

PORE SIZE DISTRIBUTION OF POROUS SUBSTANCES BY NITROGEN ADSORPTION USING A QUANTACHROME ANALYZER

UOP Method 874-88

SCOPE

This method is for determining the surface area, total pore volume, average pore radius, and pore radius distribution from 1 to approximately 30 nm (10 to 300 angstroms) of porous materials. The method describes the standard procedure for analysis and data collection using a Quantachrome Autosorb-1 or Autosorb-6 analyzer.

OUTLINE OF METHOD

The sample is degassed using heat and vacuum. After weighing, it is cooled in liquid nitrogen. The amount of nitrogen gas adsorbed on the sample is measured at various predetermined pressures. The built-in computer processes the data and calculates surface area, total pore volume, average pore radius and pore radius distribution based upon a twenty-point adsorption isotherm. The results are printed and/or plotted as desired.

APPARATUS

Balance, readability 0.1-mg, high weighing chamber to accommodate 27-cm cells, Sartorius Model No. R300S, Sargent-Welch Scientific, Cat. No. S-2700-14M, or equivalent

Cells, pellet, 4-mm ID stem, 25-mm spherical bulb, Quantachrome Corporation, Cat. No. 74028, or equivalent

Regulator, helium, two-stage, high purity, Matheson, Model 3104-580, or equivalent

Regulator, nitrogen, two-stage, high purity, Matheson, Model 3104-580, or equivalent

Sorption analyzer, automatic, Quantachrome Corporation, Autosorb-1 or Autosorb-6, or equivalent. The designations -1 and -6 refer to the number of analytical stations on the instrument.

Stem inserts, glass rod, void volume reducers for 4-mm ID stem, Quantachrome Corporation, Cat. No. 74029, or equivalent (optional)

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).

Stoppers, rubber, micro, Sargent-Welch Scientific, Cat. No. S-73460, or equivalent

REAGENTS AND MATERIALS

All reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, when such specifications are available, unless otherwise specified. References to water mean deionized or distilled water, except where noted.

Boiling chips, carborundum, Thomas Scientific, Cat. No. 1590-D30, or equivalent

Glass wool, lead-free, borosilicate glass fiber, Sargent-Welch Scientific, Cat. No. S-40225, or equivalent

Helium, 99.995% minimum purity

Nitrogen, gas, 99.997% minimum purity

Nitrogen, liquefied

PROCEDURE

Install and initialize the instrument according to the directions in the Autosorb Operation manual¹. The instructions in the manual are essential for correct operation of the instrument and should be fully understood before any attempt is made to analyze samples on the Autosorb-1 or Autosorb-6.

Sample Cell Tare Mass Determination

Attach clean, empty cells (with stem inserts if desired) to the degas positions of the instrument. On the degas manifold control panel, place the manual-auto switch in the "AUTO" position. The "READY" light should be on. Press the "LOAD" button and then the "SELECT" pushbuttons on the individual stations holding the cells to be evacuated. The indicator lights should be illuminated on the pushbuttons. When the selections have been made, press the "START" button to start the degas procedure. Evacuate the empty cells for 5 ± 1 minutes. It is not necessary to heat the cells. At the end of the evacuation period, press the "REMOVE" button and then the selector buttons for the stations containing the empty cells. All the pushbuttons should light. Backfill the cells with helium by pressing the "START" button. When the instrument has completed the operation, remove the cells and stopper immediately (see *NOTE*). Weigh to the nearest 0.1 mg. Record the mass of the helium-filled cell, stopper and stem insert. This is the sample cell tare mass. Use the same stopper and stem insert for the subsequent run.

Sample Pretreatment

Place a representative portion of the sample in the tared cell. The amount of sample to be used varies with the expected surface area of the material. The manufacturer recommends adjusting the sample mass so that between 20 and 50 m² of surface are present in the cell. Therefore, the greater the expected surface area the less sample required. For most catalysts, 0.20 ± 0.02 g is a good approximation.

Place heating mantles around each cell and hold them in place with the metal clips. Attach the cell to a port on the degas manifold. After all the desired samples have been attached, press the "LOAD" button and then the "SELECT" pushbuttons for the stations loaded. All selected pushbuttons should light. Set the pretreatment temperature at 300 C on the digital temperature dials above the stations. A lower temperature is to be used only for samples that may degrade at 300 C. The degas temperature should be clearly stated on

the printout. Turn on the toggle switch for the heater power. Press the “START” button to begin the degassing procedure. The computer will automatically reduce the pressure in the cell slowly and activate the heater at the appropriate time.

A modified procedure is used for very finely divided powdered samples that may elutriate up the stem and clog the filter disk in the pretreatment port. This applies to zeolites, light powders, and samples that are suspected of containing large amounts of moisture or light hydrocarbons. For this procedure, use a sample cell that has been tared with a stem insert. After the sample has been introduced into the cell, replace the stem insert and place a small, loosely-fitting plug of glass wool in the neck of the cell. Set the initial temperature to 90 C. Evacuate at this temperature for 10 ± 1 min. Increase the temperature to 100 C and hold for another 10 min. Repeat this procedure for 110 C. At this point, all the water has effectively been removed. Increase the temperature to 300 C. Alternatively, a contaminated sample can be dried in a vacuum oven for 2 to 4 hours at 150 C followed by the usual pretreatment on the instrument.

After a minimum of two hours, check the sample to ensure that degassing has been completed. Zeolites and other moisture absorbing samples will require a minimum of four hours. Where possible, pretreatment overnight is preferred. Press the “TEST” button and the “SELECT” pushbutton for the cell to be tested. Press the “START” button. All other stations are isolated from the manifold and the manifold connection to the vacuum pump is closed. If the manifold pressure is stable or rises very slowly (less than two millitorr/min), the sample is adequately degassed and may be analyzed. If not, repeat the degassing procedure.

Turn off the heater toggle switch and remove the heating mantle from the cell. Allow the cell to cool to room temperature. Press the “REMOVE” button and the “SELECT” buttons for the cells to be removed. Press the “START” button and allow each sample cell to be backfilled with helium before removing from the manifold. Remove the glass wool plugs if used, and stopper each cell immediately with the same stopper used during the calibration. Weigh to the nearest 0.1 mg. Subtract the tare mass to obtain the degassed sample mass.

Sample Analysis

Some Autosorb-6 instruments require a separate saturation vapor pressure measurement station. The Autosorb-1 and later models of the Autosorb-6 do not. If a separate reference station is required, place a clean, empty cell on one of the analysis stations. Station 6 is normally used as the reference station but any station can be used. Unstopper the sample cells and quickly connect them to the stations.

Fill the dewars with fresh liquid nitrogen and add a few beads of high surface area material (such as catalyst spheres or boiling chips) to the dewars as boiling chips. The level and age of the liquid nitrogen used on all stations including the P_0 station must be the same, or errors in the total pore volume can be introduced. The large dewars hold enough liquid nitrogen for an analysis time of about 60 hours and *should not* be refilled during analysis.

Select the command “P” from the main menu. The P_0 menu will be displayed. Select option “2”, the P_0 station. Select the leak test option and set the time for five minutes. Answer the last question with “Y” (Yes). The instrument will fill the empty cell with nitrogen. A small amount of the nitrogen in the cell is liquefied and the pressure of the vapor above the liquid is used as the reference pressure.

To start the analyses, select the command “I” (Initialize) from the main menu on the terminal. Select an available analysis station. Enter the sample identification information (limit 50 characters). For example:

Requestor, Sample ID Number, Test Number, Date, Pretreatment Temperature,
HALLORAN 5331-34 TEST 2345 11/02/88 300C.

Enter the degassed sample mass previously obtained.

If the instrument has been upgraded with a PC software package, the following instructions can be set as default conditions in a separate program. Follow the manufacturer's instructions for preparing and using this program. Enter "Y" for leak test option and specify five minutes. In response to the "FAST PUMPDOWN Y/N?" option, select "N" for fine powders that might elutriate under rapid evacuation and "Y" for larger particles. The "SELECT EQUILIBRATION TIME" question sets the analysis parameter used by the instrument to determine how long the pressure must be stable (1 to 5 minutes) before the instrument collects the data and starts adding nitrogen for the next point. Select "3" (three minutes). The "SELECT P/P₀ TOLERANCE" option determines how closely the actual pressure points will be to the preset table values. A value of "3" (+0.012 or -0.004) is a satisfactory compromise between speed and precision. If the final question, "ALL SELECTIONS OK (Y/N)?" is answered "N" all entered information on that station will erase so errors can be corrected. If answered "Y", the analysis menu will be displayed.

The analysis menu allows selection of the pressure points that the instrument will use for the analysis. The selections are divided into the following categories: surface area, pore volume, number of points for adsorption or desorption, pore radius size distribution, and two user specified analyses. To save the pressure points for the standard analysis in one of the user defined protected memory locations of the instrument, select the "I" (Initialize) command from the main menu. Enter dummy data for the sample ID, mass and the analysis parameters. At the "ALL SELECTIONS OK Y/N?" type "Y" to enter the analysis menu. Move the cursor to the "5 POINT BET (.10, .15, .20, .25, .30)" and press "RETURN". Move the cursor to "20 POINT ADSORPTION" and again press "RETURN". Enter "E" to access to Edit mode. This allows the points to be edited for the analysis. Select option "I" to enter a new data point and then option "2" and follow the screen instructions to add 0.967 to the list of adsorption points. Exit the add points mode back to the Edit mode and change the total pore Volume "V" indicator to the 0.967 point. Remove the Langmuir "L" designations from all of the points except those marked by an "M" for multipoint BET (.10, .15, .20, .25, .30). Once the list has been edited, select option "U" (Save Analysis) and save the analysis in location 1. Exit the Edit mode by entering "X". Answer "N" to "START ANALYSIS ON STATION n (Y/N)?" This returns the instrument to the main menu. The standard table pressure point values and their functions are given in Table 1. For surface area analysis only, select the five-point surface area option. For the standard analysis, select the option "USER SPECIFIED ANALYSIS NUMBER 1" from the analysis menu.

Table 1
Measurement Points for Standard Analysis

<u>P/P₀</u>	<u>Description</u>	
.05	A	P
.10	AML	P
.15	AML	P
.20	AML	P
.25	AML	P
.30	AML	P
.35	A	P
.40	A	P
.45	A	P
.50	A	P
.55	A	P
.60	A	P
.65	A	P
.70	A	P
.75	A	P
.80	A	P
.85	A	P
.90	A	P
.95	A	P
.967	A	VP

where:

- A** = Adsorption point
- M** = Multipoint BET point
- L** = Langmuir point
- V** = total pore Volume point
- P** = Pore size distribution point

Move the cursor to the desired option(s) and press RETURN to select the option. After selecting the desired options, the command "E" will display the pressure point table and enable editing of the table for special analyses; the command "X" will exit the analysis menu and prepare the station for analysis. Answering "Y" to the final "START ANALYSIS ON STATION n (Y/N)?" question will start the analysis, and answering the question with "N" will display the main menu, and the data entered will be lost. Similar steps should be performed for the other available stations. If PC software is used with the instrument, the PC should be left in data acquisition mode so that it will automatically print graphs at the completion of the run. Follow the manufacture's instructions.

Once started, the computer will control all operations for complete data collection under normal circumstances. Abnormal circumstances, such as power failure or instrument malfunction, will cause error messages to be displayed on the terminal and activate an audible alarm. Refer to the manufacturer's instruction manual for suggested troubleshooting procedures.

Instrument Maintenance

The following is a schedule of routine maintenance for the instrument:

1. Check gas pressures daily.
2. Empty dewars and refill with *fresh* liquid nitrogen every three days.
3. Drain moisture traps from compressor weekly.
4. Check o-rings on all stations for powder build-up every two weeks. Do this more often if primarily running powders.
5. Change filter gaskets on all stations every three months or sooner if large volume of powders are run.
6. Change pump oil every three months.

CALCULATIONS

All calculations are performed by the computer and the results are displayed on the terminal. These results can be subsequently printed or plotted as desired. If the instrument has been equipped with PC software, follow the manufacturer's instruction for report and graphics printing.

The surface area of the material is calculated by application of the Langmuir² and BET³ (Brunauer, Emmet and Teller) equations. Five points in the range of 0.1 to 0.3 P/P_0 are used in the analysis. A correlation coefficient ("CORR") is reported for both equations. The equation whose correlation coefficient is closer to 1.000 is the better fit for the data. A negative value for the "C" constant in the BET report is another indication that the Langmuir is the more correct equation.

The pore radius distribution of the sample is calculated from the volume of nitrogen gas adsorbed on the cooled samples at the selected pressures. All pressures are referenced to the saturation vapor pressure of liquid nitrogen as determined by the P_0 sensor. This will change during the analyses because of changing atmospheric pressure or changes in the composition of the liquid nitrogen cooling bath. The instrument determines the P_0 reading approximately every 40 seconds during the run and automatically updates the value. The pore radius calculations are performed by the method of Barrett, Joyner and Halenda⁴. The calculations use the actual data points collected by the instrument; interpolated values are not used. Summaries of the theoretical basis of these calculation techniques are given in the instrument manual¹.

Type "D" at the main menu and specify a station number. The "DATA REDUCTION MENU FOR STATION n" message will then be displayed on the terminal. Select the "3" (EDIT) option to display the points selected for the "M" (multipoint BET) AND "L" (Langmuir) calculations. Ensure that the same five points are selected for both calculations (.10, .15, .20, .25, .30). Check that the Total Pore Volume symbol, "V", is associated with a point near 0.967 P/P_0 . Return to the Data Reduction menu and select option "4", to generate reports. Once in the Report Menu, move the cursor by using the up and down arrow keys and press "RETURN" to select the following options: "MULTIPOINT BET," "TOTAL PORE VOLUME," "AVERAGE PORE RADIUS," "LANGMUIR SURFACE AREA," "ISOTHERM," "ADSORPTION PORE SIZE DISTRIBUTION" and "LANGMUIR ISOTHERM". Print these data tables by answering "X" (to Exit) and "Y" (Yes) to the questions.

After printing out the data report, plot the V vs. P/P_0 isotherm and pore size distributions. Place a piece of preprinted isotherm paper in the plotter, insert an uncapped pen in the first station of the holder, and turn the plotter on. From the DATA REDUCTION menu, select option “6” (Initialize Plotter). Move the pen by the arrow keys on the *plotter* (not the terminal) so that it is directly above the origin on the preprinted paper. Return to the Data Reduction menu and select option “5” (Generate Plots). From the PLOT menu, move the cursor to select the “ V vs. P/P_0 ” (isotherm) option and hit “RETURN”. The terminal will display the default y-axis range for the plot. Select option “2” (autoscale to maximum value) and ensure that a piece of isotherm paper is in the plotter. Answer the two questions with “Y” to print out the plot labels and to connect the points on the graph. The plotter will then plot the isotherm. Examine the isotherm plot to make certain that it is a smooth curve with no points out of line. Analyses with points that do not fall on a smooth curve must be rerun.

To print or plot the pore size distribution, follow the same procedure but select the “D(v)r - ADS” (Pore Size Distribution-Adsorption) from the plot menu and place a piece of preprinted pore size distribution log paper on the plotter. The plot is made using one of five preselected y-axis scales from Table 2. Choose the value that is just larger than the maximum value displayed on the terminal screen.

Table 2

Y-Scale for Pore Size Distribution Plot

<u>Upper Limit</u>	<u>Scientific Notation</u>
0.0005	0.5E-03
0.0025	0.25E-02
0.005	0.5E-02
0.0150	0.15E-01
0.05	0.5E-01

Select option “1” to enter the chosen number for the full-scale value. Answer “Y” to the two questions and the plotter will automatically record the calculated pore size distribution. Collate the plots with the corresponding printout for the same sample.

All remaining data reports are generated in the same manner. Before starting new analyses, ensure that all data have been printed out. Without a PC data acquisition package, data stored on a station is erased when the station is initialized for another run.

REPORT

An example of a typical data report is given in the *APPENDIX*. The first several lines of the printout consist of the station number, sample identification and mass, and instrumental parameters for the analysis, including the total elapsed time. The next section contains the summary properties of the material, including the multipoint BET and Langmuir surface area, the total pore volume (for pores less than 30 nm [300 Å] radius) and average pore radius of the sample. The isotherm printout appears next, listing the actual pressure points taken and the normalized amount (cc/g) of gas adsorbed. This table contains the same data that are used for the isotherm plot.

Following the isotherm printout is the pore size distribution table. The radius values are calculated from the actual pressure points. The table is divided into seven columns. The first column lists the pore radii calculated by the Kelvin equation from the pressure points in the isotherm. Several of the low pressure points are omitted from the calculation because the equations used are not valid for pore radii of less than 1 nm (10 Å). The second column, labeled V_p , is the cumulative pore volume calculated for pores of various radii. The third column (S_p) is the cumulative pore surface area (assuming cylindrical pores) calculated for the pores. The calculated total pore volume at 30 nm (300 Å) radius and the calculated cumulative surface area should be compared to that actually measured and given earlier in the printout. A large negative discrepancy between the tabulated value and the measured value usually indicates the presence of many micropores below the limit of resolution of the calculation, 1 nm (10 Å). The fourth and fifth columns list the slopes of the cumulative volume [$Dv(r)$] and surface area [$Ds(r)$] curves, $d(\text{volume})/d(\text{radius})$ vs. radius and $d(\text{surface area})/d(\text{radius})$ vs. radius. This is the change in pore volume and surface area with respect to the change in radius plotted against the radius. The pores in the upper size ranges tend to be underrepresented because the pore volume or surface area is divided by larger radii ranges. To partially compensate for this, the values in columns six and seven are $d(\text{volume})/d(\text{radius})$ vs. $\log \text{radius}$ and $d(\text{surface area})/d(\text{radius})$ vs. $\log \text{radius}$. Any of columns 2-7 can be plotted by using the plot menu described in the *CALCULATIONS* section. Examples of the two plots normally generated are shown as part of the *APPENDIX*.

NOTE

If a second helium source, apart from the instrument, is available, an alternative procedure can be used to fill the cells. Insert a thin piece of tubing, connected to a helium source, to the bottom of an inverted cell. Displace the air in the cell by flushing with helium for approximately 30 seconds. Remove the tube and immediately stopper the cell.

PRECISION

Repeatability

Based on two tests performed by each of two analysts on each of two different days (8 tests per lab) and using data collected from each of 3 laboratories, the within-laboratory estimated standard deviation (esd) was calculated for Surface Area and Pore Volume for two types of catalysts, at the levels listed in Table 3. Two tests performed in one laboratory by different analysts on different days should not differ by more than the allowable difference shown in Table 3 at the levels listed (95% probability).

Table 3

	Within-Lab, esd	Allowable Difference
Surface Area, m²/g		
680	7.2	22
180	1.8	5
Pore Volume, cc/g		
0.37	0.0063	0.02
0.80	0.0140	0.04

Reproducibility

Two tests performed in different laboratories by different analysts on different days should not differ by more than the allowable difference shown in Table 4 at the levels listed (95% probability).

	Table 4	
	Between-Labs, esd	Allowable Difference
Surface Area, m²/g		
680	11.1	68
180	3.0	18
Pore Volume, cc/g		
0.37	0.0082	0.05
0.30	0.0213	0.13

The surface area of the higher surface area catalyst was calculated according to the Langmuir equation; the lower, according to the BET equation. However, the precision stated applies regardless of which equation is used.

TIME FOR ANALYSIS

The elapsed time for an analysis varies with the degree of macroporosity of the sample. Typical elapsed times for catalyst samples are in the range of 12-20 hours.

The labor requirement is 1.0 hour per sample.

REFERENCES

1. Autosorb-1 or Autosorb-6 Instrument Manual, Quantachrome Corporation, supplied with instrument
2. I. Langmuir, *J. Amer. Chem. Soc.*, **40**, 1361 (1918)
3. S. Brunauer, P. Emmet and E. Teller, *J. Amer. Chem. Soc.*, **60**, 309 (1938)
4. E. Barrett, L. Joyner and P. Halenda, *J. Amer. Chem. Soc.*, **73**, 373 (1951)

SUGGESTED SUPPLIERS

Matheson, P.O. Box 96, Joliet, IL 60434
 Quantachrome Corp., 6 Aerial Way, Syosset, NY 11791
 Sargent-Welch Scientific Co., 7300 N. Linder Ave., Skokie, IL 60077
 Thomas Scientific, P.O. Box 99, Swedesboro, NJ 08085-0099

APPENDIX

REPORT FOR STATION 1
 SAMPLE ID REFERENCE CATALYST TEST-6251 11/3/88 300C
 SAMPLE WEIGHT = .2025 g
 P/Po TOLERANCE = 3
 EQUILIBRATION TIME = 3
 ANALYSIS TIME = 1142.9 MINUTES

MULTI POINT BET

P/Po	VOLUME (cc/g)	1/(W(Po/P-1))
.101	49.75	.18037E+01
.153	55.20	.26222E+01
.204	59.86	.34200E+01
.254	64.45	.42361E+01
.301	68.90	.50096E+01

AREA = .21560E+03 SQ M/g
 SLOPE = .15975E+02 INTERCEPT = .17983E+00 CORR = .9999 C = .89832E+02

TOTAL PORE VOLUME .110330E+01 (cc/g) FOR PORES LESS THAN 295.8 ANGSTROMS
 AT A P/Po OF .965

AVERAGE PORE RADIUS = .10235E+03 ANGSTROMS

LANGMUIR SURFACE = .37220E+03 SQ M/g CORR = .99625

ISOTHERM

P/Po	VOLUME (cc/g)	P/Po TOLERANCE	EQUILIBRATION TIME
.05379	42.94581	3	3
.10085	49.74829	3	3
.15321	55.19971	3	3
.20374	59.85679	3	3
.25442	64.44808	3	3
.30139	68.89552	3	3
.35461	74.28550	3	3
.40321	79.69095	3	3
.45099	85.51968	3	3
.50115	92.41714	3	3
.55214	100.65356	3	3
.60045	110.16466	3	3
.65025	122.46028	3	3
.69989	138.38933	3	3
.74953	161.00101	3	3
.79888	195.57452	3	3
.85009	249.89841	3	3
.89931	352.90506	3	3
.94948	597.02234	3	3
.96512	713.05066	3	3

ADSORPTION

PORE SIZE DISTRIBUTION

RADIUS A	Vp cc/g	Sp SQ M/g	Dv(r) cc/A-g	Ds(r) SQ M/A-g	Dv(LOG r) cc/g	Ds(LOG r) SQ M/g
9.77	.0000E+01	.0000E+01	.0000E+01	.0000E+01	.0000E+01	.0000E+01
11.04	.0000E+01	.0000E+01	.0000E+01	.0000E+01	.0000E+01	.0000E+01
12.34	.0000E+01	.0000E+01	.0000E+01	.0000E+01	.0000E+01	.0000E+01
13.78	.8862E-03	.1286E+01	.5531E-03	.8026E+00	.1753E-01	.2544E+02
15.40	.2809E-02	.3783E+01	.1172E-02	.1521E+01	.4152E-01	.5391E+02
17.14	.5629E-02	.7073E+01	.1539E-02	.1795E+01	.6067E-01	.7079E+02
19.17	.1001E-01	.1164E+02	.1960E-02	.2044E+01	.8642E-01	.9015E+02
21.64	.1683E-01	.1795E+02	.2524E-02	.2333E+01	.1256E+00	.1161E+03
24.56	.2678E-01	.2605E+02	.3189E-02	.2597E+01	.1801E+00	.1467E+03
28.13	.4214E-01	.3697E+02	.3816E-02	.2714E+01	.2468E+00	.1755E+03
32.75	.6490E-01	.5087E+02	.4362E-02	.2664E+01	.3282E+00	.2004E+03
38.92	.1018E+00	.6985E+02	.5181E-02	.2662E+01	.4630E+00	.2379E+03
47.66	.1641E+00	.9600E+02	.6024E-02	.2528E+01	.6585E+00	.2763E+03
61.59	.2649E+00	.1287E+03	.5751E-02	.1868E+01	.8101E+00	.2631E+03
86.76	.4607E+00	.1739E+03	.5966E-02	.1375E+01	.1177E+01	.2714E+03
151.53	.9071E+00	.2328E+03	.4616E-02	.6092E+00	.1554E+01	.2051E+03
242.82	.1111E+01	.2496E+03	.2379E-02	.1960E+00	.1316E+01	.1084E+03

LANGMUIR ISOTHERM

P/Po	P/Po/W
.101	.16218E+01
.153	.22205E+01
.204	.27232E+01
.254	.31583E+01
.301	.34998E+01

<

A-2

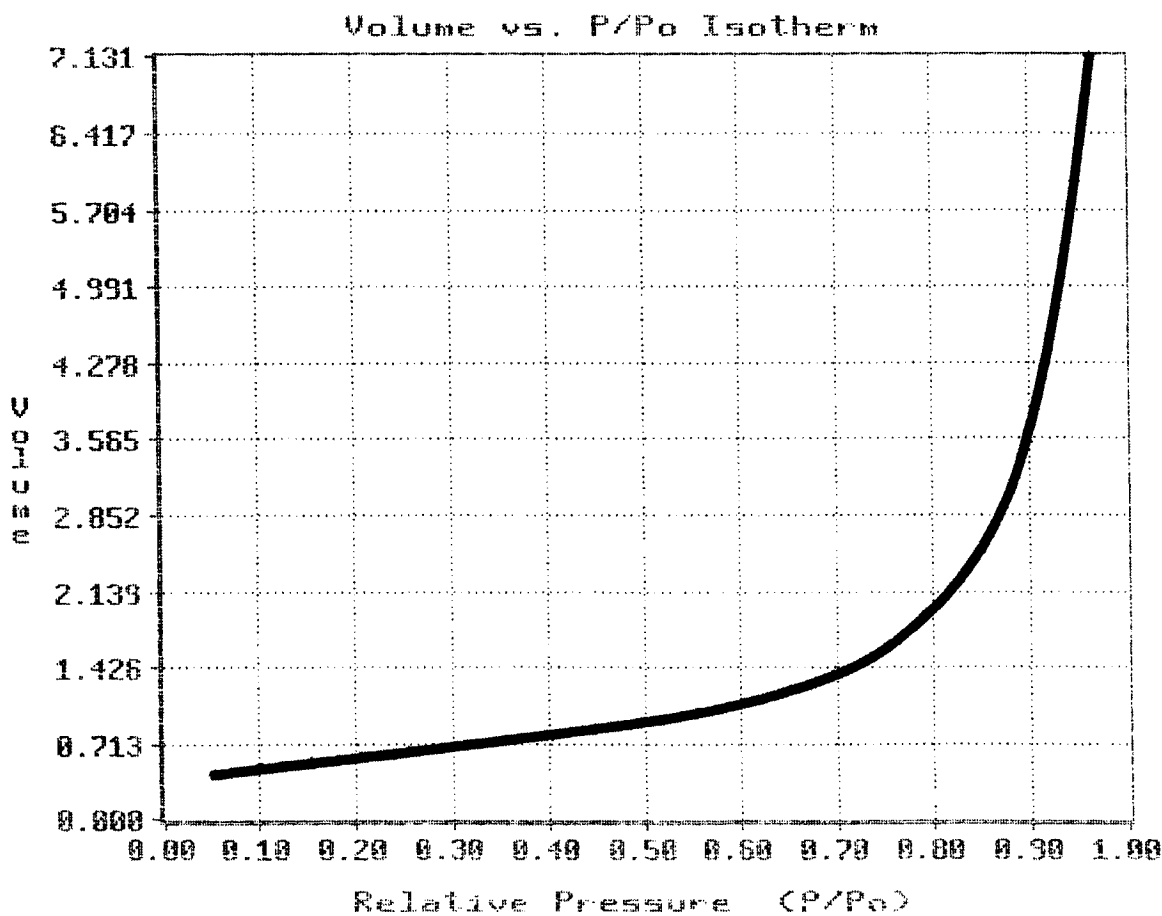
Quantachrome Corporation
Autosorb Automated Gas Adsorption System Report

Sample ID..... REFERENCE CATALYST TEST-6251 11/3/88 300C
Sample Desc.....
Comments.....

Sample Weight... 0.2025 gm
Analysis Time...1142.9 min
Outgas Time..... 16.0 hrs
Station Number...1

P/Po Toler...3
Equil Time...3
Outgas Temp...300C
End of Run...11-04-88 07:01 AM

Filename...AS8B0307.RPT
Operator...CLL
Date.....11/3/88



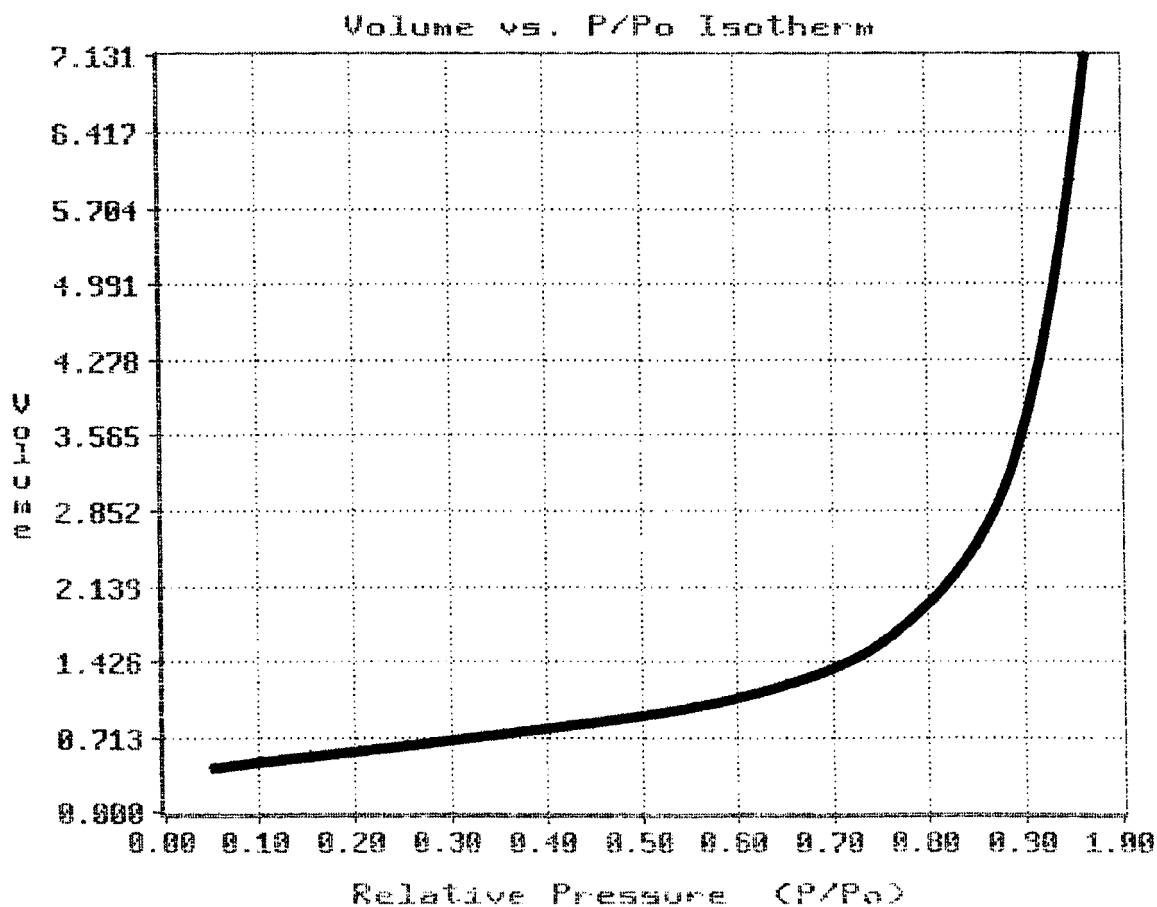
X axis scale units...
Y axis scale units... cu-cm/gram x10E2

A-3

Quantachrome Corporation
Autosorb Automated Gas Adsorption System Report

Sample ID..... REFERENCE CATALYST TEST-6251 11/3/88 300C
Sample Desc.....
Comments.....

Sample Weight... 0.2025 gm	P/Po Toler...3	Filename...AS8B0307.RPT
Analysis Time...1142.9 min	Equil Time...3	Operator...CLL
Outgas Time..... 16.0 hrs	Outgas Temp...300C	Date.....11/3/88
Station Number..1	End of Run...11-04-88 07:01 AM	



X axis scale units...
Y axis scale units... cu-cm/gram x10E2