VOLUMETRIC REFERENCE CAPSULE

INFORMATION MANUAL

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TRADEMARKS

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ABOUT THIS MANUAL

The following icons may be found in this manual:

NOTE - Notes contain important information applicable to the topic.

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

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

References to the AutoPore Operator Manual are found throughout this document. The operator manual is accessed using *Help* > *Operator's Manual* (or use the **F1** key) within the AutoPore application. A PDF copy of the manual can be found by logging in to your customer portal.

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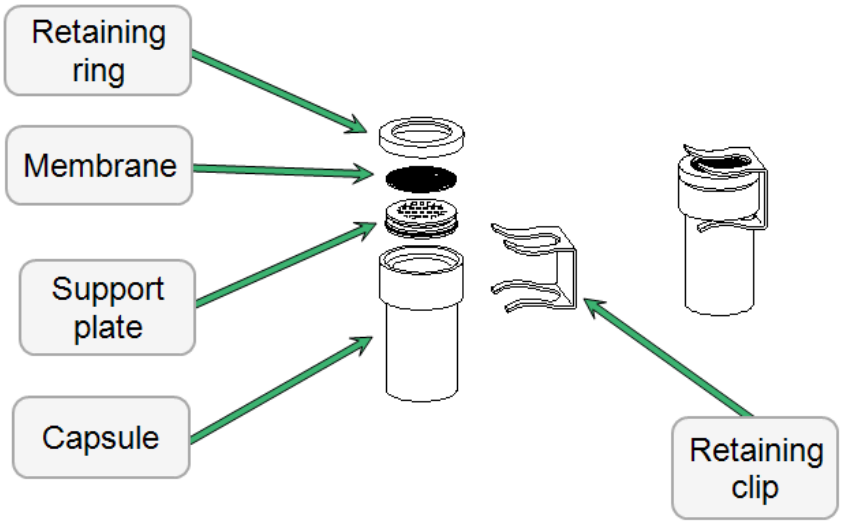
ABOUT THE VOLUMETRIC REFERENCE CAPSULE

VOLUMETRIC REFERENCE CAPSULE KIT CONTENTS



The reference capsule is used to verify the accuracy of AutoPore Series mercury intrusion volume measurements.

Capsule sizes:	• A large capsule having an internal volume of approximately 0.8 mL • A small capsule having an internal volume of approximately 0.24 mL
Membrane sizes	• An 8µm membrane for the verification of low pressure analysis • A 3µm membrane for the verification of high pressure analysis

KIT CONTENTS

- 2 large capsule body
- 2 small capsule body
- 4 retaining rings
- 4 support plates
- 25 O-rings
- 4 retaining clips
- 1 3µm membrane set
- 1 8µm membrane set
- 1 wooden case
- 1 information manual

Reference Capsule

The reference capsule is used to verify that the accuracy of mercury intrusion volume measurements, made by the AutoPore and its associated penetrometer, are within specifications. The reference capsule volume test confirms that a given sample port and associated penetrometer are measuring intrusion volumes accurately. The reference capsule volume test is performed after pressure transducer and capacitance detector readings have been calibrated. In the case of a high pressure reference capsule volume test, an empty chamber test must have been successfully completed to confirm that no errors are induced from the feed-thru.

Mercury Porosimetry Calibration/Verification

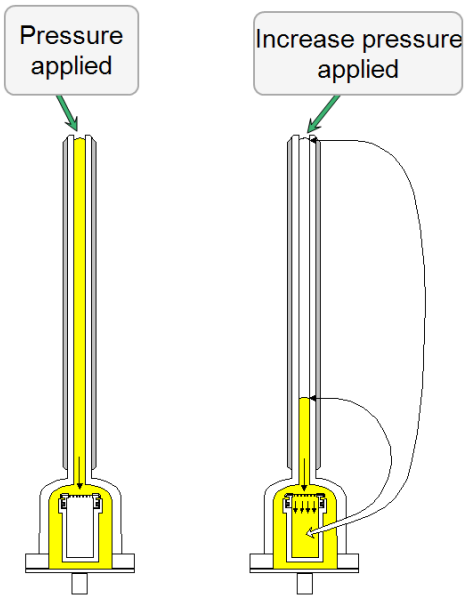
The AutoPore mercury porosimeter determines pore volume distribution by measuring the pressure applied to a mercury filled penetrometer and the change in electrical capacitance of the penetrometer caused by the intrusion of mercury into the sample. These two quantities make up each data point. The diameter of a pore is measured by the amount of pressure required to force the mercury into the pore while a change in the capacitance value of the penetrometer is used to compute the volume of mercury that filled the pores of that size.

Calibration of the pressure measurement function, from which pore diameters are computed in mercury porosimetry, is done directly by using an external NIST traceable pressure measuring device.

Calibration of the volume/capacitance measurement function in mercury porosimetry has been done indirectly by using a chain of separate, interacting calibrations. These have included the penetrometer capillary diameter, capillary cross-sectional area, capillary length, and electrical capacitance to an outer electrode of a unit length of mercury capillary.

Theoretical Basis of the Reference Capsule Volume Test

The reference capsule volume calibration verification system uses a small cylindrical device (volumetric reference capsule assembly) inserted into the penetrometer bulb in the same manner as a sample. The capsule assembly has its internal cavity separated from the exterior by a replaceable membrane. The membrane has numerous uniform pores of precisely controlled cross-sectional shape that are oriented uniformly by the top surface plane of the membrane. The porous membrane permits the reference capsule's internal cavity, along with the penetrometer, to be evacuated of air. The porous membrane then allows mercury filling of the penetrometer to be done at a few pounds per square inch without the entry of mercury into the reference capsule's internal cavity. It then permits the internal cavity to be filled with mercury at a specific higher pressure threshold. This allows the volume of mercury entering the reference capsule's internal cavity to be registered by the mercury porosimeter as if the capsule were a real sample.



The mercury intruded into the reference capsule's internal cavity does not significantly retract when the pressure is lowered back to atmosphere but remains inside the capsule's internal cavity where, upon removal of the capsule from the penetrometer bulb, the mercury weight gain of the reference capsule can be determined on a balance which has been NIST certified.

The true volume of this mercury can then be determined by a computation using the certified handbook values of mercury density at the prevailing temperature. Comparison of this true volume to the volume reported by the mercury porosimeter, as the total intrusion volume, will reveal the presence and amount of any volumetric error made by the entire volumetric measurement system of the mercury porosimeter, including the associated penetrometer.

Prior to regular use, each volumetric reference capsule should have its internal mercury volume capacity measured and recorded. Although it is preferable to do weighing upon each use to confirm that no malfunction of the filling step has occurred, comparison of the capsule's known (recorded) volume and the intrusion volume reported by the mercury porosimeter has proven to be quite accurate, with less than 1% variance in the total intrusion volume.

Verification of accuracy of the volume data is the primary need addressed by the volumetric reference capsule. Useful, though not fundamentally certifiable, information about the accuracy of the diameter/pressure data can also be obtained. The pores of the membrane used in the volumetric reference capsule are sufficiently precise and uniform enough in that the midpoint of the pore filling can be specified to about 4% of the mean pore size, within a given lot of membrane material. This pore size information can give a quick verification that the measured pore size is not in substantial error. More precise calibration or verification of the pore diameter and pressure data is best done by the use of NIST traceable pressure standards. These pressure standards are best derived from primary standards that use the *dead weight* pressure-tester principle.

DETERMINE THE REFERENCE CAPSULE VOLUME

This test should be performed three times to determine an average volume for the capsule. The calculated intrusion volume and the AutoPore reported intrusion volume for each of the three tests must agree with the capsule's average internal volume to within ± 1 %.

1. Disassemble the capsule assembly.
2. Clean all parts with soap and water, then rinse with IPA. Dry using clean, compressed nitrogen or clean, compressed air.
3. Reassemble with a membrane and weigh to get an empty capsule assembly weight.
4. Perform a mercury intrusion analysis on the capsule assembly.
5. Remove the capsule assembly from the penetrometer.
6. Remove any extraneous mercury droplets adhering to the exterior of the capsule.
7. Reweigh the mercury filled capsule assembly.

   The difference between the empty capsule assembly mass and the mercury filled capsule assembly mass is the mass of mercury retained in the capsule assembly. Divide this mass by the density of mercury at the test temperature to obtain the volume of mercury intruded in the volumetric reference capsule's internal cavity. The calculated intrusion volume and the AutoPore's reported intrusion volume for each of the three tests must agree with the capsule's average internal volume to within ± 1 % to be considered valid.

VOLUME REFERENCE CAPSULE ANALYSIS

The reference capsule is used to ensure the accuracy of mercury intrusion volume measurements, made by the AutoPore analyzers and associated penetrometers, are within specifications. The reference capsule volume test confirms that a given sample port and associated penetrometer are measuring intrusion volumes accurately. The reference capsule volume test is performed after pressure transducer and capacitance detector readings have been calibrated. In the case of a high pressure reference capsule volume test, an empty chamber test must have been successfully completed to confirm that no errors are induced by the feed-thru.

REFERENCE CAPSULE ANALYSIS WORKFLOW SUMMARY

1. Ensure all required materials are available. See ***Required Materials below***.
2. Assemble, weigh, and install the reference capsule. See ***Assemble, Weigh, and Install the Reference Capsule on the next page***.
3. Create two sample files for the 8µm membrane capsules and two sample files for the 3µm membrane capsules. See ***Low Pressure Analysis on page 8***.
4. Run low pressure analyses using the four capsules. See ***Start a Low Pressure Analysis on page 8***.
5. Transfer the two 3µm membrane capsules to the high pressure chambers.
6. Run high pressure analyses using the sample files for the 3µm membrane. See ***Start a High Pressure Analysis on page 9***.
7. Verify the volumetric reference capsule test results. See ***Verify Reference Capsule Analysis Results on page 13***.

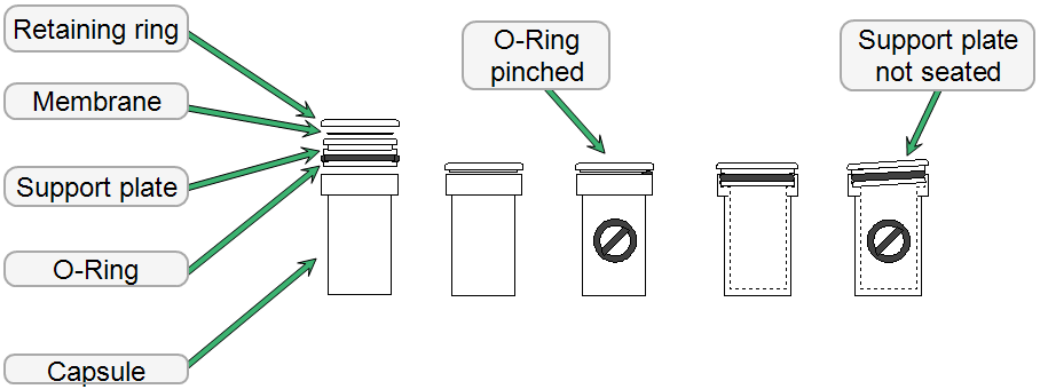
REQUIRED MATERIALS

- Laboratory thermometer, accurate to ± 0.25 °C in the 20 to 30 °C range
- Laboratory balance with 1 milligram resolution in the 1 to 10 gram range
- Suction device for removing small droplets of mercury
- 940-34800-00 Volumetric Reference Capsule Assembly — Small
  940-34800-01 Volumetric Reference Capsule Assembly — Large
- 950-61707-00 Penetrometer Assembly — 5 cc Solid, 0.392 stem volume for a small capsule
  950-61709-00 Penetrometer Assembly — 5 cc Solid, 1.131 stem volume for a large capsule
- Calibrated AutoPore analyzer

ASSEMBLE, WEIGH, AND INSTALL THE REFERENCE CAPSULE

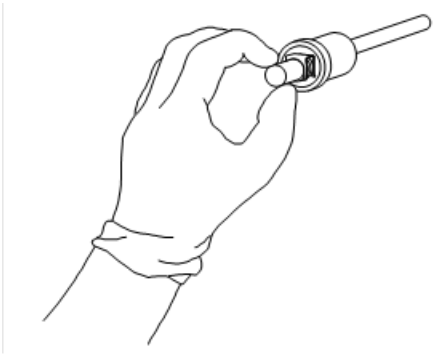
1. Clean the capsule with soap and water, then rinse with IPA. Dry using clean, compressed nitrogen or clean, compressed air.
2. Assemble the capsule with a membrane:
   - Use a 3µm membrane for high pressure analysis verification.
   - Use an 8µm membrane for low pressure analysis verification.

   a. Install the support plate into the capsule with the flat side up. The support plate must be properly seated, level, and the O-ring not pinched.



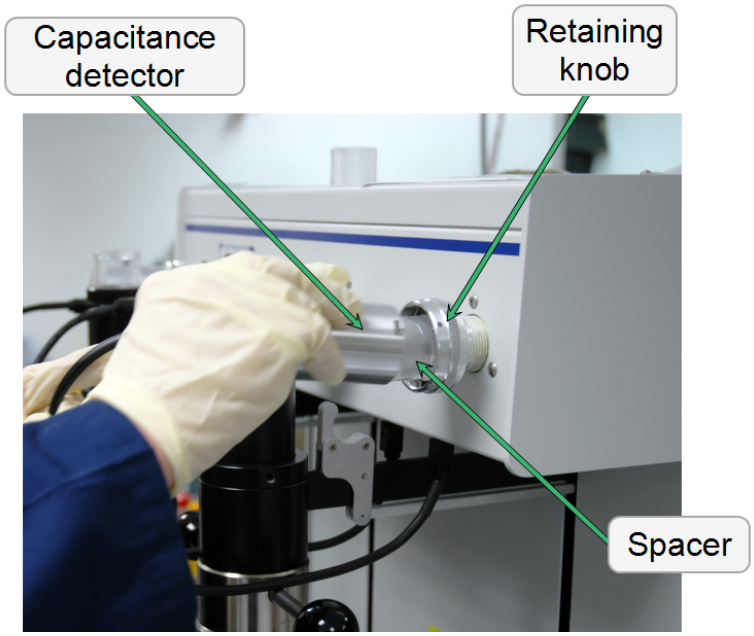
   b. Center the membrane on the support plate.
   c. Center the retaining ring over the support plate.
   d. Secure the retaining ring to the capsule with the retaining clip.

3. Weigh the capsule assembly with the membrane installed and record on the *Volumetric Reference Capsule Worksheet on page 11*.
4. Load the capsule assembly (membrane side first) into the penetrometer and seal.



Refer to the AutoPore Operator Manual for information on loading a sample into the penetrometer.

5. Remove the capacitance detector assembly and blank rod from the low pressure port and install the loaded penetrometer assembly.
6. Tighten the seal around the penetrometer.
7. Reinstall the capacitance detector assembly.



LOW PRESSURE ANALYSIS

Create the sample file. Reference the AutoPore Operator Manual for instructions. Ensure ***Options > Option Presentation > Advanced*** is selected.

Create two *LP Reference Capsule* sample files and two *HP Reference Capsule* sample files.

- If the *LP Ref Capsule Large.SMP* file is available, go to ***File > Open > [LP Ref Capsule Large.SMP]*** and save the file under a different file name rather than modifying the original. If the *LP Ref Capsule Large.SMP* file is not available, see ***LP Reference Capsule Large on page 31*** for parameters.
- If the *HP Ref Capsule Large.SMP* file is available, go to ***File > Open > [HP Ref Capsule Large.SMP]*** and save the file under a different file name rather than modifying the original. If the *HP Ref Capsule Large.SMP* file is not available, see ***HP Reference Capsule Large on page 27*** for parameters.

START A LOW PRESSURE ANALYSIS

Unit > Low Pressure Analysis

1. Run a low pressure analysis. Use the AutoPore Operator Manual for information on running a low pressure analysis. Ensure ***Options > Option Presentation > Advanced*** is selected.

After the analysis is complete:

2. Remove the penetrometers and spacers from the low pressure ports.
3. Reinstall the blank rods and capacitance detectors.
4. For the 3µm membrane, install the penetrometers in the high pressure ports. Omit the remaining steps in this section and proceed to ***Start a High Pressure Analysis on the facing page***.
5. For the 8µm membrane, disassemble the penetrometer assembly and retrieve the capsule assembly.
6. Use a suction device to remove any mercury droplets clinging to the exterior surface of the capsule assembly.
7. Weigh the mercury filled capsule assembly and enter the value on the ***Volumetric Reference Capsule Worksheet on page 11***.
8. Proceed to ***Verify Reference Capsule Analysis Results on page 13***.

HIGH PRESSURE ANALYSIS

Use the two *HP Reference Capsule* sample files created in the section ***HP Reference Capsule Large on page 27***. Ensure ***Options > Option Presentation > Advanced*** is selected.

START A HIGH PRESSURE ANALYSIS

Unit [n] > High Pressure Analysis

1. Use the AutoPore Operator Manual for information on running a high pressure analysis. Ensure ***Options > Option Presentation > Advanced*** is selected.

When the high pressure analysis is complete:

2. Remove the penetrometers from the high pressure ports.
3. Disassemble the penetrometer assembly and retrieve the capsule assembly.
4. Use a suction device to remove any mercury droplets clinging to the exterior surface of the capsule assembly.
5. Weigh the mercury filled capsule assembly and enter the value on the ***Volumetric Reference Capsule Worksheet on page 11***.

VOLUMETRIC REFERENCE CAPSULE WORKSHEET INSTRUCTIONS

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

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

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

VOLUMETRIC REFERENCE CAPSULE WORKSHEET

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

Penetrometer Number _____ Date: _____

By: _____

First Calibration of Reference Capsule Volume:

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

Room Temp = _____ °C Density of mercury * = _____ g/ml

4. Volume of capsule (line 3 ÷ density of mercury) _____ ml

Second Calibration of Reference Capsule Volume:

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

Room Temp = _____ °C Density of mercury * = _____ g/ml

4. Volume of capsule (line 3 ÷ density of mercury) _____ ml

Third Calibration of Reference Capsule Volume:

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

Room Temp = _____ °C Density of mercury * = _____ g/ml

4. Volume of capsule (line 3 ÷ density of mercury) _____ ml

Average Reference Capsule Volume: _____ ml

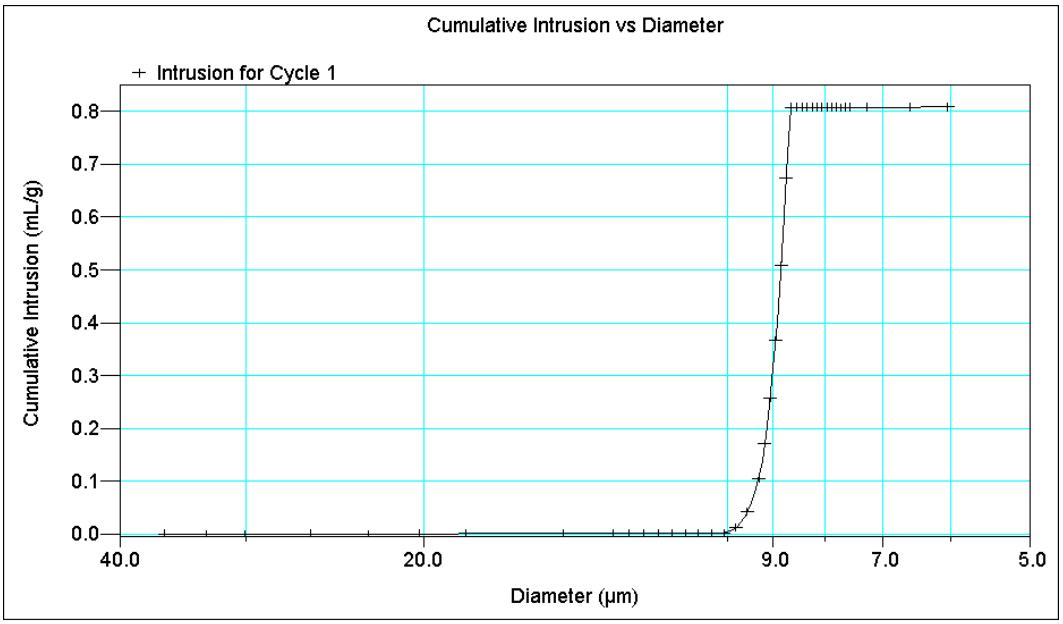
* See ***Density of Mercury Table on the next page*** for values.

DENSITY OF MERCURY TABLE

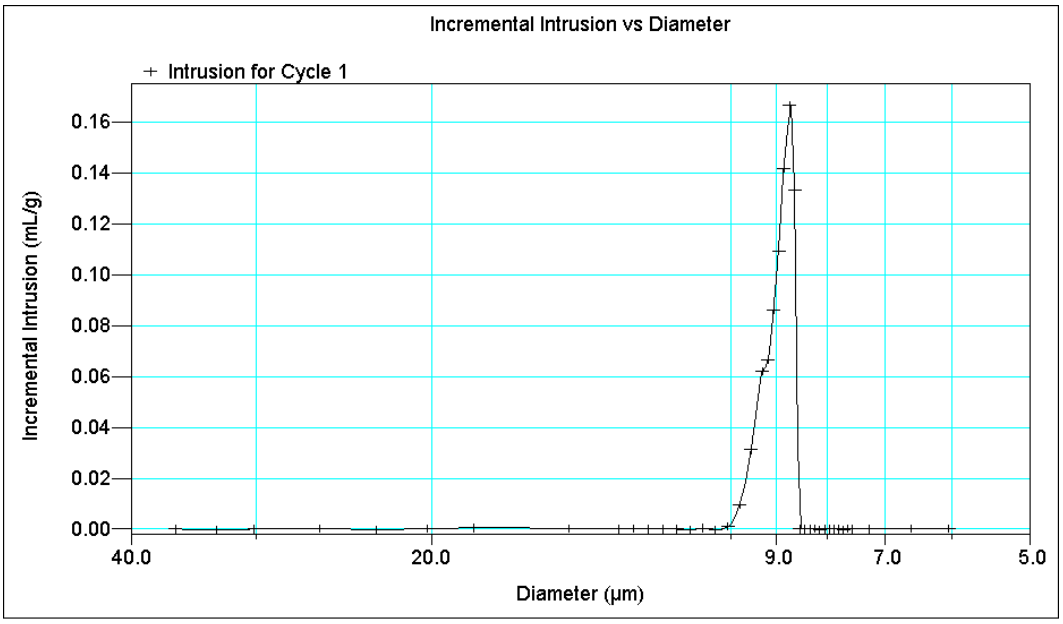
Density of Mercury Table

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

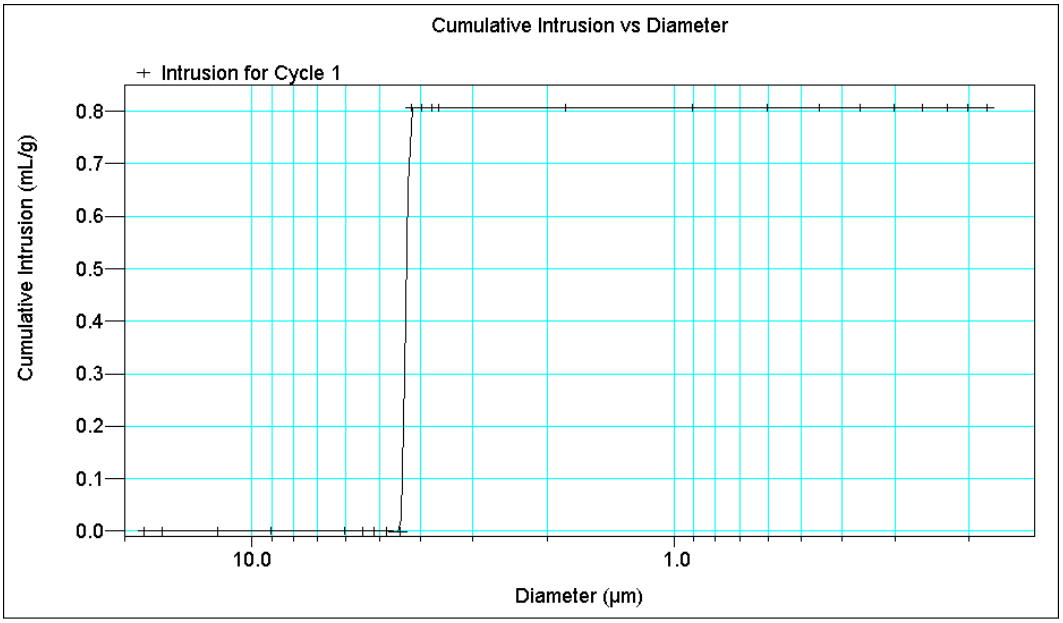
VERIFY REFERENCE CAPSULE ANALYSIS RESULTS



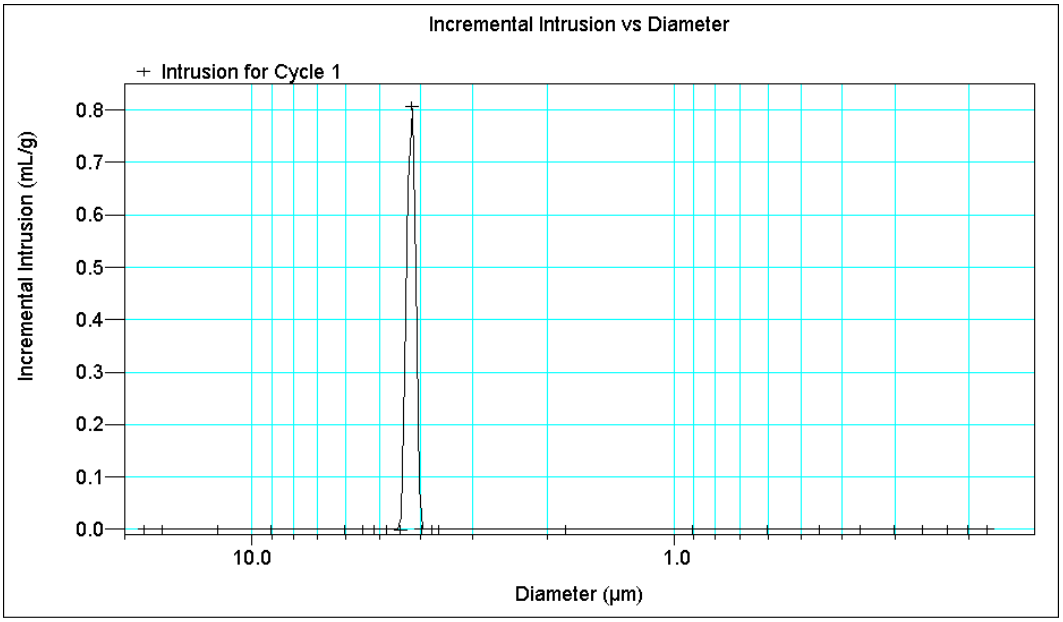
Typical Cumulative Intrusion on large capsule with 8µm membrane



Typical Incremental Intrusion on large capsule with 8µm membrane



Typical Cumulative Intrusion on large capsule with 3µm membrane



Typical Incremental Intrusion on large capsule with 3µm membrane

EMPTY CHAMBER TEST

EMPTY CHAMBER TEST WORKFLOW SUMMARY

1. Create the empty chamber test sample files. See ***Create the Empty Chamber Test Sample File below***.
2. Run low pressure analyses using the empty chamber sample file. See ***Start Low Pressure Analysis on the next page***.
3. Run high pressure analyses using the empty chamber sample file. See ***Start High Pressure Analysis on the next page***.
4. Verify the empty chamber test results. See ***Verify Empty Chamber Test Final Analysis Results on the next page***.

EMPTY CHAMBER TEST PREPARATION

1. Confirm blank rods and capacitance detectors are installed on all low pressure ports.
2. Lower the high pressure chamber plug(s) into the high pressure chamber(s). Add high pressure fluid to the relief valve and purge all the air from the chamber(s). Refer to the AuroPore Operator Manual for details on purging air from the high pressure chamber.

CREATE THE EMPTY CHAMBER TEST SAMPLE FILE

Create the sample file using the AutoPore Operator Manual for instructions. Ensure ***Options > Option Presentation > Advanced*** is selected.

If testing on an AutoPore IV 9505 and the *Empty Chamber Run 30K.SMP* file is available, go to ***File > Open > [Empty Chamber Run 30K.SMP]*** and save the file under a different file name rather than modifying the original. If the *Empty Chamber Run 30K.SMP* file is not available, see ***Empty Chamber Run 30K on page 17*** for parameters.

If testing on an AutoPore IV 9520 and the *Empty Chamber Run 60K.SMP* file is available, go to ***File > Open > [Empty Chamber Run 60K.SMP]*** and save the file under a different file name rather than modifying the original. If the *Empty Chamber Run 60K.SMP* file is not available, see ***Empty Chamber Run 60K on page 22*** for parameters.

START LOW PRESSURE ANALYSIS

Unit [n] > Low Pressure Analysis

Use the AutoPore Operator Manual for additional information on running a low pressure analysis. Ensure ***Options > Option Presentation > Advanced*** is selected.

When the low pressure analysis is complete, go to ***Start High Pressure Analysis below***.

START HIGH PRESSURE ANALYSIS

Unit [n] > High Pressure Analysis

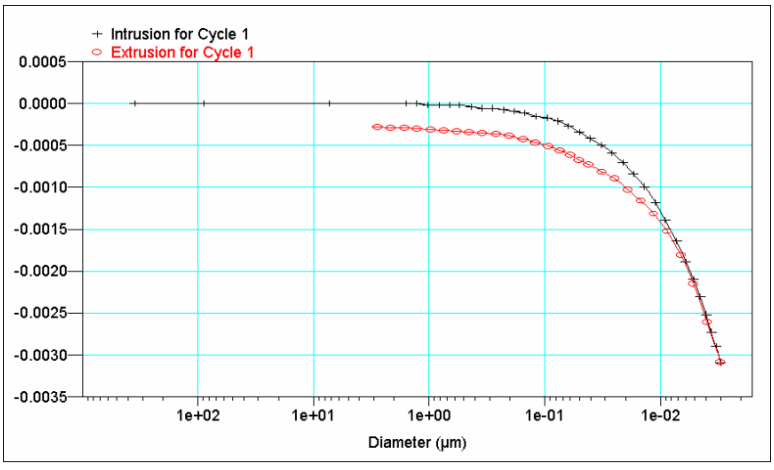
Penetrometers are not used for this analysis.

Use the AutoPore Operator Manual for additional information on running a high pressure analysis. Ensure ***Options > Option Presentation > Advanced*** is selected.

1. Lower the chamber plugs into the high pressure chambers.
2. Fill with high pressure fluid and purge the air from the system
3. Run a high pressure analysis using the instructions from the AutoPore Operator Manual.
4. When the high pressure analysis is complete, remove the excess oil from the high pressure chambers.

VERIFY EMPTY CHAMBER TEST FINAL ANALYSIS RESULTS

The *Cum. Vol. vs. Pressure* plot should be smooth with only minor jumps. Spikes more than ± 0.0005 ml are not acceptable.



SAMPLE FILES

Sample files in this section reference the AutoPore 9600, however the settings are the same for the AutoPore 9500.

EMPTY CHAMBER RUN 30K

```
Sample Information

                    Sample Information

                     Method:    Default
                      Sample    30K EMPTY CHAMBER TEST
                    Operator
                   Submitter
               Type of data:    Automatically collected
            Instrument type:    9600
  Original instrument type:     9600
                   Comments:
          Penetrometer mass:    1.0000 g
              Assembly mass:    1.0000 g

                             Material Properties

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

            Penetrometer Properties

         Penetrometer:    EMPTY CHAMBER TEST
   Penetrometer mass:     1.0000 g
              Volume:     1.0000 mL
            Constant:     10.000 µL/pF
          Stem volume:    0.4120 mL
  Max. head pressure:     4.680 psia
    Correction method:    None

Analysis Conditions
```

Analysis Conditions

```
Analysis Conditions:    30K EMPTY CHAMBER TEST

                  Mercury Properties

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

                Evacuation Options

            Sample type:    Other
  Initially evacuate at:    10.0 psia/min
    Switch to medium at:    Use pressure transducer
                            0.50 psia
      Switch to fast at:    900 µmHg
      Evacuation target:    50 µmHg
Continue evacuating for:    1 min

          Low Pressure

  Filling pressure:    1.500 psia
Equilibration time:    1 s

         High Pressure

      Equilibration time:    1 s
Hold at maximum pressure:    No

           Reverberi Options

Autocalculate Reverberi pressures:    No
```

Pressure Table

	Pressure Increment (psia)	Points per Decade	Ending Pressure (psia)	Maximum Intrusion (mL/g)	Pressure Scan Rate (min/decade)	Intrusion Scan Rate (mL/g·s)
1			2.00		5.0	0.00100
2			3.00		5.0	0.00100
3			4.00		5.0	0.00100
4			5.00		5.0	0.00100
5			6.00		5.0	0.00100
6			7.00		5.0	0.00100
7			8.00		5.0	0.00100
8			10.00		5.0	0.00100
9			13.00		5.0	0.00100
10			16.00		5.0	0.00100
11			20.00		5.0	0.00100
12			25.00		5.0	0.00100
* 13			30.00		5.0	0.00100
14			40.00		5.0	0.01000
15			50.00		5.0	0.01000
16			60.00		5.0	0.01000
17			75.00		5.0	0.01000
18			90.00		5.0	0.01000
19			115.00		5.0	0.01000
20			140.00		5.0	0.01000
21			175.00		5.0	0.01000
22			220.00		5.0	0.01000
23			270.00		5.0	0.01000
24			330.00		5.0	0.01000
25			420.00		5.0	0.01000
26			520.00		5.0	0.01000
27			640.00		5.0	0.01000
28			800.00		5.0	0.01000
29			990.00		5.0	0.01000
30			1,200.00		5.0	0.01000
31			1,500.00		5.0	0.01000
32			1,900.00		5.0	0.01000
33			2,350.00		5.0	0.01000
34			2,900.00		5.0	0.01000
35			3,600.00		5.0	0.01000
36			4,500.00		5.0	0.01000
37			5,600.00		5.0	0.01000
38			6,900.00		5.0	0.01000
39			8,600.00		5.0	0.01000
40			10,600.00		5.0	0.01000
41			13,200.00		5.0	0.01000
42			16,400.00		5.0	0.01000
43			20,000.00		5.0	0.01000
44			25,000.00		5.0	0.01000
45			30,000.00		5.0	0.01000
46			27,300.00		5.0	0.01000
47			21,000.00		5.0	0.01000
48			16,000.00		5.0	0.01000
49			12,400.00		5.0	0.01000
50			9,600.00		5.0	0.01000

51	7,300.00	5.0	0.01000
52	5,700.00	5.0	0.01000
53	4,300.00	5.0	0.01000
54	3,300.00	5.0	0.01000
55	2,600.00	5.0	0.01000
56	2,000.00	5.0	0.01000
57	1,500.00	5.0	0.01000
58	1,200.00	5.0	0.01000
59	900.00	5.0	0.01000
60	700.00	5.0	0.01000
61	500.00	5.0	0.01000
62	400.00	5.0	0.01000
63	300.00	5.0	0.01000
64	240.00	5.0	0.01000
65	190.00	5.0	0.01000
66	145.00	5.0	0.01000
67	110.00	5.0	0.01000
68	85.00	5.0	0.01000
69	65.00	5.0	0.01000
70	50.00	5.0	0.01000
71	30.00	5.0	0.01000
72	15.00	5.0	0.01000

Report Options

Report Options:	30 K EMPTY CHAMBER TEST
Show report title:	Yes
	EMPTY CHAMBER RUN 30K
Show bitmap:	Yes
Height:	0.250 in
Width:	2.000 in
Overlay Files:	None
Specification Files:	None
Reference Files:	None

Intrusion Data Options

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

Reports

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

Cum. Vol. vs Pressure

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

X-Axis

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

Y-Axis

Variable:	Cumulative Intrusion
Overlay:	
Plot intrusion:	All
Plot extrusion:	All
Autoscale:	No
Range:	-0.0050 - 0.0010 mL/g

Sample log

Sample log

Date	Time	Log Message

EMPTY CHAMBER RUN 60K

Sample Information

Sample Information

Method:	Default
Sample	60K EMPTY CHAMBER TEST
Operator	
Submitter	
Type of data:	Automatically collected
Instrument type:	9600
Original instrument type:	9600
Comments:	
Penetrometer mass:	1.0000 g
Assembly mass:	1.0000 g

Material Properties

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

Penetrometer Properties

Penetrometer:	EMPTY CHAMBER TEST
Penetrometer mass:	1.0000 g
Volume:	1.0000 mL
Constant:	10.000 µL/pF
Stem volume:	0.4120 mL
Max. head pressure:	4.680 psia
Correction method:	None

Analysis Conditions

```
Analysis Conditions:    60K EMPTY CHAMBER TEST

                  Mercury Properties

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

                Evacuation Options

            Sample type:    Other
  Initially evacuate at:    10.0 psia/min
    Switch to medium at:    Use pressure transducer
                            0.50 psia
      Switch to fast at:    900 µmHg
      Evacuation target:    50 µmHg
Continue evacuating for:    1 min

          Low Pressure

  Filling pressure:    1.500 psia
Equilibration time:    1 s

         High Pressure

      Equilibration time:    1 s
Hold at maximum pressure:    No

           Reverberi Options

Autocalculate Reverberi pressures:    No
```

Pressure Table

	Pressure Increment (psia)	Points per Decade	Ending Pressure (psia)	Maximum Intrusion (mL/g)	Pressure Scan Rate (min/decade)	Intrusion Scan Rate (mL/g·s)
1			2.00		5.0	0.00100
2			3.00		5.0	0.00100
3			4.00		5.0	0.00100
4			5.00		5.0	0.00100
5			6.00		5.0	0.00100
6			7.00		5.0	0.00100
7			8.00		5.0	0.00100
8			10.00		5.0	0.00100
9			13.00		5.0	0.00100
10			16.00		5.0	0.00100
11			20.00		5.0	0.00100
12			25.00		5.0	0.00100
* 13			30.00		5.0	0.00100
14			40.00		5.0	0.01000
15			50.00		5.0	0.01000
16			60.00		5.0	0.01000
17			75.00		5.0	0.01000
18			90.00		5.0	0.01000
19			115.00		5.0	0.01000
20			140.00		5.0	0.01000
21			175.00		5.0	0.01000
22			220.00		5.0	0.01000
23			270.00		5.0	0.01000
24			330.00		5.0	0.01000
25			420.00		5.0	0.01000
26			520.00		5.0	0.01000
27			640.00		5.0	0.01000
28			800.00		5.0	0.01000
29			990.00		5.0	0.01000
30			1,200.00		5.0	0.01000
31			1,500.00		5.0	0.01000
32			1,900.00		5.0	0.01000
33			2,350.00		5.0	0.01000
34			2,900.00		5.0	0.01000
35			3,600.00		5.0	0.01000
36			4,500.00		5.0	0.01000
37			5,600.00		5.0	0.01000
38			6,900.00		5.0	0.01000
39			8,600.00		5.0	0.01000
40			10,600.00		5.0	0.01000
41			13,200.00		5.0	0.01000
42			16,400.00		5.0	0.01000
43			20,000.00		5.0	0.01000
44			25,000.00		5.0	0.01000
45			30,000.00		5.0	0.01000
46			35,000.00		5.0	0.01000
47			40,000.00		5.0	0.01000
48			45,000.00		5.0	0.01000
49			50,000.00		5.0	0.01000
50			55,000.00		5.0	0.01000

51	60,000.00	5.0	0.01000
52	46,100.00	5.0	0.01000
53	35,500.00	5.0	0.01000
54	27,300.00	5.0	0.01000
55	21,000.00	5.0	0.01000
56	16,000.00	5.0	0.01000
57	12,400.00	5.0	0.01000
58	9,600.00	5.0	0.01000
59	7,300.00	5.0	0.01000
60	5,700.00	5.0	0.01000
61	4,300.00	5.0	0.01000
62	3,300.00	5.0	0.01000
63	2,600.00	5.0	0.01000
64	2,000.00	5.0	0.01000
65	1,500.00	5.0	0.01000
66	1,200.00	5.0	0.01000
67	900.00	5.0	0.01000
68	700.00	5.0	0.01000
69	500.00	5.0	0.01000
70	400.00	5.0	0.01000
71	300.00	5.0	0.01000
72	240.00	5.0	0.01000
73	190.00	5.0	0.01000
74	145.00	5.0	0.01000
75	110.00	5.0	0.01000
76	85.00	5.0	0.01000
77	65.00	5.0	0.01000
78	50.00	5.0	0.01000
79	30.00	5.0	0.01000
80	15.00	5.0	0.01000

Report Options

Report Options:	60K EMPTY CHAMBER TEST
Show report title:	Yes
	EMPTY CHAMBER RUN 60K
Show bitmap:	Yes
Height:	0.250 in
Width:	2.000 in
Overlay Files:	None
Specification Files:	None
Reference Files:	None

Intrusion Data Options

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

Reports

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

Cum. Vol. vs Pressure

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

X-Axis

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

Y-Axis

Variable:	Cumulative Intrusion
Overlay:	
Plot intrusion:	All
Plot extrusion:	All
Autoscale:	No
Range:	-0.0050 - 0.0010 mL/g

Sample log

Sample log

Log Date	Time	Message
-		

HP REFERENCE CAPSULE LARGE

Sample Information

Sample Information

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

Material Properties

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

Penetrometer Properties

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

Analysis Conditions

Analysis Conditions: HP REF CAPSULE (LARGE)

Mercury Properties

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

Evacuation Options

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

Low Pressure

Filling pressure:	10.000 psia
Equilibration time:	2 s

High Pressure

Equilibration time:	20 s
Hold at maximum pressure:	No

Reverberi Options

Autocalculate Reverberi pressures: No

Pressure Table

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

Report Options

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

           Intrusion Data Options

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

              Reports

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

                   Summary Report

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

```
        Pore Structure Summary

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

          Physical Properties Summary

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

                Tabular Report

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

           Cum. Vol. vs Pressure

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

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

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

LP REFERENCE CAPSULE LARGE

Sample Information

Sample Information

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

Material Properties

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

Penetrometer Properties

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

Analysis Conditions

Analysis Conditions: LP REF CAPSULE (LARGE)

Mercury Properties

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

Evacuation Options

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

Low Pressure

Filling pressure:	5.000 psia
Maximum intrusion volume:	0.050 mL/g
Equilibration time:	20 s

High Pressure

Equilibration time:	1 s
Hold at maximum pressure:	No

Reverberi Options

Autocalculate Reverberi pressures: No

Pressure Table

	Pressure Increment (psia)	Points per Decade	Ending Pressure (psia)	Maximum Intrusion (mL/g)	Pressure Scan Rate (min/decade)	Intrusion Scan Rate (mL/g·s)
1			5.50	0.050	5.0	0.00100
2			6.00	0.050	5.0	0.00100
3	1.00		10.00	0.050	5.0	0.00100
4	2.00		14.00	0.050	5.0	0.00100
5	0.50		19.50	0.050	5.0	0.00100
6	0.25		24.00	0.050	5.0	0.00100
7			25.00	0.050	5.0	0.00100
* 8	2.50		30.00	0.050	5.0	0.00100

Report Options

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

            Intrusion Data Options

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

               Reports

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

                     Summary Report

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

     Pass/Fail

 Pass/Fail 1:    No
 Pass/Fail 2:    No
 Pass/Fail 3:    No
 Pass/Fail 4:    No
```

```
       Pore Structure Summary

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

        Physical Properties Summary

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

               Tabular Report

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

          Cum. Vol. vs Pressure

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

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

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