



A Honeywell Company

Automated Pore Volume and Pore Size Distribution of Porous Substances by Mercury Porosimetry

UOP Method 578-11

Scope

This method is for determining the pore size distribution and pore area of materials by mercury intrusion. Total pore volume (intrusion volume), total pore area, bulk (piece) density and apparent (skeletal) density are also determined. At the maximum pressure of 413,700 kPa (60,000 psia), pores as small as 0.003 μm are filled. This method is applicable to materials which are essentially non-compressible at the pressures attained during analysis. The sample must also be non-wetting and non-reactive with mercury, and not larger than a right circular cylinder having 25-mm diameter with a length of 25 mm.

References

Instruction Manual, supplied with instrument

UOP Method 999, "Precision Statements in UOP Methods," www.astm.org

Outline of Method

Previously outgassed samples are weighed into penetrometers and placed in the low-pressure ports where they are evacuated and automatically filled with mercury. Due to the unique non-wetting property of mercury, it will not enter pores or capillaries except under pressure. Filling pressures between 3.4 kPa (0.5 psia) and atmospheric pressure can be used depending on whether larger pores are of interest. The samples are then removed from the low-pressure ports and placed in the high pressure chambers. Using high-pressure fluid, the mercury is forced into the pores at incrementally increasing pressure to measure the intrusion data. Pressure is increased to a maximum of 413,700 kPa (60,000 psia). Pore volume readings are taken at predetermined pressures. If extrusion data, a measure of the mercury coming out of the pores as the pressure is reduced, is desired, pore volume readings are also taken as the pressure is reduced to atmospheric. A maximum of 2,500 pressure points can be chosen for the intrusion/extrusion curve. The standard pressure points and their corresponding pore diameters are listed in Table 1. Other pressure tables are available for use based on the submitter's needs or the specific pore ranges needed to be highlighted.

Apparatus

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).

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Nonconfidential UOP Methods are available from ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959, USA. The UOP Methods may be obtained through the ASTM website, www.astm.org, or by contacting Customer Service at service@astm.org, 610.832.9555 FAX, or 610.832.9585 PHONE.

Balance, readability 0.0001 g, chamber accessible from both top and sides, Ohaus™ Analytical Plus Series, Model AP 310, Fisher Scientific, Cat. No. 01-920-80

Desiccator, with porcelain plate, 250-mm ID, Fisher Scientific, Cat. No. 08-615B

Dishes, Coors, shallow form porcelain, 80-mm diameter, 50-ml capacity, Fisher Scientific, Cat. No. 08-693B

Gauge, vacuum, range 0-0.001 kPa (0-250 mm Hg), Fisher Scientific, Cat. No. 11-282A, used with vacuum oven

Gloves, Kelkave Autoclave, temperature resistant up to 288°C (550°F), length 11 inches, size large, Fisher Scientific, Cat. No. 19-013-586

Hood, laboratory

Mercury intrusion porosimeter, supplied with data terminal and printer, Micromeritics, Autopore IV. This method is written assuming the use of the specified porosimeter. If other equipment is used, adapt the method accordingly.

Oven, vacuum, maximum temperature 200°C, maximum vacuum 0.01 kPa, chamber 20 x 20 x 30 cm, Precision™, Model 19, Fisher Scientific, Cat. No. 13-264A

Penetrometers, powder samples, 3-mL sample volume, 0.39-mL intrusion volume, Micromeritics, Cat. No. 942-61714-00, several required. Other penetrometers, with larger sample volumes and intrusion volumes are also available.

Penetrometers, solid samples, 3-mL sample volume, 0.39-mL intrusion volume, Micromeritics, Cat. No. 942-61713-00, several required. Other penetrometers, with larger sample volumes and intrusion volumes are also available.

Pump, vacuum, 91-L/min, Welch™ Duo Seal™, Model 1405, Fisher Scientific, Cat. No. 01-099

Regulator, nitrogen, single-stage, Matheson Gas Products, Model 1-580

Software, Autopore IV 9500, Version 1.09, Micromeritics

Thermometer, for measuring ambient temperature, Fisher Scientific, Cat. No. 15-060-274

Tongs, stainless steel, 23-cm, Fisher Scientific, Cat. No. 15-186

Reagents and Materials

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

Dessicant, Drierite, Fisher Scientific, Cat. No. 07-578-3A

Detergent, Alconox, Fisher Scientific, Cat. No. 50821294

Fluid, high pressure, Micromeritics, Cat. No. 920-16001-00

Gas, dry, air or nitrogen

Gloves, Best™ NitrilSolve™ Nitrile, Fisher Scientific, Cat. No. 11-395 series

Grease, stopcock, Apiezon™ H, Micromeritics, Cat. No. 004-16007-00

Grease, Dow Corning™, high vacuum, Fisher Scientific, Cat. No. 14-635-5D

Mercury, quadruple distilled, Bethlehem Apparatus. Caution: see *Precaution*.

Nitrogen, gas, 99.98% minimum purity

Procedure

The analyst is expected to be familiar with general laboratory practices, the technique of mercury porosimetry, and the equipment being used.

Calibrate the instrument as outlined in the Instruction Manual. Instrument operation must be thoroughly understood before any attempt is made to analyze samples. Enter the standard pressure points from Table 1.

- This Procedure is written assuming the use of the equipment specified under *Apparatus*. If other equipment is used, adapt the procedure accordingly.
- Although the use of metric units is preferred, the pressure units used by the listed porosimeter are psia, and thus, are listed in Table 1.

Table 1

Standard Pressure Points and Corresponding Pore Diameters
(Contact Angle $[\theta] = 130^\circ$, Surface Tension $[\gamma]$, = 485 dynes/cm)

<u>Pressure, psia</u>	<u>Pore Diameter, μm</u>	<u>Pressure, psia</u>	<u>Pore Diameter, μm</u>
8.9	20.0000	4486.0	0.0400
18.0	10.0000	5127.0	0.0350
31.0	5.8333	5981.0	0.0300
45.0	4.0000	6188.0	0.0290
103.0	1.7500	6409.0	0.0280
150.0	1.2000	6646.0	0.0270
199.0	0.9000	6902.0	0.0260
239.0	0.7500	7178.0	0.0250
299.0	0.6000	7477.0	0.0240
359.0	0.5000	7802.0	0.0230
513.0	0.3500	8156.0	0.0220
598.0	0.3000	8545.0	0.0210
665.0	0.2700	8972.0	0.0200
748.0	0.2400	9444.0	0.0190
854.0	0.2100	9969.0	0.0180
1025.0	0.1750	10555.0	0.0170
1196.0	0.1500	11215.0	0.0160
1495.0	0.1200	11963.0	0.0150
1794.0	0.1000	12817.0	0.0140
1889.0	0.0950	13803.0	0.0130
1994.0	0.0900	15337.0	0.0117
2111.0	0.0850	16313.0	0.0110
2243.0	0.0800	17944.0	0.0100
2393.0	0.0750	19938.0	0.0090
2563.0	0.0700	22430.0	0.0080
2761.0	0.0650	25634.0	0.0070
2991.0	0.0600	29907.0	0.0060
3263.0	0.0550	35888.0	0.0050
3589.0	0.0500	44860.0	0.0040
3988.0	0.0450	60000.0	0.0030

Calibration of Penetrometer Volume

To calibrate a penetrometer, simply perform a low pressure analysis with no sample. This fills the penetrometer with mercury so that the volume can be determined. Up to four penetrometers can be

calibrated at one time, utilizing all four low-pressure ports. Clean, grease, and seal each penetrometer according to the instructions in the manual.

1. Weigh the empty penetrometers to the nearest 0.0001 g and record the weight. See Appendix A in the manual for the penetrometer volume calibration form.
2. Place a penetrometer into each of the low-pressure ports.
3. Create and select sample file for analysis following manufacturer's instructions.
4. Remove the mercury filled penetrometers according to manual instructions, and reweigh to the nearest 0.0001 g, recording the weight.
5. Determine the ambient temperature.
6. Calculate the volume of the penetrometer, to three decimal places, using Equation 1:

$$V = \frac{B - A}{D} \quad (1)$$

where:

A = mass of empty penetrometer, g

B = mass of penetrometer filled with mercury, g

D = density of mercury at ambient temperature, g/mL. See Table 2. Extrapolate between listed temperatures as needed.

V = volume of penetrometer, mL

This procedure must be performed three times for each penetrometer. Use the average volume of the empty penetrometer in all further calculations involving that penetrometer. This must be done only once if all the parts of the penetrometer are kept together and a record of the results is maintained.

- Penetrometer volumes for the three replicates must all be within 0.01 mL. If greater deviation occurs, recheck the equipment and technique, and make additional calibrations until the required repeatability is obtained on three consecutive calibrations.

Table 2
Density of Mercury

<u>°C</u>	<u>g/mL</u>	<u>°C</u>	<u>g/mL</u>	<u>°C</u>	<u>g/mL</u>	<u>°C</u>	<u>g/mL</u>
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

Sample Preparation

1. Place enough sample in a shallow porcelain dish to run this test, noting what sample is in which dish.
2. Place the dishes in a vacuum oven and evacuate down to at least 0.04 kPa.
3. Heat the oven to $150 \pm 5^\circ\text{C}$ and allow samples to outgas for a minimum of 8 hours.

4. Release the seal on the vacuum oven allowing it to come to atmospheric pressure with dry air or dry nitrogen.
5. Remove the hot dishes and place in a desiccator to cool to room temperature. Samples should remain in the desiccator until they can be analyzed.

Sample Analysis

Referring to the instrument manual, determine the approximate amount of sample required and the appropriate calibrated penetrometer.

1. Apply Apiezon H stopcock grease to the empty penetrometer, but do not seal.
2. Weigh the complete disassembled penetrometer to the nearest 0.0001 g and record the weight.
3. Weigh and record the predetermined amount of sample from the cooled dishes. Fill the penetrometer with the weighed sample, making sure not to get any sample in the penetrometer grease.
4. Seal the cap assembly.
5. Place the penetrometer in one of the four low pressure ports, add a spacer behind each penetrometer if needed, making sure to put rods in all unused ports if four samples are not being analyzed. Cover with capacitance transducers.
6. Make sure that analysis conditions, penetrometer properties, and report options have been preset for your requirements. If not, refer back to the instruction manual for entering these steps.
7. Make sure that the mercury reservoir has been filled with clean mercury.
8. Select File, Open, and Sample information. Check that the sequence file name is the next determined number and click OK, then Yes, to create a sample file. Fill in the appropriate sample information, choose the appropriate analysis condition, Penetrometer properties and Report option for each sample being analyzed. When this information is entered select the unit number, Low pressure analysis, next, and the proper sample information from the list for each port being analyzed. When this has been completed, press Start to start the Low pressure analysis.
9. After completion of the low pressure run, remove each filled penetrometer, weigh in an upright position to the nearest 0.0001 g, and record the weight.
10. Place the full weighed penetrometer in one of the two high-pressure ports. Tighten down each chamber so oil fills the top vent valve. If more than one sample was run at low pressure, repeat this procedure on another sample in the second high pressure port. If more than two samples were run at low pressure, leave the remaining samples on the low pressure ports until a high pressure port becomes available.
11. Start the high pressure analysis, select the unit number, High pressure analysis, Browse, find the appropriate sample for each port, enter the penetrometer plus mercury weight and choose Start to analyze samples. Remember to tighten the vent valve caps when instructed to do so.
12. When the high pressure analysis is complete the data will have to be reviewed. Select Report, Start report, find the appropriate sample file number or numbers and click OK to review report
13. Repeat above steps for other samples.

Calculations

All calculations are performed by the computer via Version 1.09 software and printed out by the terminal. See the *Appendix* for examples of the reports and explanations of terms. The following equations are used and are given here for information only.

Assuming a cylindrical pore model, the relationship of the diameter of the pore to the pressure required to force mercury into it is described by the Washburn Equation (Equation 2):

$$P = \frac{-4\gamma \cos \theta}{D} \quad (2)$$

where:

D = diameter of the pore, μm

P = pressure, psia

γ = surface tension of mercury, dynes/cm

θ = contact angle between the mercury and the material being tested, degrees

Assuming a contact angle of 130° to be a reliable value for a wide range of materials, and 485 dynes/cm to be the most common value for the surface tension of mercury, the above equation reduces to Equation 3:

$$D = \frac{180}{P} \quad (3)$$

where:

D = diameter of the pore, μm

P = pressure, psia

Precaution

Mercury and mercury vapors are known health hazards. An exhaust and ventilating system is built into the instrument. Exercise caution to prevent spills when weighing and transporting filled penetrometers. It is mandatory that penetrometers be cleaned in a hood. All used mercury must be disposed in an environmentally safe manner according to local regulations.

Precision

Precision data were obtained using UOP Method 999.

Repeatability and Site Precision

A nested design was carried out for determining the intrusion volume of a catalyst with three analysts. Each analyst carried out tests on two separate days, performing two tests each day. The total number of tests performed was 12. The precision data are summarized in Table 3. Two tests performed by the same analyst on the same day should not differ by more than the repeatability allowable difference with 95% confidence. Two tests performed in one laboratory by different analysts on different days should not differ by more than the site precision allowable difference with 95% confidence.

The data in Table 3 are a short-term estimate of repeatability. When the test is run routinely, a control standard and chart should be used to develop a better estimate of the long-term repeatability.

Reproducibility

There is insufficient data to calculate the reproducibility of the test at this time.

Table 3
Repeatability and Site Precision, mL/g

<u>Total Mean Intrusion Volume*</u>	<u>Repeatability</u>		<u>Site Precision</u>	
	<u>Within- Day esd</u>	<u>Allowable Difference</u>	<u>Within- Lab esd</u>	<u>Allowable Difference</u>
1.71	0.019	0.06	0.042	0.16

*See definition in *Appendix*

The data in Table 3 are a short-term estimate of repeatability. When the test is run routinely, a control standard and chart should be used to develop a better estimate of the long-term repeatability.

Reproducibility

There is insufficient data to calculate the reproducibility of the test at this time.

Time for Analysis

The elapsed time for the sample pretreatment is 8 to 24 hours. The labor requirement is 0.25 hour. The elapsed time for one analysis is 1.0 hour. The total labor requirement is 0.75 hour.

Suggested Suppliers

Bethlehem Apparatus Co., Inc., 890 Front St., Hellertown, PA 18055, USA (610-838-7034)
www.bethlehemapparatus.com

Fisher Scientific, 711 Forbes Ave., Pittsburgh, PA 15219-4785, USA (412-490-8300)
www.fishersci.com

Matheson Tri-Gas, 166 Keystone Dr., Montgomeryville, PA 18936, USA (215-641-2700)
www.mathesontrigas.com

Micromeritics Instrument Corporation, One Micromeritics Dr., Norcross, GA 30093-1877, USA
(770-662-3620) www.micromeritics.com

Appendix

Explanation of Terms

TOTAL INTRUSION VOLUME is the maximum volume of mercury intrusion (penetration) into the pores of the sample at the maximum pressure obtained during the analysis.

CUMULATIVE PORE VOLUME PLOT is the change in the slope of a curve versus diameter to a quick overview of how porosity is distributed in the sample, how the penetrometer fills, sample failure, and an indication of sample compression due to the increased applied pressure.

dV/d(logD) PLOT is the difference between the logarithms of 2 quantities. The quantities are calculated using the log of the differential pore size.

TOTAL PORE AREA is the area of the pore walls based on the assumption of cylindrical geometry and summed over the complete pressure range of mercury intrusion.

MEDIAN PORE DIAMETER (VOLUME) is the pore diameter at which equal quantities of pore volume occur at both larger and smaller diameter.

MEDIAN PORE DIAMETER (AREA) is the pore diameter at which equal quantities of pore wall area occur at larger and smaller diameters.

The Median Pore Diameter (Volume) and Median Pore Diameter (Area) will frequently differ because larger pores in a distribution contribute heavily to the total volume while smaller pores contribute more to the total pore area. The difference between the two numbers will become greater as the pore distributions become wider or bimodal.

AVERAGE PORE DIAMETER (4 V/A) computation is based upon the assumption that all pores are right circular cylinders. When the pore volume ($V = \pi d^2 l / 4$) is divided by the pore area ($A = \pi d l$), the pore diameter (**d**) is $4 V / A$. A known volume of cylindrical capillaries having a known wall area yields an average pore diameter from this computation.

BULK DENSITY (PIECE DENSITY) is calculated from the sample weight, g, and the sample volume, mL, at the initial filling pressure. Penetrometer volume minus the volume of the mercury in the penetrometer with the sample gives the sample volume. The volume of the mercury is obtained from the mass of mercury, g, divided by the density of mercury (Table 2). The mass of the mercury is the difference between the weight of the penetrometer filled with sample and filled mercury and sample.

APPARENT (SKELETAL) DENSITY is the density of the sample as calculated from the bulk density and corrected for the pore volume measured by the mercury intrusion at the maximum pressure attained. However, if the sample contains pores smaller than 0.0030 μm , this value can only approximate the true value.

% CAPILLARY is for operator information only. It is the percentage of capillary volume used during the analysis. It should be between 25 and 90 for the analysis to be considered acceptable, unless the operator has justified otherwise. It is not used in any calculations.

Example, Typical Data Report

A typical data report from the instrument is shown in Figures A1 through A5.

Figure A1 is the summary report. It consists of two groupings of information. The upper grouping repeats the information that was entered by the operator and the constants used in the calculations. The lower grouping is a summary of the computed results.

Figures A2 and A3 list six columns of numerical data as follows:

Column 1 lists the actual pressure points (psia) obtained during the analysis corresponding to the table of pressure points loaded into the computer.

Column 2 lists the corresponding pore diameters (μm) as calculated from the actual pressure points in Column 1 using the Washburn equation, the constants for the contact angle, and the surface tension listed on Page 1 of the report.

Column 3 lists the mean pore diameter (D).

Column 4 lists the actual cumulative volume of the mercury intruded into the sample at the given pressure points in Column 1. The volumes are normalized to unit mass basis, mL/g, by dividing each penetration increment by the sample weight.

Column 5 lists the smoothed pore volume distribution function ($dV/d\log D$).

Column 6 lists the cumulative pore area

Any of the columns above may be customized for individual needs.

Additional pages consisting of normalized graphical presentations of the computed data are available if desired. Two of these pages are shown on Figures A4 and A5. If the extrusion volumes have also been requested, addition pages of information and plots will result.

LP Analysis Time: 9/1/2010 3:46:54AM
HP Analysis Time: 9/1/2010 7:20:36AM
Report Time: 12/1/2010 7:45:50AM

Sample Weight: 0.1599 g
Correction Type: None
Show Neg. Int: No

Summary Report

Penetrometer parameters

Penetrometer:	5cc Powder Penetrometer - #070		
Pen. Constant:	10.898 $\mu\text{L/pF}$	Pen. Weight:	64.9337 g
Stem Volume:	0.3920 mL	Max. Head Pressure:	4.4500 psia
Pen. Volume:	5.2500 mL	Assembly Weight:	131.7074 g

Hg Parameters

Adv. Contact Angle:	130.000 degrees	Rec. Contact Angle:	130.000 degrees
Hg Surface Tension:	485.000 dynes/cm	Hg Density:	13.5335 g/mL

Low Pressure:

Evacuation Pressure:	50 μmHg
Evacuation Time:	10 mins
Mercury Filling Pressure:	8.87 psia
Equilibration Time:	10 secs

High Pressure:

Equilibration Time:	10 secs
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No Blank Correction

Intrusion Data Summary

Total Intrusion Volume =	1.7021 mL/g
Total Pore Area =	227.595 m^2/g
Median Pore Diameter (Volume) =	0.0566 μm
Median Pore Diameter (Area) =	0.0186 μm
Average Pore Diameter ($4V/A$) =	0.0299 μm
Bulk Density at 14.70 psia =	0.4883 g/mL
Apparent (skeletal) Density =	2.8715 g/mL
Porosity =	83.0154 %
Stem Volume Used =	69 %

Figure A1

Typical Data Report Micromeritics Autopore IV 9500 V1.09 Page 1

LP Analysis Time: 9/1/2010 3:46:54AM
 HP Analysis Time: 9/1/2010 7:20:36AM
 Report Time: 12/1/2010 7:45:50AM

Sample Weight: 0.1599 g
 Correction Type: None
 Show Neg. Int: No

Tabular Report					
Pressure (psia)	Pore Diameter (μm)	Mean Diameter (μm)	Cumulative Pore Volume (mL/g)	Smoothed dV/dlogD Pore Volume (mL/g)	Cumulative Pore Area (m ² /g)
8.87	20.3917	20.3917	0.0000	9.548×10^{-3}	0.000
17.96	10.0701	15.2309	0.0027	2.917×10^{-3}	0.001
28.68	6.3060	8.1880	0.0027	0.000×10^0	0.001
42.35	4.2708	5.2884	0.0027	0.000×10^0	0.001
100.35	1.8024	3.0366	0.0027	0.000×10^0	0.001
147.17	1.2289	1.5156	0.0027	0.000×10^0	0.001
196.24	0.9217	1.0753	0.0027	0.000×10^0	0.001
235.90	0.7667	0.8442	0.0027	0.000×10^0	0.001
296.31	0.6104	0.6885	0.0027	0.000×10^0	0.001
355.78	0.5084	0.5594	0.0027	0.000×10^0	0.001
509.93	0.3547	0.4315	0.0027	0.000×10^0	0.001
696.82	0.3036	0.3291	0.0027	0.000×10^0	0.001
661.52	0.2734	0.2885	0.0027	9.224×10^{-4}	0.001
744.88	0.2428	0.2581	0.0027	2.987×10^{-2}	0.001
852.87	0.2121	0.2274	0.0065	7.081×10^{-2}	0.067
1023.32	0.1767	0.1944	0.0124	9.062×10^{-2}	0.188
1194.50	0.1514	0.1641	0.0197	1.310×10^{-1}	0.367
1493.38	0.1211	0.1363	0.0363	2.812×10^{-1}	0.852
1790.61	0.1010	0.1111	0.0719	7.233×10^{-1}	2.136
1885.18	0.0959	0.0985	0.0898	9.590×10^{-1}	2.861
1989.34	0.0909	0.0934	0.1115	1.398×10^0	3.794
2106.73	0.0859	0.0884	0.1457	2.079×10^0	5.338
2239.42	0.0808	0.0833	0.2124	2.941×10^0	8.540
2389.16	0.0757	0.0782	0.3139	3.826×10^0	13.732
2558.34	0.0707	0.0732	0.4419	4.379×10^0	20.725
2756.33	0.0656	0.0682	0.5988	4.393×10^0	29.937
2984.98	0.0606	0.0631	0.7485	3.855×10^0	39.426
3581.30	0.0505	0.0555	0.9796	2.212×10^0	56.064
3980.18	0.0454	0.0480	1.0642	1.681×10^0	63.123
4472.32	0.0404	0.0429	1.1394	1.376×10^0	70.127
5109.63	0.0354	0.0379	1.2117	1.144×10^0	77.750
5963.41	0.0303	0.0329	1.2816	9.770×10^{-1}	86.262

Figure A2
Typical Data Report Micromeritics Autopore IV 9500 V1.09 Page 2

LP Analysis Time: 9/1/2010 3:46:54AM
 HP Analysis Time: 9/1/2010 7:20:36AM
 Report Time: 12/1/2010 7:45:50AM

Sample Weight: 0.1599 g
 Correction Type: None
 Show Neg. Int: No

Tabular Report					
Pressure (psia)	Pore Diameter (μm)	Mean Diameter (μm)	Cumulative Pore Volume (mL/g)	Smoothed dV/dlogD Pore Volume (mL/g)	Cumulative Pore Area (m ² /g)
6167.54	0.0293	0.0298	1.2960	9.462×10^{-1}	88.187
6384.34	0.0283	0.0288	1.3097	9.137×10^{-1}	90.086
6625.13	0.0273	0.0278	1.3243	8.794×10^{-1}	92.191
6876.63	0.0263	0.0268	1.3376	8.466×10^{-1}	94.179
7150.24	0.0253	0.0258	1.3518	8.138×10^{-1}	96.380
7451.42	0.0243	0.0248	1.3661	7.818×10^{-1}	98.680
7773.50	0.0233	0.0238	1.3804	7.529×10^{-1}	101.087
8130.27	0.0222	0.0228	1.3946	7.246×10^{-1}	103.598
8518.07	0.0212	0.0217	1.4086	6.956×10^{-1}	106.165
8947.94	0.0202	0.0207	1.4234	6.678×10^{-1}	109.030
9416.29	0.0192	0.0197	1.4378	6.441×10^{-1}	111.952
9943.22	0.0182	0.0187	1.4527	6.191×10^{-1}	115.123
10532.52	0.0172	0.0177	1.4678	5.938×10^{-1}	118.549
11190.90	0.0162	0.0167	1.4832	5.723×10^{-1}	122.241
11940.45	0.0151	0.0157	1.4988	5.509×10^{-1}	126.231
12793.60	0.0141	0.0146	1.5151	5.300×10^{-1}	130.676
13782.48	0.0131	0.0136	1.5319	5.116×10^{-1}	135.600
15317.84	0.0118	0.0125	1.5547	4.905×10^{-1}	142.923
16289.49	0.0111	0.0115	1.5677	4.791×10^{-1}	147.482
17921.77	0.0101	0.0106	1.5872	4.531×10^{-1}	154.838
19914.04	0.0091	0.0096	1.6072	4.129×10^{-1}	163.163
22419.02	0.0081	0.0086	1.6271	3.581×10^{-1}	172.464
25627.56	0.0071	0.0076	1.6460	2.919×10^{-1}	182.444
29901.32	0.0060	0.0066	1.6631	2.285×10^{-1}	192.905
35881.74	0.0050	0.0055	1.6787	1.634×10^{-1}	204.122
44853.96	0.0040	0.0045	1.6912	1.072×10^{-1}	215.183
59943.95	0.0030	0.0035	1.7021	7.531×10^{-2}	227.595

Figure A3
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LP Analysis Time: 9/1/2010 3:46:54AM
HP Analysis Time: 9/1/2010 7:20:36AM
Report Time: 12/1/2010 7:45:50AM

Sample Weight: 0.1599 g
Correction Type: None
Show Neg. Int: No

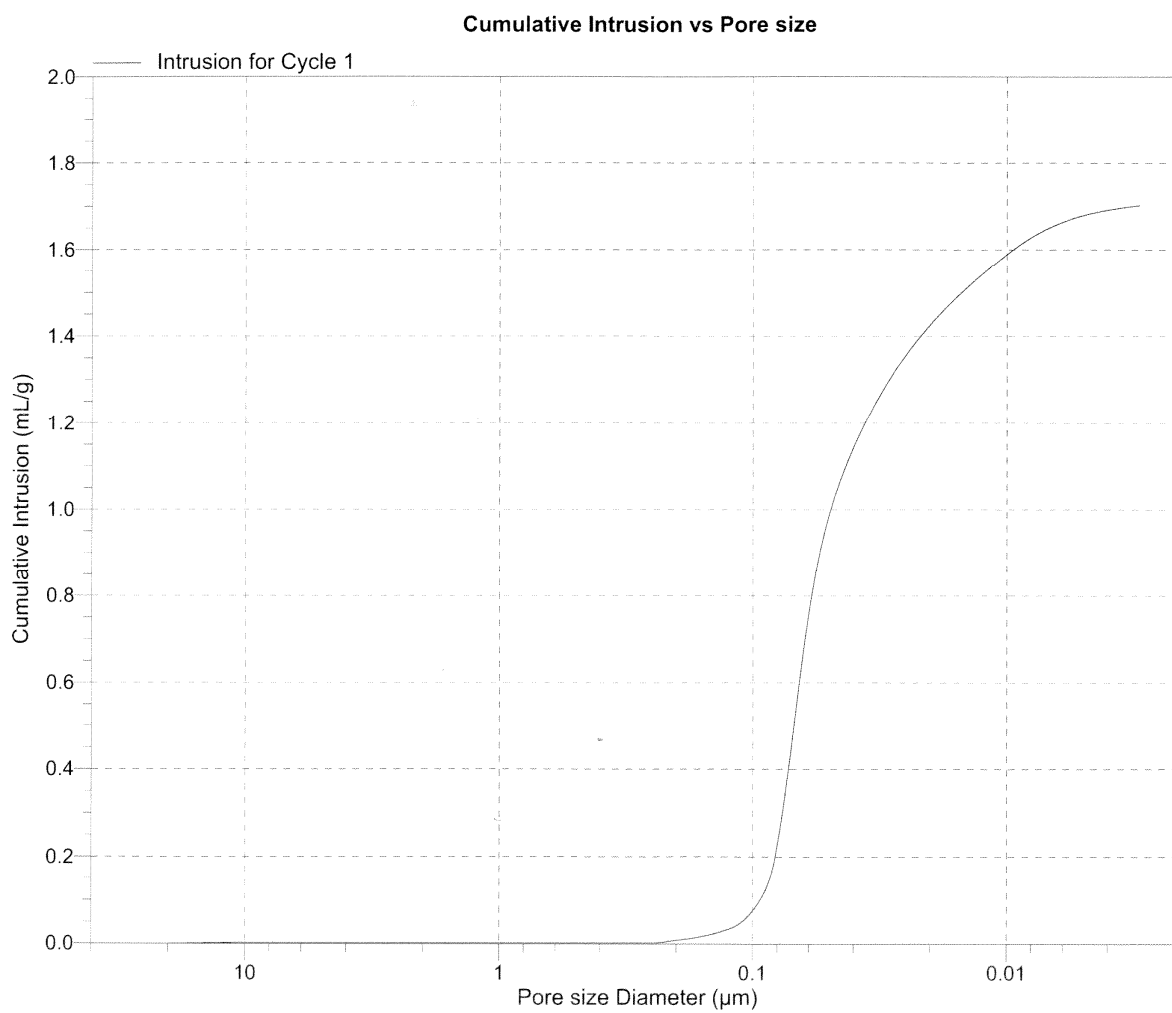


Figure A4
Typical Data Report Micromeritics Autopore IV 9500 V1.09 Page 4

LP Analysis Time: 9/1/2010 3:46:54AM
HP Analysis Time: 9/1/2010 7:20:36AM
Report Time: 12/1/2010 7:45:50AM

Sample Weight: 0.1599 g
Correction Type: None
Show Neg. Int: No

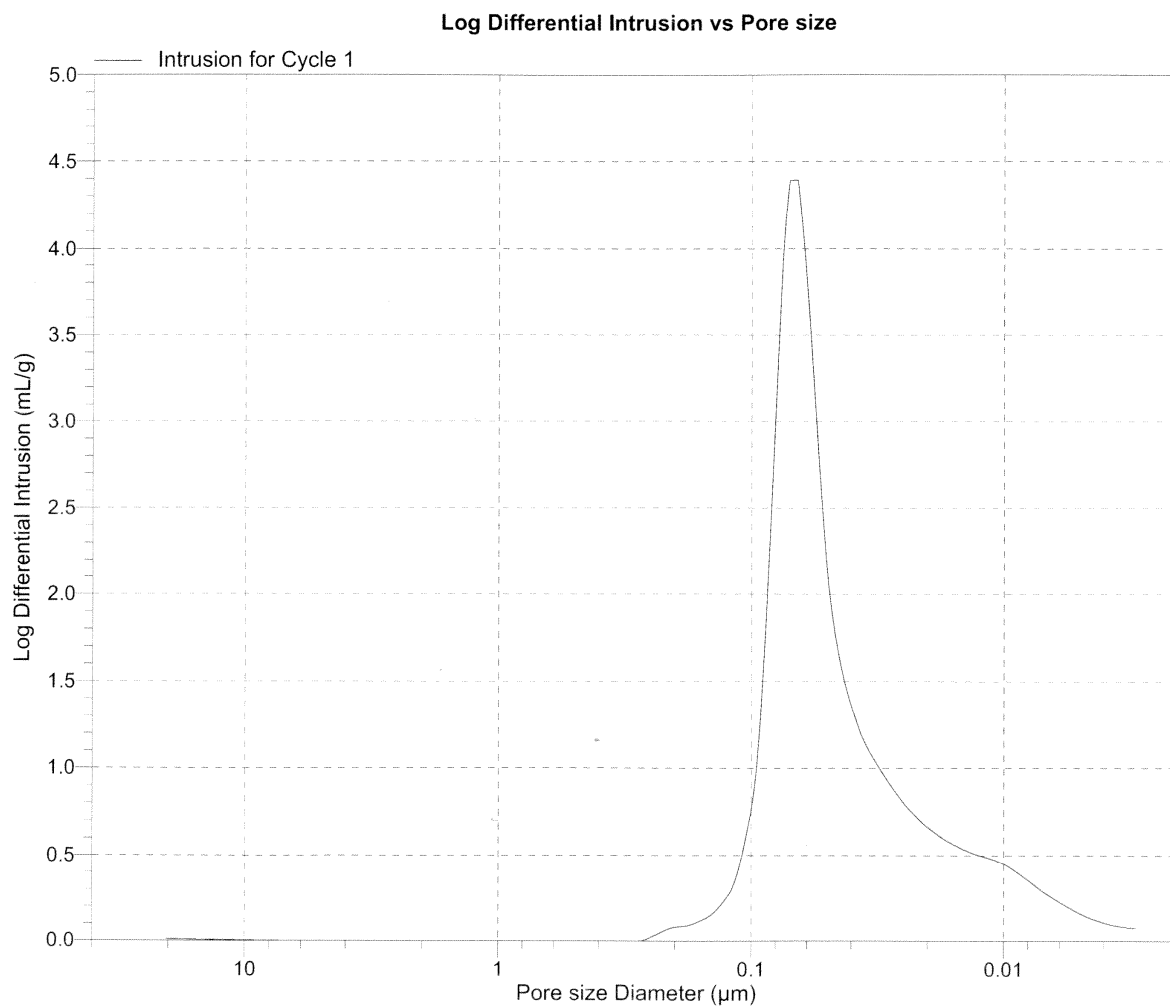


Figure A5
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