.500	47.3	128	2.48	.0171
.510	48.2	126	2.44	.0168
.520	49.2	124	2.40	.0166
.530	50.1	122	2.36	.0163
.540	51.1	120	2.32	.0160
.550	52.0	118	2.28	.0157
.560	53.0	116	2.25	.0155
.570	53.9	114	2.21	.0152
.580	54.9	112	2.17	.0149
.590	55.8	109	2.13	.0147
.600	56.8	107	2.09	.0144
.610	57.7	105	2.05	.0141
.620	58.7	103	2.01	.0138
.630	59.6	101	1.97	.0136
.640	60.5	99	1.93	.0133
.650	61.5	97	1.89	.0130
.660	62.4	95	1.85	.0128
.670	63.4	93	1.81	.0125
.680	64.3	91	1.77	.0122
.690	65.3	89	1.73	.0120
.700	66.2	87	1.69	.0117
.710	67.2	85	1.66	.0114
.720	68.1	83	1.62	.0111
.730	69.1	81	1.58	.0109
.740	70.0	79	1.54	.0106
.750	71.0	77	1.50	.0103
.760	71.9	75	1.46	.0101

Intrusion Volume (cc)	% Stem Used	Head Pressure (mmHg)	(psia)	(MPa)
.770	72.8	73	1.42	.0098
.780	73.8	71	1.38	.0095
.790	74.7	69	1.34	.0092
.800	75.7	67	1.30	.0090
.810	76.6	65	1.26	.0087
.820	77.6	63	1.22	.0084
.830	78.5	61	1.18	.0082
.840	79.5	59	1.14	.0079
.850	80.4	57	1.10	.0076
.860	81.4	55	1.07	.0073
.870	82.3	53	1.03	.0071
.880	83.3	51	.99	.0068
.890	84.2	48	.95	.0065
.900	85.1	46	.91	.0063
.910	86.1	44	.87	.0060
.920	87.0	42	.83	.0057
.930	88.0	40	.79	.0054
.940	88.9	38	.75	.0052
.950	89.9	36	.71	.0049
.960	90.8	34	.67	.0046
.970	91.8	32	.63	.0044
.980	92.7	30	.59	.0041
.990	93.7	28	.55	.0038
1.000	94.6	26	.51	.0035
1.010	95.6	24	.47	.0033
1.020	96.5	22	.44	.0030
1.030	97.4	20	.40	.0027
1.040	98.4	18	.36	.0025
1.050	99.3	16	.32	.0022
1.057	100.0	15	.29	.0020

Mercury Head-Pressure Corrections for 1.116 cc Volume

Total Capillary Length (H):	242 mm
Measurable Capillary (Stem) Length (I):	227 mm
Capillary Diameter (D):	2.502 mm
Maximum Measurable Intrusion Volume:	1.116 cc

Intrusion Volume (cc)	% Stem Used	Head Pressure		
		(mmHg)	(psia)	(MPa)
.000	.0	242	4.68	.0323
.020	1.8	237	4.60	.0317
.040	3.6	233	4.52	.0312
.060	5.4	229	4.44	.0306
.080	7.2	225	4.37	.0301
.100	9.0	221	4.29	.0295
.120	10.8	217	4.21	.0290
.140	12.5	213	4.13	.0285
.160	14.3	209	4.05	.0279
.180	16.1	205	3.97	.0274
.200	17.9	201	3.89	.0268
.220	19.7	197	3.81	.0263
.240	21.5	193	3.74	.0258
.260	23.3	189	3.66	.0252
.280	25.1	185	3.58	.0247
.300	26.9	180	3.50	.0241
.320	28.7	176	3.42	.0236
.340	30.5	172	3.34	.0230
.360	32.3	168	3.26	.0235
.380	34.1	164	3.19	.0220
.400	35.8	160	3.11	.0214
.420	37.6	156	3.03	.0209
.440	39.4	152	2.95	.0203
.460	41.2	148	2.87	.0198
.480	43.0	144	2.79	.0192
.500	44.8	140	2.71	.0187
.520	46.6	136	2.63	.0182
.540	48.4	132	2.56	.0176
.560	50.2	128	2.48	.0171
.580	52.0	124	2.40	.0165
.600	53.8	119	2.32	.0160
.620	55.6	115	2.24	.0154
.640	57.3	111	2.16	.0149
.660	59.1	107	2.08	.0144
.680	60.9	103	2.01	.0138
.700	62.7	99	1.93	.0133

Intrusion Volume (cc)	% Stem Used	Head Pressure (mmHg)	Head Pressure (psia)	Head Pressure (MPa)
.720	64.5	95	1.85	.0127
.740	66.3	91	1.77	.0122
.760	68.1	87	1.69	.0117
.780	69.9	83	1.61	.0111
.800	71.7	79	1.53	.0106
.820	73.5	75	1.45	.0100
.840	75.3	71	1.38	.0095
.860	77.1	67	1.30	.0089
.880	78.9	63	1.22	.0084
.900	80.6	58	1.14	.0079
.920	82.4	54	1.06	.0073
.940	84.2	50	.98	.0068
.960	86.0	46	.90	.0062
.980	87.8	42	.83	.0057
1.000	89.6	38	.75	.0051
1.020	91.4	34	.67	.0046
1.040	93.2	30	.59	.0041
1.060	95.0	26	.51	.0035
1.080	96.8	22	.43	.0030
1.100	98.6	18	.35	.0024
1.116	100.0	15	.29	.0020

Mercury Head-Pressure Corrections for 1.716 cc Volume

Total Capillary Length (H):	230 mm
Measurable Capillary (Stem) Length (I):	215 mm
Capillary Diameter (D):	3.188 mm
Maximum Measurable Intrusion Volume:	1.716 cc

Intrusion Volume (cc)	% Stem Used	Head Pressure		
		(mmHg)	(psia)	(MPa)
.000	.0	230	4.45	.0307
.020	1.2	227	4.40	.0303
.040	2.3	224	4.35	.0300
.060	3.5	222	4.30	.0297
.080	4.7	219	4.25	.0293
.100	5.8	217	4.21	.0290
.120	7.0	214	4.16	.0287
.140	8.2	212	4.11	.0283
.160	9.3	209	4.06	.0280
.180	10.5	207	4.01	.0277
.200	11.7	204	3.96	.0273
.220	12.8	202	3.92	.0270
.240	14.0	199	3.87	.0267
.260	15.2	197	3.82	.0263
.280	16.3	194	3.77	.0260
.300	17.5	192	3.72	.0256
.320	18.6	189	3.67	.0253
.340	19.8	187	3.62	.0250
.360	21.0	184	3.58	.0246
.380	22.1	182	3.53	.0243
.400	23.3	179	3.48	.0240
.420	24.5	177	3.43	.0236
.440	25.6	174	3.38	.0233
.460	26.8	172	3.33	.0230
.480	28.0	169	3.29	.0226
.500	29.1	167	3.24	.0223
.520	30.3	164	3.19	.0220
.540	31.5	162	3.14	.0216
.560	32.6	159	3.09	.0213
.580	33.8	157	3.04	.0210
.600	35.0	154	2.99	.0206
.620	36.1	152	2.95	.0203
.640	37.3	149	2.90	.0200
.660	38.5	147	2.85	.0196
.680	39.6	144	2.80	.0193
.700	40.8	142	2.75	.0190

Intrusion Volume (cc)	% Stem Used	Head Pressure (mmHg)	Head Pressure (psia)	Head Pressure (MPa)
.720	42.0	139	2.70	.0186
.740	43.1	137	2.66	.0103
.760	44.3	134	2.61	.0180
.780	45.5	132	2.56	.0176
.800	46.6	129	2.51	.0173
.820	47.8	127	2.46	.0170
.840	49.0	124	2.41	.0166
.860	50.1	122	2.36	.0163
.880	51.3	119	2.32	.0160
.900	52.4	117	2.27	.0156
.920	53.6	114	2.22	.0153
.940	54.8	112	2.17	.0150
.960	55.9	109	2.12	.0146
.980	57.1	107	2.07	.0143
1.000	58.3	104	2.03	.0140
1.020	59.4	102	1.98	.0136
1.040	60.6	99	1.93	.0133
1.060	61.8	97	1.88	.0130
1.080	62.9	94	1.83	.0126
1.100	64.1	92	1.78	.0123
1.120	65.3	89	1.73	.0120
1.140	66.4	87	1.69	.0116
1.160	67.6	84	1.64	.0113
1.180	68.8	82	1.59	.0110
1.200	69.9	79	1.54	.0106
1.220	71.1	77	1.49	.0103
1.240	72.3	74	1.44	.0099
1.260	73.4	72	1.40	.0096
1.280	74.6	69	1.35	.0093
1.300	75.8	67	1.30	.0089
1.320	76.9	64	1.25	.0086
1.340	78.1	62	1.20	.0083
1.360	79.3	59	1.15	.0079
1.380	80.4	57	1.10	.0076
1.400	81.6	54	1.06	.0073
1.420	82.8	52	1.01	.0069
1.440	83.9	49	.96	.0066
1.460	85.1	47	.91	.0063
1.480	86.2	44	.86	.0059
1.500	87.4	42	.81	.0056
1.520	88.6	39	.77	.0053

Intrusion Volume (cc)	% Stem Used	Head Pressure		
		(mmHg)	(psia)	(MPa)
1.540	89.7	37	.72	.0049
1.560	90.9	34	.67	.0046
1.580	92.1	32	.62	.0043
1.600	93.2	29	.57	.0039
1.620	94.4	27	.52	.0036
1.640	95.6	24	.47	.0033
1.660	96.7	22	.43	.0029
1.680	97.9	19	.38	.0026
1.700	99.1	17	.33	.0023
1.716	100.0	15	.29	.0020

Mercury Head-Pressure Corrections for 3.007 cc Volume

Total Capillary Length (H):	230 mm
Measurable Capillary (Stem) Length (I):	212 mm
Capillary Diameter (D):	4.25 mm
Maximum Measurable Intrusion Volume:	3.007 cc

Intrusion Volume (cc)	% Stem Used	Head Pressure (mmHg)	(psia)	(MPa)
.000	.0	230	4.45	.0307
.040	1.3	227	4.39	.0303
.080	2.7	224	4.34	.0299
.120	4.0	221	4.28	.0295
.160	5.3	218	4.23	.0292
.200	6.7	215	4.18	.0288
.240	8.0	213	4.12	.0284
.280	9.3	210	4.07	.0280
.320	10.6	207	4.01	.0277
.360	12.0	204	3.96	.0273
.400	13.3	201	3.90	.0269
.440	14.6	198	3.85	.0265
.480	16.0	196	3.79	.0261
.520	17.3	193	3.74	.0258
.560	18.6	190	3.68	.0254
.600	20.0	187	3.63	.0250
.640	21.3	184	3.58	.0246
.680	22.6	182	3.52	.0243
.720	23.9	179	3.47	.0239
.760	25.3	176	3.41	.0235
.800	26.6	173	3.36	.0231
.840	27.9	170	3.30	.0228
.880	29.3	167	3.25	.0224
.920	30.6	165	3.19	.0220
.960	31.9	162	3.14	.0216
1.000	33.3	159	3.08	.0213
1.040	34.6	156	3.03	.0209
1.080	35.9	153	2.98	.0205
1.120	37.2	151	2.92	.0201
1.160	38.6	148	2.87	.0198
1.200	39.9	145	2.81	.0194
1.240	41.2	142	2.76	.0190
1.280	42.6	139	2.70	.0186
1.320	43.9	136	2.65	.0183
1.360	45.2	134	2.59	.0179
1.400	46.6	131	2.54	.0175

Intrusion Volume (cc)	% Stem Used	Head Pressure (mmHg)	Head Pressure (psia)	Head Pressure (MPa)
1.440	47.9	128	2.48	.0171
1.480	49.2	125	2.43	.0168
1.520	50.5	122	2.38	.0164
1.560	51.9	120	2.32	.0160
1.600	53.2	117	2.27	.0156
1.640	54.5	114	2.21	.0152
1.680	55.9	111	2.16	.0149
1.720	57.2	108	2.10	.0145
1.760	58.5	105	2.05	.0141
1.800	59.9	103	1.99	.0137
1.840	61.2	100	1.94	.0134
1.880	62.5	97	1.88	.0130
1.920	63.9	94	1.83	.0126
1.960	65.2	91	1.78	.0122
2.000	66.5	88	1.72	.0119
2.040	67.8	86	1.67	.0115
2.080	69.2	83	1.61	.0111
2.120	70.5	80	1.56	.0107
2.160	71.8	77	1.50	.0104
2.200	73.2	74	1.45	.0100
2.240	74.5	72	1.39	.0096
2.280	75.8	69	1.34	.0092
2.320	77.2	66	1.28	.0089
2.360	78.5	63	1.23	.0085
2.400	79.8	60	1.18	.0081
2.440	81.1	57	1.12	.0077
2.480	82.5	55	1.07	.0074
2.520	83.8	52	1.01	.0070
2.560	85.1	49	.96	.0066
2.600	86.5	46	.90	.0062
2.640	87.8	43	.85	.0058
2.680	89.1	41	.79	.0055
2.720	90.5	38	.74	.0051
2.760	91.8	35	.68	.0047
2.800	93.1	32	.63	.0043
2.840	94.4	29	.58	.0040
2.880	95.8	26	.52	.0036
2.920	97.1	24	.47	.0032
2.960	98.4	21	.41	.0028
3.000	99.8	18	.36	.0025
3.007	100.0	18	.35	.0024

Mercury Head-Pressure Corrections for 3.857 cc Volume

Total Capillary Length (H):	230 mm
Measurable Capillary (Stem) Length (I):	212 mm
Capillary Diameter (D):	4.813 mm
Maximum Measurable Intrusion Volume:	3.857 cc

Intrusion Volume (cc)	% Stem Used	Head Pressure		
		(mmHg)	(psia)	(MPa)
.000	.0	230	4.45	.0307
.040	1.0	227	4.41	.0304
.080	2.1	225	4.36	.0301
.120	3.1	223	4.32	.0298
.160	4.1	221	4.28	.0295
.200	5.2	219	4.24	.0292
.240	6.2	216	4.19	.0289
.280	7.3	214	4.15	.0286
.320	8.3	212	4.11	.0283
.360	9.3	210	4.07	.0280
.400	10.4	208	4.02	.0277
.440	11.4	205	3.98	.0274
.480	12.4	203	3.94	.0271
.520	13.5	201	3.90	.0268
.560	14.5	199	3.85	.0266
.600	15.6	197	3.81	.0263
.640	16.6	194	3.77	.0260
.680	17.6	192	3.73	.0257
.720	18.7	190	3.68	.0254
.760	19.7	188	3.64	.0251
.800	20.7	186	3.60	.0248
.840	21.8	183	3.56	.0245
.880	22.8	181	3.51	.0242
.920	23.9	179	3.47	.0239
.960	24.9	177	3.43	.0236
1.000	25.9	175	3.39	.0233
1.040	27.0	172	3.34	.0230
1.080	28.0	170	3.30	.0227
1.120	29.0	168	3.26	.0225
1.160	30.1	166	3.22	.0222
1.200	31.1	164	3.17	.0219
1.240	32.1	161	3.13	.0216
1.280	33.2	159	3.09	.0213
1.320	34.2	157	3.05	.0210
1.360	35.3	155	3.00	.0207
1.400	36.3	153	2.96	.0204

Intrusion Volume (cc)	% Stem Used	Head Pressure (mmHg)	(psia)	(MPa)
1.440	37.3	150	2.92	.0201
1.480	38.4	148	2.87	.0198
1.520	39.4	146	2.83	.0195
1.560	40.4	144	2.79	.0192
1.600	41.5	142	2.75	.0189
1.640	42.5	139	2.70	.0186
1.680	43.6	137	2.66	.0183
1.720	44.6	135	2.62	.0181
1.760	45.6	133	2.58	.0178
1.800	46.7	131	2.53	.0175
1.840	47.7	128	2.49	.0172
1.880	48.7	126	2.45	.0169
1.920	49.8	124	2.41	.0166
1.960	50.8	122	2.36	.0163
2.000	51.9	120	2.32	.0160
2.040	52.9	117	2.28	.0157
2.080	53.9	115	2.24	.0154
2.120	55.0	113	2.19	.0151
2.160	56.0	111	2.15	.0148
2.200	57.0	109	2.11	.0145
2.240	58.1	106	2.07	.0142
2.280	59.1	104	2.02	.0140
2.320	60.2	102	1.98	.0137
2.360	61.2	100	1.94	.0134
2.400	62.2	98	1.90	.0131
2.440	63.3	95	1.85	.0128
2.480	64.3	93	1.81	.0125
2.520	65.3	91	1.77	.0122
2.560	66.4	89	1.73	.0119
2.600	67.4	87	1.68	.0116
2.640	68.4	84	1.64	.0113
2.680	69.5	82	1.60	.0110
2.720	70.5	80	1.56	.0107
2.760	71.6	78	1.51	.0104
2.800	72.6	76	1.47	.0101
2.840	73.6	73	1.43	.0099
2.880	74.7	71	1.39	.0096
2.920	75.7	69	1.34	.0093
2.960	76.7	67	1.30	.0090
3.000	77.8	65	1.26	.0087
3.040	78.8	62	1.22	.0084

Intrusion Volume (cc)	% Stem Used	Head Pressure		
		(mmHg)	(psia)	(MPa)
3.080	79.9	60	1.17	.0081
3.120	80.9	58	1.13	.0078
3.160	81.9	56	1.09	.0075
3.200	83.0	54	1.05	.0072
3.240	84.0	51	1.00	.0069
3.280	85.0	49	.96	.0066
3.320	86.1	47	.92	.0063
3.360	87.1	45	.88	.0060
3.400	88.2	43	.83	.0057
3.440	89.2	40	.79	.0055
3.480	90.2	38	.75	.0052
3.520	91.3	36	.71	.0049
3.560	92.3	34	.66	.0046
3.600	93.3	32	.62	.0043
3.640	94.4	29	.58	.0040
3.680	95.4	27	.54	.0037
3.720	96.4	25	.49	.0034
3.760	97.5	23	.45	.0031
3.800	98.5	21	.41	.0028
3.840	99.6	18	.37	.0025
3.857	100.0	18	.35	.0024

APPENDIX E

COMPRESSIBILITY EFFECT

COMPRESSIBILITY EFFECT

Mercury, glass, and the material being tested for porosity are compressed to different degrees under elevated pressures. In addition, the dielectric constant of the hydraulic fluid shifts slightly with pressure. The combined effects of the mercury, glass, and the fluid counteract one another to a large extent in the PoreSizer 9320 as designed. Their effect could be reduced essentially to zero for a sample of specified volume and compressibility, but doing so is not justified since sample properties are variables.

Consideration of second-order compressibility effects is rarely justified except where porosity is low and high precision is required. A desirable way to proceed if conditions can be met is to make a test with a nonporous sample of the same size and composition as the material of low porosity. The apparent penetration generated under this circumstance can then be subtracted at comparable pressures from the penetration data with actual pores to yield only pore penetration results.

Alternately when sample compressibility is known or can be determined, a test can be made with any nonporous sample of the same volume, perhaps steel or glass, also having a known compressibility. The resulting penetration data can then be adjusted in accordance with the ratio of the two compressibilities.

Care should be exercised in making either of these tests that temperature effects are not allowed to compromise the results. Pressure generation is inevitably accompanied by an increase in temperature. Either wait a sufficient time between pressure steps for temperature to equilibrate or generate pressures at the same rate in both parts of the test to make temperature increases comparable.

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. One 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 PoreSizer 9320 will automatically add a pore spectrum point anytime 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 250 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

FORMAT OF UNREDUCED REPORTS

FORMAT OF UNREDUCED REPORTS

The following information details the format and meaning of data in unreduced reports. Each record is terminated by a carriage return and line feed. All character strings are delimited with double quotation marks

Record Number	Information Conveyed	Form
1	Sample run status. 0 = no runs started 1 = LP run in progress 2 = LP run completed 3 = HP run in progress 4 = HP run completed	1 numeric char.
2	Sample ID	41 characters
3	Submitter ID	41 characters
4	Operator ID	21 characters
5	Sample weight	1 floating pt.
6	Sample + penetrometer + Hg weight	1 floating pt.
7	Reserved	25 characters
8	Penetrometer number	11 characters
9	Penetrometer constant	1 floating pt.
10	Penetrometer weight	1 floating pt.
11	Penetrometer volume	1 floating pt.
12	Stem volume	1 floating pt.
13	Maximum head pressure	1 floating pt.
14	Advancing contact angle	1 floating pt.
15	Receding contact angle	1 floating pt.
16	Mercury surface tension	1 floating pt.
17	Mercury density	1 floating pt.
18	Reserved	9 characters
19	Reserved	1 integer
20	Reserved	41 characters
21	Reserved	1 floating pt.
22	Reserved	1 integer
23	Reserved	1 floating pt.
24	Reserved	1 numeric char.
25	Reserved	1 integer
26	Reserved	1 floating pt.
27	High pressure maximum intrusion volume	1 floating pt.
28	Reserved	1 numeric char.
29	High pressure equilibration time	1 integer
30	High pressure intrusion rate	1 floating pt.
31	Reserved	1 integer
32	Reserved	25 characters
33	Run type 0 = automatic 1 = manual	1 numeric char.

Record Number	Information Conveyed	Form
34	Run method 0 = equilibrated 1 = rate sequence	1 numeric char.
35	Report ID	25 characters
36	Report negative intrusion? 0 = Yes 1 = No	1 numeric char.
37	Report title	51 characters
38	Summary page? 0 = Yes 1 = No	1 numeric char.
39	Calculation range specified as 0 = Pressure 1 = Size	1 numeric char.
40	Calculation range from	1 floating pt.
41	Calculation range to	1 floating pt.
42	Tabular data page? 0 = Yes 1 = No	1 numeric char.
43	Tabular data defined by 0 = Collected points 1 = Table	1 numeric char.
44	Table number	1 integer
45	Heading, column 1	1 numeric char.
46	Heading, column 2	1 numeric char.
47	Heading, column 3	1 numeric char.
48	Heading, column 4	1 numeric char.
49	Heading, column 5	1 numeric char.
50	Heading, column 6 0 = None 1 = Pressure 2 = Size 3 = Mean size 4 = Cumulative intrusion 5 = Incremental intrusion 6 = Differential intrusion 7 = Log differential intrusion 8 = Cumulative pore area 9 = Incremental pore area 10 = Percent total intrusion	1 numeric char.
51	Number of graph pages (0 - 5)	1 integer
52	X-axis choice 0 = Pressure 1 = Size	1 numeric char.
53	X-axis scale 0 = Log 1 = Normal	1 numeric char.
54	X-axis range from	1 floating pt.
55	X-axis range to	1 floating pt.

Record Number	Information Conveyed	Form
56	Y-axis choice 0 = Cumulative intrusion 1 = Incremental intrusion 2 = Differential intrusion 3 = Log differential intrusion 4 = Cumulative pore area 5 = Incremental pore area	1 numeric char.
57	Y-axis range from	1 floating pt.
58	Y-axis range to	1 floating pt.
59	Type of data to plot 0 = Collected data 1 = Continuous curve 2 = Both	1 numeric char.
60	Intrusion data to plot 0 = None 1 = First 2 = All	1 numeric char.
61	Extrusion data to plot 0 = None 1 = First 2 = All	1 numeric char.
62	X-axis choice 0 = Pressure 1 = Size	1 numeric char
63	X-axis scale 0 = Log 1 = Normal	1 numeric char.
64	X-axis range from	1 floating pt.
65	X-axis range to	1 floating pt.
66	Y-axis choice 0 = Cumulative intrusion 1 = Incremental intrusion 2 = Differential intrusion 3 = Log differential intrusion 4 = Cumulative pore area 5 = Incremental pore area	1 numeric char.
67	Y-axis range from	1 floating pt.
68	Y-axis range to	1 floating pt.
69	Type of data to plot 0 = Collected data 1 = Continuous curve 2 = Both	1 numeric char.
70	Intrusion data to plot 0 = None 1 = First 2 = All	1 numeric char.
71	Extrusion data to plot 0 = None 1 = First 2 = All	1 numeric char.

Record Number	Information Conveyed	Form
72	X-axis choice 0 = Pressure 1 = Size	1 numeric char.
73	X-axis scale 0 = Log 1 = Normal	1 numeric char.
74	X-axis range from	1 floating pt.
75	X-axis range to	1 floating pt.
76	Y-axis choice 0 = Cumulative intrusion 1 = Incremental intrusion 2 = Differential intrusion 3 = Log differential intrusion 4 = Cumulative pore area 5 = Incremental pore area	1 numeric char.
77	Y-axis range from	1 floating pt.
78	Y-axis range to	1 floating pt.
79	Type of data to plot 0 = Collected data 1 = First 2 = Both	1 numeric char.
80	Intrusion data to plot 0 = None 1 = First 2 = All	1 numeric char.
81	Extrusion data to plot 0 = None 1 = First 2 = All	1 numeric char.
82	X-axis choice 0 = Pressure 1 = Size	1 numeric char.
83	X-axis scale 0 = Log 1 = Normal	1 numeric char.
84	X-axis range from	1 floating pt.
85	X-axis range to	1 floating pt.
86	Y-axis choice 0 = Cumulative intrusion 1 = Incremental intrusion 2 = Differential intrusion 3 = Log differential intrusion 4 = Cumulative pore area 5 = Incremental pore area	1 numeric char.
87	Y-axis range from	1 floating pt.
88	Y-axis range to	1 floating pt.
89	Type of data to plot 0 = Collected data 1 = Continuous curve 2 = Both	1 numeric char.

Record Number	Information Conveyed	Form
90	Intrusion data to plot 0 = None 1 = First 2 = All	1 numeric char.
91	Extrusion data to plot 0 = None 1 = First 2 = All	1 numeric char.
92	X-axis choice 0 = Pressure 1 = Size	1 numeric char.
93	X-axis scale 0 = Log 1 = Normal	1 numeric char.
94	X-axis range from	1 floating pt.
95	X-axis range to	1 floating pt.
96	Y-axis choice 0 = Cumulative intrusion 1 = Incremental intrusion 2 = Differential intrusion 3 = Log differential intrusion 4 = Cumulative pore area 5 = Incremental pore area	1 numeric char.
97	Y-axis range from	1 floating pt.
98	Y-axis range to	1 floating pt.
99	Type of data to plot 0 = Collected data 1 = Continuous curve 2 = Both	1 numeric char.
100	Intrusion data to plot 0 = None 1 = First 2 = All	1 numeric char.
101	Extrusion data to plot 0 = None 1 = First 2 = All	1 numeric char.
102	Low pressure run date	9 characters
103	Low pressure run time	9 characters
104	High pressure run date	9 characters
105	High pressure run time	9 characters
106	Number of data points taken so far	1 integer

The matching point between low and high pressure analyses is indicated by expressing the pressure datum as a negative number.

107	Pressure	1 floating pt.
108	Intrusion volume	1 floating pt.
109	Pressure	1 floating pt.

Record Number	Information Conveyed	Form
110	Intrusion volume	1 floating pt.
.		
.		
.		
.		
605	Pressure	1 floating pt.
606	Intrusion volume	1 floating pt.

APPENDIX H

MANUAL DATA REDUCTION

MANUAL DATA REDUCTION

Mercury porosimetry measurements permit calculation of material densities, pore volumes, pore diameters, pore wall surface areas, and the change of any one of the latter parameters with change in any other. Formalized procedures and tabular aids are provided in the following sections for simplifying these computations and presenting the results without the use of the control module.

NOTE

Blank forms for computations and recording data are included in Appendix A. Plotting sheets for Cumulative Pore Size Distribution are supplied with the PoreSizer. Additional plotting sheets may be ordered from Micromeritics.

Density

Sample density determination requires, first, that penetrometer volume be accurately known. Figure H-1 shows an example of this evaluation.

Figure H-2 illustrates the calculation of both bulk and apparent (absolute if there are no closed or unintruded pores) densities. Space is also provided for one determination of penetrometer volume. This form is convenient for record keeping even if density information is not desired and these calculations are never completed.

The calculations cannot be completed until pressure testing is finished. Item 7, however, must be determined before testing is begun, and item 6 must be established between the low pressure and high pressure testing. Item 13 is obtained from the high pressure data.

Pore Volume and Diameter

The basic form for recording mercury intrusion (and extrusion) data is Figure H-3, Pore Size Distribution Data. On it are entered directly both pressure and intrusion indications under the columns headed "Pressure Reading" and "Intrusion Reading." The pressure indicated by the digital readout is pounds (force) per square inch absolute or, if desired, megapascals, and the intrusion indication is picofarads.

Converting intrusion meter readings to pore volumes requires, first, calculating cumulative changes in capacitance (initial value taken as zero). These changes in capacitances are then multiplied by the conversion factor (penetrometer constant) supplied for the penetrometer (and a units conversion factor) to give the cumulative pore volume. Cumulative pore volumes per gram of sample are obtained by dividing by the weight of the sample.

When the sample is pressurized in the filling device, the penetrometer is horizontal, and, practically, there is no need to make allowance for a head pressure of mercury. The penetrometer is mounted vertically for high pressure measurement, however, and the weight of the mercury head adds to the indicated pressure reading. Initially, the head pressure can amount to as much as 4.7 psia, but it decreases as mercury intrudes. The exact head pressure contribution is determined by taking the appropriate head value from Table D-2 in Appendix D corresponding to penetrometer stem length and the cumulative pore volume (cc) calculated from the intrusion data.

PENETROMETER VOLUME CALIBRATION

Penetrometer: 3cc Solid, 0.412cc capacity Date: 17 July 1989
Number: 14 By: LP

First Calibration of Penetrometer Volume:

1. Weight of penetrometer filled with mercury . . . . 121.6948 g
2. Weight of sealed, empty penetrometer . . . . 70.5174 g
3. Weight of mercury (No. 1 minus No. 2) . . . . 51.1774 g
   Room temperature = 22 °C
4. Volume of penetrometer (No. 3 divided by Density of Mercury*) . . . . 3.7794 cc

Second Calibration of Penetrometer volume:

1. Weight of penetrometer filled with mercury . . . . 120.8541 g
2. Weight of sealed, empty penetrometer . . . . 69.7167 g
3. Weight of mercury (No. 1 minus No. 2) . . . . 51.1374 g
   Room temperature = 22 °C
4. Volume of penetrometer (No. 3 divided by Density of Mercury*) . . . . 3.7764 cc

Third Calibration of Penetrometer Volume:

1. Weight of penetrometer filled with mercury . . . . 120.6116 g
2. Weight of sealed, empty penetrometer . . . . 69.4900 g
3. Weight of mercury (No. 1 minus No. 2) . . . . 51.1216 g
   Room temperature = 21 °C
4. Volume of penetrometer (No. 3 divided by Density of Mercury*) . . . . 3.7745 cc

Average Volume of Penetrometer ($\overline{V}$) . . . . 3.7768 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: ______________________________

micromeritics®

Figure H-1. Penetrometer Volume Calibration Form (completed sample)

DENSITY DETERMINATION BY MERCURY POROSIMETRY

Sample: Silica Alumina Catalyst Support

Date: 17 July 1989

By: LP

Calibration of Penetrometer Volume:

1. Weight of penetrometer filled with mercury . . . . 120.8990 g
2. Weight of sealed, empty penetrometer . . . . 69.7857 g
3. Weight of mercury (No. 1 minus No. 2) . . . . 51.1133 g
   Room temperature = 25.2 °C
4. Volume of penetrometer (No. 3 divided by Density of Mercury*) . . . . 3.7768 cc

Bulk Density Calculation:

5. Weight of penetrometer and sample . . . . 70.2923 g
6. Weight of penetrometer, sample and mercury . . . . 114.9058 g
7. Weight of sample (No. 5 minus No. 2) . . . . 0.5066 g
8. Weight of mercury (No. 6 minus No. 5) . . . . 44.6135 g
   Room temperature = 25.2 °C
9. Volume of mercury (No. 8 divided by Density of Mercury*) . . . . 3.2965 cc
10. Volume of sample (No. 4 minus No. 9) . . . . 0.4803 cc
11. Bulk volume of sample (No. 10 divided by No. 7) . . . . 0.9481 cc/g
12. Bulk density of sample (reciprocal of No. 11) . . . . 1.0548 g/cc

Apparent Density Calculation:

13. Pore volume, mercury displaced in penetrometer stem . . . . 0.1766 cc
    at a maximum pressure of . . . . 29,867 psia
14. Volume of sample, less pore volume (No. 10 minus No. 13) . . . . 0.3037 cc
15. Specific volume of sample (No. 14 divided by No. 7) . . . . 0.5995 cc/g
16. Apparent density of sample (reciprocal of No. 15) . . . . 1.6681 g/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: ___

micromeritics®

Figure H-2. Density Determination by Mercury Porosimetry Form (completed sample)

PORE SIZE DISTRIBUTION DATA

Sample: Silica-Alumina Catalyst Support Date: 17 July 1989

Sample Weight: 0.5066 g Penetrometer No: 14 By: LP

Penetrometer Volume: 3.7768 cc, Penetrometer Constant: 10.79 µL/pF

Mercury Surface Tension: 485 dynes/cm, Contact Angle: 130 ° Sheet 1 of 3

PRESSURE ☒ psia or ☐ MPa			PORE SIZE ☒ Diameter ☐ Radius	INTRUSION			
Mercury Head Correction	Pressure Reading	Corrected Pressure	(µm)	Intrusion Reading (pF)	Cumulative Intrusion (ΣΔpF)	Cumulative Pore Volume (cc)	Cumulative Pore Volume (cc/g)
N/A	1.917	1.917	94.3	40.95	—	—	—
N/A	2.160	2.160	83.7	40.95	0.00	0.0000	0.0000
N/A	3.150	3.150	57.4	40.95	0.00	0.0000	0.0000
~~N/A~~	4.077	4.077	44.4	40.95	0.00	0.0000	0.0000
N/A	5.598	5.598	32.3	40.83	0.12	0.0013	0.0026
N/A	7.254	7.254	24.9	40.83	0.12	0.0013	0.0026
N/A	8.649	8.649	20.9	40.83	0.12	0.0013	0.0026
N/A	11.1	11.1	16.3	40.76	0.19	0.0021	0.0040
N/A	13.4	13.4	13.5	40.71	0.24	0.0026	0.0051
N/A	16.7	16.7	10.8	40.71	0.24	0.0026	0.0051
N/A	18.9	18.9	9.57	40.71	0.24	0.0026	0.0051
N/A	20.5	20.5	8.82	40.64	0.31	0.0033	0.0066
N/A	23.8	23.8	7.60	40.64	0.31	0.0033	0.0066
N/A	25.6	25.6	7.06	40.64	0.31	0.0033	0.0066
	Transfer sample to high pressure chamber						
4.64	21.0	25.6	7.06	43.43	0.00/0.31	0.0033	0.0066
4.63	30.0	34.6	5.23	43.37	0.06/0.37	0.0040	0.0079
4.63	40.0	44.6	4.06	43.37	0.06/0.37	0.0040	0.0079
4.63	50.0	54.6	3.31	43.37	0.06/0.37	0.0040	0.0079
4.62	60.0	64.6	2.80	43.25	0.18/0.49	0.0053	0.0104
4.61	75.0	79.6	2.27	43.13	0.30/0.61	0.0066	0.0130
4.59	91.0	95.6	1.89	43.01	0.42/0.73	0.0079	0.0155

micromeritics

Figure H-3. Pore Size Distribution Data Form (completed sample)

The true pressure is found when the mercury head correction is added to the pressure reading.

Calculating the pore diameter intruded by mercury at each pressure requires solving the basic equation (refer to Appendix B).

$$D = \frac{-4\gamma\cos\theta}{P}$$

where

D = the diameter,
γ = the surface tension,
θ = the contact angle, and
P = the pressure.

Presently, the generally accepted value for γ is 485 dynes/cm. The contact angle generally varies around 130 degrees for which the cosine is -0.6428. The true angle depends on the material being examined, and this true value should be employed. For purposes of illustration, 130 degrees will be assumed here. Pore diameters are usually desired in units of micrometers (μm), and here pressures are measured in pounds per square inch. This use of a variety of units requires inclusion of conversion factors.

The final result is that diameters are calculated by

$$D\,\mu\text{m} = \frac{-4 \times 485 \frac{\text{dynes}}{\text{cm}} \times 10^4 \frac{\mu\text{m}}{\text{cm}} \times -0.6428}{P\,\text{psia} \times 6.8948 \times 10^4 \frac{\text{dynes}}{\text{cm psia}}}$$

which, in this illustration using a contact angle of 130 degrees, reduces to

$$D\,\mu\text{m} = \frac{180\,\mu\text{m psia}}{P\,\text{psia}}$$

Plotting calculated cumulative pore volumes vs. calculated pore diameters gives the convenient form of data presentation illustrated by Figure H-4.

Often it is desired to present mercury intrusion data in a differential form as an alternative to a cumulative plot. There are several choices for obtaining a derivative plot, or differentiating the cumulative curve: DV/DD ($\Delta V \div \Delta D$), DV/DlogD ($\Delta V \div \Delta \log D$), or just the incremental volume different (DV or ΔV) plotted vs. mean pore diameter (D). DV is easy to obtain by differences between adjacent cumulative volume points on the tabulation.

Mean pore diameter is the diameter value halfway between the two points, and is obtained by adding them together and dividing by two, for example, ($D_1 + D_2/2$), etc.

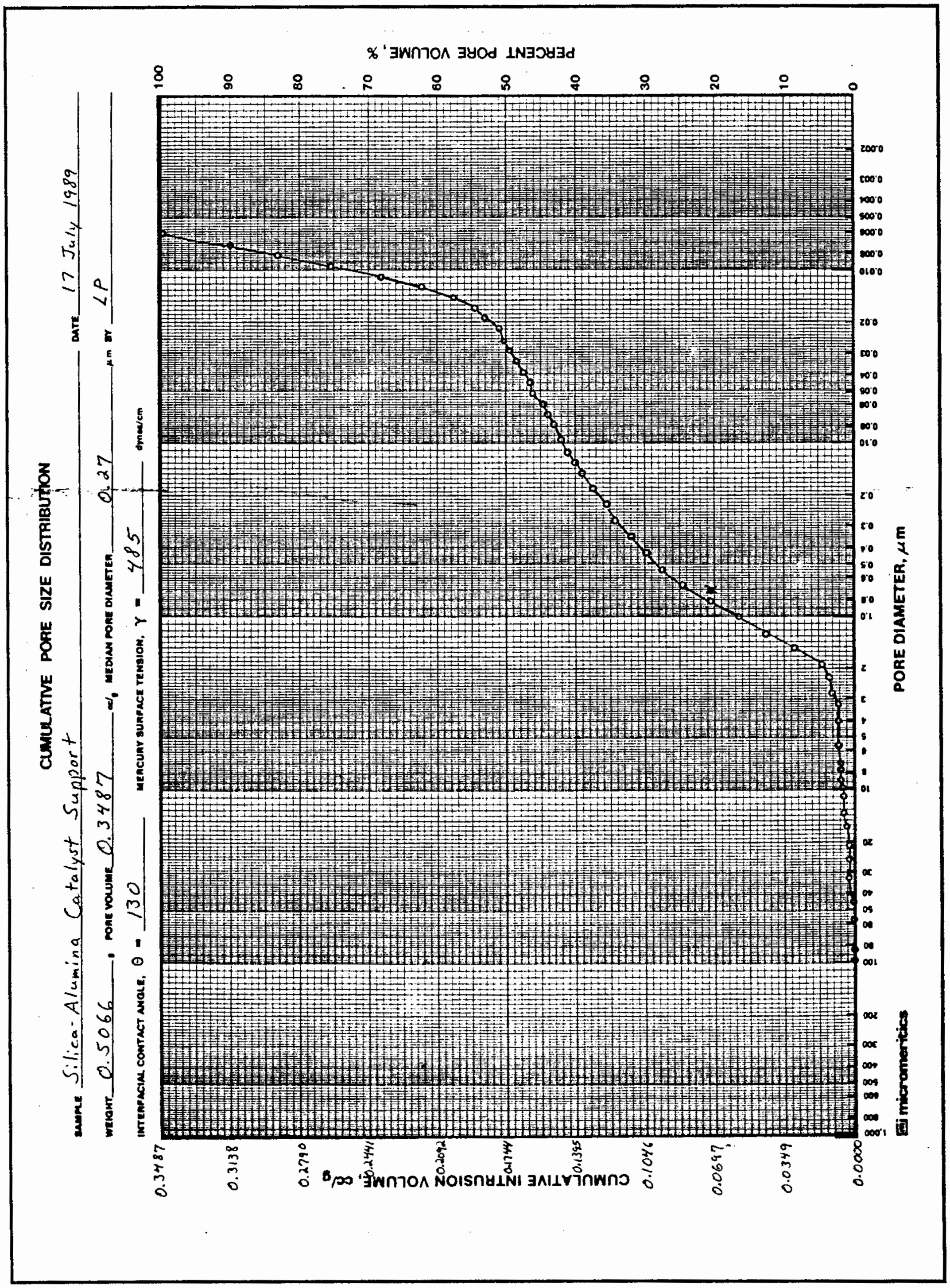


Figure H-4. Cumulative Pore Size Distribution Graph (plotted sample)

Pore Surface Area

The total pore surface area can be obtained by assuming that all the pores are cylindrical capillaries. Then the pore surface area (A) for each diameter increment is simply related to incremental pore volume (V) and the average pore diameter (D) by the equation

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

The cumulative surface area for each point is the sum of these for all preceding points. Cumulative surface area can also be calculated by integrating the cumulative pore volume distribution (PdV) curve. This calculation is based on the PV work done (dW) on the system when mercury is intruded into the pores (see Appendix I).

The basic equality is

$$dW = \gamma \cos\theta \, dA = -PdV$$

The required integration of cumulative pore volume data is performed over the pressure range where all the pores are filled as shown in Figure H-4. Basically, this solves the expression

$$\text{Pore Surface Area: } A = \frac{0.0221}{m} \int_{V_{min}}^{V_{max}} PdV \ \ m^2/g$$

Applied to discreet data points, this reduces to

$$A = \frac{0.0221}{m} \sum_{i=1}^{max} \bar{P}(i)\, V(i)$$

where $\bar{P}(i)$ is the mean pressure for interval (i) and V(i) is the incremental pore volume for (i).

When pressure is measured in pounds per square inch, volume in cubic centimeters, and sample weight (m) in grams, the surface area will be in square meters per gram. See Appendix I for further details.

For calculating the pore area distribution (see Figure H-5), record the actual intrusion data taken from columns 3 and 8 of the Pore Size Distribution Data form in columns 1 and 3, respectively, as absolute pressure (P) (psia) and cumulative pore volume (V) (cc/g). Since all intrusion volumes are already normalized on a per unit of mass basis (cc/g), there will be no need to divide the areas by the sample weight (m) later.

Going across the page one step at a time, average pressure ($\bar{P}$) which is half way between two points is calculated by adding the two pressure readings (P_1 + P_2) and dividing by two. Thus

$$\bar{P} = \frac{P_n + P_{n+1}}{2}$$

Record these $\bar{P}$ values in column 2.

PORE AREA DISTRIBUTION DATA

Sample: Silica-Alumina Catalyst Support Date: ________
Sample Weight: 0.5066 g Penetrometer No: 14 By: ________
Penetrometer Volume: 3.7768 cc, Penetrometer Constant: 10.79 µL/pF
Mercury Surface Tension: 485 dynes/cm, Contact Angle: 130 ° Sheet 1 of 3

PRESSURE ☒ psia or ☐ MPa		INTRUSION VOLUME		PORE SIZE ☒ Diameter ☐ Radius (µm)	PORE AREA	
Corrected Pressure	Average Pressure	Cumulative (cc/g)	Incremental (cc/g)		Incremental (m^2/g)	Cumulative (m^2/g)
1.917	—	—	—	—	—	—
2.160	2.039	0.0000	0.0000	88.7	0.00000	0.00000
3.150	2.655	0.0000	0.0000	68.1	0.00000	0.00000
4.077	3.614	0.0000	0.0000	50.1	0.00000	0.00000
5.598	4.838	0.0026	0.0026	37.4	0.00028	0.00028
7.254	6.426	0.0026	0.0000	28.1	0.00000	0.00028
8.649	7.952	0.0026	0.0000	22.7	0.00000	0.00028
11.1	9.9	0.0040	0.0014	18.3	0.00031	0.00059
13.4	12.3	0.0051	0.0011	14.8	0.00030	0.00089
16.7	15.1	0.0051	0.0000	12.0	0.00000	0.00089
18.9	17.8	0.0051	0.0000	10.2	0.00000	0.00089
20.5	19.7	0.0066	0.0015	9.18	0.00065	0.00154
23.8	22.2	0.0066	0.0000	8.17	0.00000	0.00154
25.6	24.7	0.0066	0.0000	7.32	0.00000	0.00154
34.6	30.1	0.0079	0.0013	6.01	0.00087	0.00241
44.6	39.6	0.0079	0.0000	4.57	0.00000	0.00241
54.6	49.6	0.0079	0.0000	3.65	0.00000	0.00241
64.6	59.6	0.0104	0.0025	3.03	0.0033	0.0057
79.6	72.1	0.0130	0.0026	2.51	0.0041	0.0098
95.6	87.6	0.0155	0.0025	2.06	0.0048	0.0146
119	107	0.0298	0.0143	1.69	0.0339	0.0485
144	132	0.0428	0.0130	1.38	0.0378	0.0863
180	162	0.0573	0.0145	1.12	0.0520	0.1383

micromeritics®

Figure H-5. Pore Area Distribution Data Form (completed sample)

Average pore diameter ($\bar{D}$) is obtained by dividing the $\bar{P}$ value into the constant 180, when using a contact angle value of 130 degrees and mercury surface tension of 485 dynes/cm, and recording them in column 5, for example, by solving

$$D = \frac{180}{P}$$

Incremental pore volume (ΔV) values are recorded in column 4, which are obtained by the difference between two sequential pore volume points from column 3, for example, from

$$\Delta V = V_{n+1} - V_n$$

Column 6, the incremental pore surface area (A, m^2/g) is the product of the values in columns 2 and 4, multiplied by 0.0221. Column 7, the cumulative surface area (ΣA, m^2/g) is obtained by adding the incremental surface area for the current interval and all preceding intervals. At this point, a cumulative surface area plot may be made vs. mean pore diameter ($\bar{D}$) by plotting column 7 vs. column 5.

An ~~area~~ distribution curve can also be differentiated or plotted in a derivative mode. It is possible to plot $\overline{P\Delta V}$ vs. D, or to take $\overline{\Delta A}$ from column 6.

Plotting ΔA vs. ΔD then yields an incremental area distribution curve.

APPENDIX I

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[1] is

$$dW = \gamma \cos\theta \; dA \tag{1}$$

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

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

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

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

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

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

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[2] 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

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

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

2. 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 J

AUTOMATIC DATA REDUCTION

AUTOMATIC DATA REDUCTION

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

P_i = head-corrected pressure as stored
Vr_i = intrusion volume as stored
θ = user-entered contact angle
Υ = user-entered surface tension
Ws = user-entered sample weight
Wp = user-entered weight for penetrometer
Wpsm = user-entered weight for penetrometer + sample + mercury
Vp = user-entered volume for penetrometer
Vc = user-entered volume for capillary (stem)
Ym = user-entered density for mercury

$$\text{WASHCON} = \text{Washburn constant} = \frac{10^4\ \mu\text{m/cm}}{68947.6\ \text{dynes/cm}^2\text{–psia}} = 0.145038$$

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

Diameter for the ith point is:

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

Radius for the ith point is:

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

Specific cumulative intrusion volume for the ith point is:

$$I_i = \frac{Vr_i}{Ws}$$

Mean diameter for the ith point is:

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

Incremental specific intrusion volume for the ith point is:

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

---

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

Incremental specific pore area for the ith point is:

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

Cumulative specific pore area for the ith 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 ith 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 ith 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:

$$Vtot = Vr_j$$

where the jth point is the first such that:

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

Total specific intrusion volume is:

$$Itot = \frac{Vtot}{Ws}$$

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

$$Ip_i = \frac{100 \times I_i}{Itot}$$

Total specific pore area is:

$$Atot = A_j$$

for point j as defined above.

Median diameter by volume is:

$$Dmv = D_k$$

where

$$I_k = \frac{Itot}{2}$$

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:

$$Dma = D_k$$

where

$$A_k = \frac{Atot}{2}$$

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

Average diameter is:

$$Dav = \frac{4 \text{ x } Itot}{Atot}$$

Bulk volume is:

$$Vb = Vp - \frac{Wpsm - Ws - Wp}{Ym}$$

Bulk density is:

$$Yb = \frac{Ws}{Vb}$$

Skeletal volume is:

$$Vs = Vb - Vtot$$

Skeletal density is:

$$Ys = \frac{Ws}{Vs}$$

Porosity % is:

$$Ppc = \frac{100 \text{ x } Vtot}{Vb}$$

Percent capillary used is:

$$Vpc = \frac{100 \text{ x } Vtot}{Vc}$$