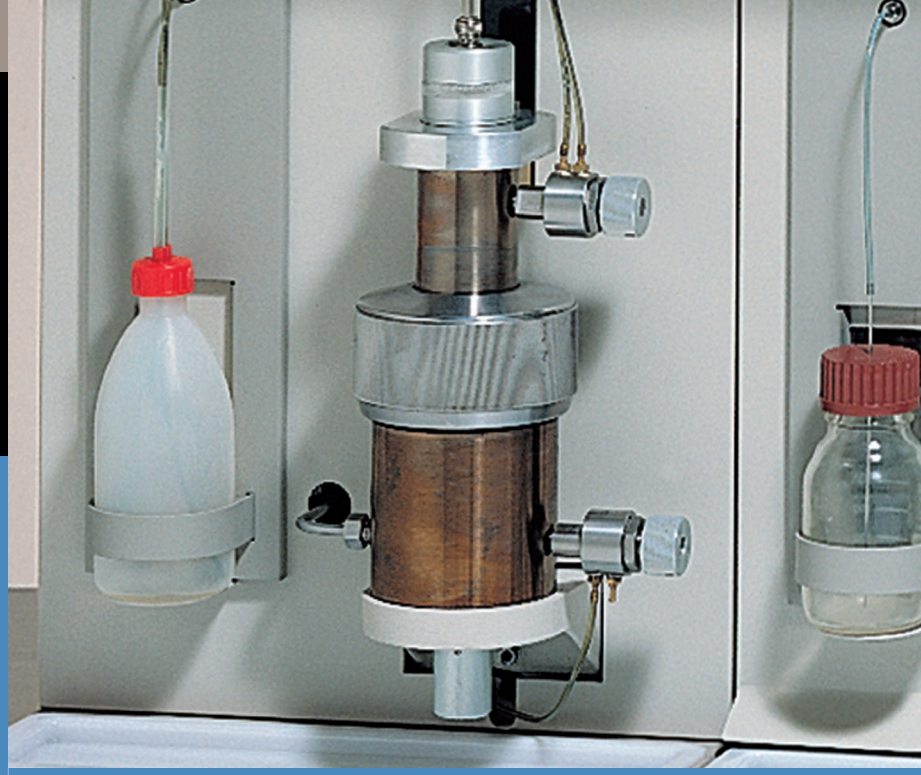
# Pascal Series
# Mercury Intrusion Porosimeters



***Maximum resolution in minimum time!***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Pascal Series
Mercury Intrusion Porosimeters
Maximum resolution in minimum time!

# Pascal 140, 240 and 440

## Maximum resolution in minimum time!

The mercury porosimetry technique is one of the most useful methods to investigate the porous structure of solid samples in a quantitative way. It provides reliable information about pore size/volume distribution, particle size distribution, bulk (envelope) density and specific surface for most porous solids.

Pressurization by Automatic Speed-up and Continuous Adjustment Logic, or, in short Pascal, is a special operating principle developed by Thermo Fisher Scientific and used in the new generation of automatic mercury porosimeters described herein.

### Pascal self-optimizes analysis parameters

Mercury porosimetry analysis is based on the measure of the intrusion of mercury into the pores of the sample at various pressures. The pressurization procedure is critical to the accuracy of the analysis because a certain equilibrium time is required for the mercury to fill the pore at each pressure. This depends on the external pore access diameter and on the shape and complexity of the pore network. In principle it is impossible to know which pressure rate is the most suitable for the sample as the pore size range and pore shape in different solid samples are unknown. An excessively high pressurization rate gives wrong results because the pores are not completely filled at the corresponding pressure, whereas a low pressurization rate wastes valuable laboratory time. The solution is Pascal.

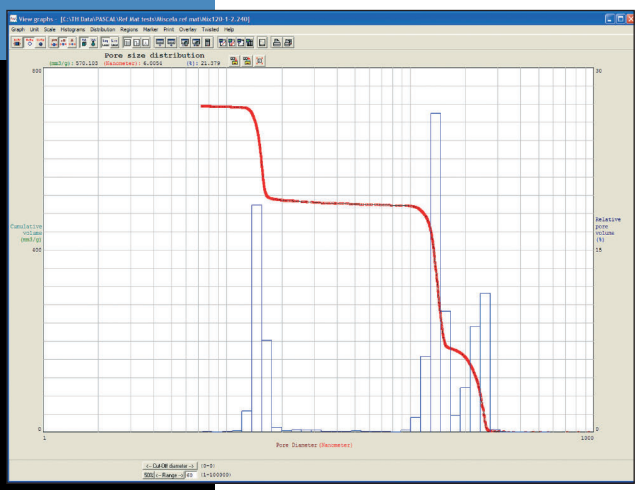
### Pascal does it faster than ever!

The pressurization starts "softly" and in absence of penetration increases quickly to the maximum predetermined speed. Nine (9) different speed characteristics are available covering different application fields and analytical purposes and making it possible to choose a balance between the scientific quality of the information and the total run time. When mercury intrusion is detected by the mercury level detector, the pressurization immediately slows down without stopping completely. The acceleration and deceleration of the pumping system are balanced to assure a constant pressure over the sample during the mercury intrusion into the pores with the same access size, thus providing proper equilibrium time.

### Pascal fits your sample

The Pascal method automatically determines the correct pressurization speed according to the presence of pores and to the actual mercury penetration rate thereby eliminating dead times during the analysis. This operation is performed at any pressure. The Pascal method combines all the benefits in one system:

- Optimum information quality at every speed and at every pressure
- Highest resolution
- Minimum analysis time




# Common features and benefits of the Pascal Series

## Pascal method of pressurization

Decreases run times by up to 30% without sacrificing the reliability of the curves and does not require any prior knowledge about the sample characteristics.

## Extremely high resolution

Pascal units can provide curves with up to 2500 data points depending on the selected speed and sample nature. At this incredibly high resolution the data points on the curve form a continuous line.

## Separately selectable speed ranges for intrusion and extrusion

Nine speed ranges are selectable so that both sophisticated researchers, who don't want to miss anything, and those who are in a hurry, as in the case of quality control, can be satisfied. Decreased speed ranges can be selected independently of increased speed ranges to better define the extrusion curve and minimize instrumental hysteresis effects.

## On-board microprocessor and memory data buffer for stand-alone operation

The on-board microprocessor controls all the instrument functions and collects the experimental data storing them in a memory buffer, therefore a PC is not required to run the Pascal but only for data elaboration. On the other hand the whole analysis can be programmed and controlled from a PC, which can be disconnected at any time during the analysis freeing it for other tasks. The operator can verify the instrument status and can transfer the experimental data to the PC to verify the analysis results at any time.

## One data station – four instruments via multi RS232 link

The instruments are connected to the PC through a standard serial RS232 port. A single data station can control up to four instruments from a serial port (Pascal 140/240/440 and the Sorptomatic for example). Thus a complete Microstructure Lab can be realized minimizing costs and bench space.

## Choice of operation via control panel or PC

Both the analysis and the instrument can be programmed and controlled in real time either from an easy-to-use control panel with liquid crystal display or from the computer screen. Thus the instruments can be operated even if they are not in the same room as the data station, as often occurs in QC labs.

## Automatic porosimeter calibration

A special calibration kit is available as an option to verify the calibration of the system. In addition, a number of certified reference materials are nowadays available to regularly check the instrument performance.

## Pascal porosimeters achieve utmost safety certifications

All Pascal devices are fully CE certified according to electromagnetic compatibility and safety. In addition, the high pressure devices achieved the full PED (Pressure Equipment Directive) certification regularly controlled by an external certification body. The utmost certified safety and unmatched analytical performance are the best added values of these products.

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# Pascal 140

## a fully automated low pressure porosimeter and sample preparation unit


**The Pascal 140 has a dual role:**
it prepares the sample and the dilatometer for the analysis and carries out low pressure porosimetry measurements

Operations are done automatically thus freeing the operator for other tasks. Thanks to its modular concept, it can be used by itself or together with other Pascal porosimeters, thus modularity of the Pascal system features a "buy what you need" approach. Data from the low pressure intrusion can be combined with data from the other high pressure modules (240 or 440) to get a complete porosity spectrum of the sample.

### Automated sample preparation, mercury filling and analysis

The sample preparation process is started by degassing the sample under vacuum for a certain time. A special proportional valve makes a "soft start" vacuum, avoiding any risk of powder elutriation. The vacuum is measured by a gauge whose signal is displayed on the instrument control panel. When the minimum vacuum is reached, the system proceeds with the mercury filling operation up to the required volume level. Finally, the analysis begins and the pressure is increased from vacuum at the rate determined by the Pascal system. Depressurization (extrusion) starts when the maximum pressure is reached and can proceed down to atmospheric pressure or vacuum. For powder samples, a second run can be made to assure there is no aggregation, thus permitting a correct particle size distribution measurement.

### Data collection and analysis

The experimental data are memorized by the on-board microprocessor and kept in the internal memory buffer. At any time during the analysis the operator can check the status of the analysis and/or transfer the equilibrium data to the PC. The low pressure analysis can be performed also simultaneously with the high pressure analyses (by the Pascal 240/440) on another sample. The Pascal 140 is characterized by its high level of automation which simplifies the operator's work and assures a very high reproducibility of results.


# Many reasons to choose **Pascal 140**

## Automatic sample degassing

All samples are pre-treated in the same way regardless of who is operating the equipment thus improving analytical reproducibility. A special design proportional valve generates an automatic soft-start vacuum avoiding any risk of dragging out light powders from the dilatometer.

## Digital vacuum gauge to control sample degassing

The accurate control and display of vacuum during sample degassing assures that samples are properly dried before starting the experiment.

## Automatic mercury filling

The amount of mercury introduced in the dilatometer is measured with the utmost accuracy assuring reliable and reproducible measurements of bulk and apparent densities.

## Double run for powders de-agglomeration

Powders can be properly analyzed in terms of particle size distribution avoiding agglomeration effects. The first pressurization run breaks all the agglomerates while the second run provides a penetration curve related only to the real particle size distribution.

## Special kit for ultra-macropore analysis

A special design dilatometer compensates the hydrostatic pressure over the sample and significantly increases the pore diameter or the particle size detectable by the instrument.

## Special vacuum circuit with mercury trap

Safety is designed into the Pascal 140. The vacuum circuit can be completely isolated from the ambient and the waste outlet can be connected to an external exhaust system. An additional mercury trap prevents any contamination of the internal tubing.

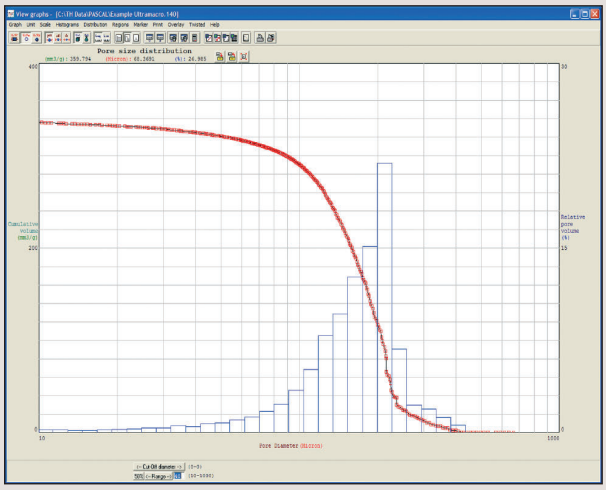
## Simple, easy to handle and inexpensive dilatometers

The five different dilatometers models can accept and measure samples of various dimensions and shapes. They are easy to mount and to clean avoiding any complexity that makes the operator's life difficult and leads to errors.

## Dilatometer in vertical position

The vertical position of the dilatometer prevents any mercury spilling during the filling operation and permits a continuous degassing, thus avoiding the risk of air bubble trapping in the dilatometer.

## Example of analysis with the Ultramacropore Option installed in the Pascal 140




Pore size distribution (custom ranges)

| Pore diameter ranges | Specific Volume | Relative Volume | Relative Volume | Specific Surface | Relative Surfac |
|---|---|---|---|---|---|
| (µM) | (mm3/g) | (mm3/g) | (%) | (m²/g) | (m²/g) |
| 495.6508-435.086 | 0.00 | 0.00 | 0 | 0 | 0 |
| 435.086-381.922 | 2.12 | 2.12 | .63 | 0 | 0 |
| 381.922-335.254 | 6.28 | 4.15 | 1.24 | .0001 | 0 |
| 335.254-294.2886 | 12.73 | 6.46 | 1.93 | .0001 | .0001 |
| 294.2886-258.3288 | 20.27 | 7.54 | 2.25 | .0003 | .0001 |
| 258.3288-226.763 | 43.02 | 22.75 | 6.79 | .0006 | .0004 |
| 226.763-199.0544 | 116.39 | 73.36 | 21.88 | .002 | .0014 |
| 199.0544-174.7316 | 167.13 | 50.74 | 15.13 | .0031 | .0011 |
| 174.7316-153.3806 | 209.53 | 42.39 | 12.64 | .0041 | .001 |
| 153.3806-134.6388 | 241.99 | 32.46 | 9.68 | .005 | .0009 |
| 134.6388-118.1868 | 268.40 | 26.41 | 7.88 | .0059 | .0008 |
| 118.1868-103.7454 | 285.69 | 17.29 | 5.16 | .0065 | .0006 |
| 103.7454-91.0684 | 297.20 | 11.51 | 3.43 | .007 | .0005 |
| 91.0684-79.9406 | 304.97 | 7.77 | 2.32 | .0073 | .0004 |
| 79.9406-70.1726 | 310.74 | 5.78 | 1.72 | .0076 | .0003 |
| 70.1726-61.598 | 315.03 | 4.29 | 1.28 | .0079 | .0003 |
| 61.598-54.0712 | 318.56 | 3.52 | 1.05 | .0081 | .0002 |
| 54.0712-47.464 | 321.22 | 2.66 | .79 | .0084 | .0002 |
| 47.464-41.6642 | 323.66 | 2.44 | .73 | .0086 | .0002 |
| 41.6642-36.5732 | 325.37 | 1.72 | .51 | .0087 | .0002 |
| 36.5732-32.1044 | 327.31 | 1.94 | .58 | .009 | .0002 |
| 32.1044-28.1814 | 328.58 | 1.26 | .38 | .0091 | .0002 |
| 28.1814-24.7378 | 329.89 | 1.31 | .39 | .0093 | .0002 |
| 24.7378-21.715 | 330.97 | 1.08 | .32 | .0095 | .0002 |
| 21.715-19.0618 | 331.87 | 0.90 | .27 | .0097 | .0002 |
| 19.0618-16.7326 | 332.64 | 0.77 | .23 | .0099 | .0002 |
| 16.7326-14.6878 | 333.23 | 0.59 | .18 | .01 | .0002 |
| 14.6878-12.8932 | 333.81 | 0.59 | .18 | .0102 | .0002 |
| 12.8932-11.3178 | 334.63 | 0.81 | .24 | .0105 | .0003 |
| 11.3178-9.9348 | 335.35 | 0.72 | .22 | .0107 | .0003 |

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# Pascal 240 and Pascal 440

## Speed and resolution to match any requirement

The Pascal 240 and Pascal 440 high pressure porosimeters take over where the Pascal 140 leaves off to measure pores down to the lower mesopore region. They incorporate a new pressurization system, developed to meet the sophisticated analytical requirements of laboratories working with modern materials. The pressurization system uses a reversible pump operating continuously, which permits a perfect control in increasing/decreasing the pressurization speed, and a new type of pressure multiplier. The key features of this system are the extremely high maximum speed of pressurization and the immediate acceleration or deceleration response permitting the optimal application of the Pascal system during the analysis. The easy-to-use control panel with liquid crystal display permits the unit to be operated also without a PC. It lets the operator program the analysis or the calibration and provides a useful guide to operate correctly. It shows the analysis status in real time and indicates incorrect operations by means of error messages.

### PED conformity assures utmost safety for high pressure devices

Pascal porosimeters assure to customers the utmost certified safety and maximum quality. Pascal 240 and 440 models, reaching 200 and 400 MPa respectively, are completely certified according to the PED (Pressure Equipment Directive) certification. PED is intended to guarantee essential safety requirements to users, and it is applied according to several parameters related to fluids, volume-pressure rates, materials, etc. In the case of Pascal 240 and 440, it is relevant to all the hydraulic parts submitted to high pressures, specifically the pressure multiplier, the high pressure inlet pipe and the two bodies of the autoclave. These components have been certified as a system, and single components are regularly verified by an official certification institute. Safety is the best added value of our products.

### Pascal 240

The Pascal 240 porosimeter operates up to the maximum pressure of 200 MPa. Its large autoclave together with the special electrode system makes it an extremely versatile porosimeter as it can accept all the dilatometers and is thus able to measure a wide range of solid materials: homogeneous, heterogeneous, low and high porosity. The accuracy and reproducibility are the utmost thanks to the Pascal system.

### Pascal 440

The Pascal 440 is not only the highest pressure model but it also offers the highest speed of the series. It reaches its maximum pressure of 400 MPa in the same time as the Pascal 240 reaches its maximum of 200 MPa. It is particularly suited for ceramics, sintered metals, very hard materials and, in general, for all the solids which have a

porosity approaching the micropore region. Thanks to its high pressurization speed, it is highly recommended in QC labs where short run times and productivity are the priorities.

# The Pascal 240 and 440
## offer more than meets the eye

### Automatic autoclave opening/closing

The motor driven raising/lowering of the autoclave simplifies the operation and contributes to the userfriendliness of the unit.

### Autoclave oil filling by a pumping system

The pumping system reduces the time necessary to fill the autoclave before the analysis begins and assures that air bubbles are removed.

### Real blank correction

The Pascal 240/440 permit a real "blank" analysis, that is a run without any sample. The blank curve is memorized by the data station and is then automatically subtracted from the normal analyses. This prevents erroneous interpretations of the sample porosity due to the presence of "ghost" pores which result when the mercury compression and temperature effect are not taken into consideration.

### Control panel with liquid crystal display

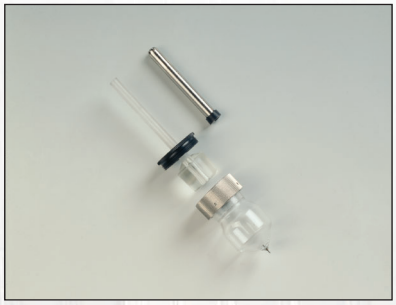
It informs the user about the instrument status in real time while the analysis is running regardless of whether or not the PC is connected. It increases safety by continuously monitoring the piston position and displaying error messages if anything goes wrong. This information can also be shown on the PC's screen.

### Up to 2500 experimental points in every run

This means an almost limitless curve resolution. When the low and high pressure analyses are joined the total number of points can be even more!

### "Hold pressure" function

The analysis can be stopped on request by the operator and the pressure is maintained constant at the actual value without time limits. This permits the study of extremely slow penetration phenomena.

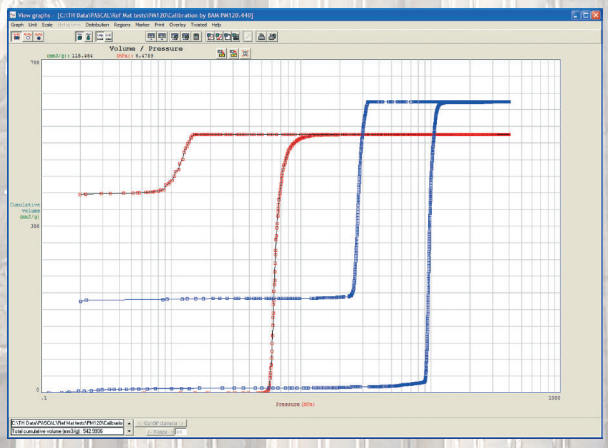


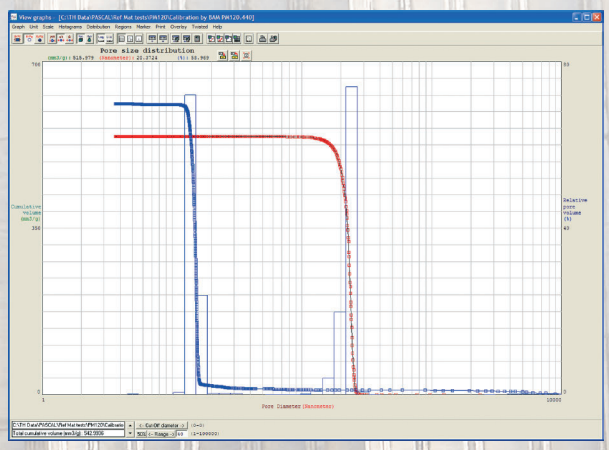
### Pascal 240 accepts all type dilatometers

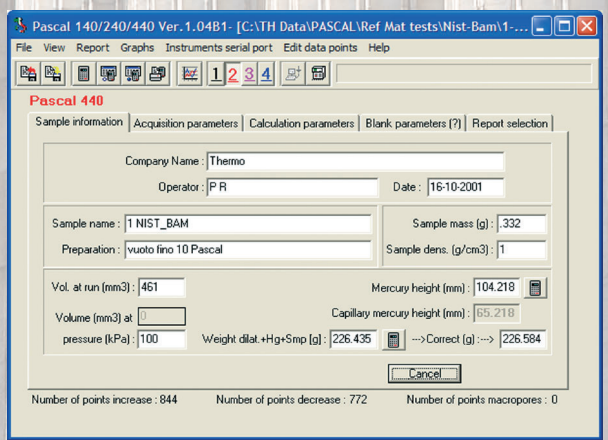
The Pascal 240 is exceptionally versatile due to its unusually large autoclave which accepts the complete range of dilatometers and thus can analyze practically any kind of sample.

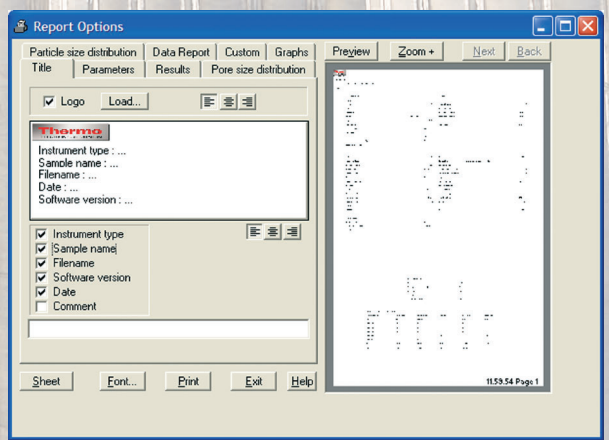
### Only 9 minutes to reach the maximum pressure

The incredibly short time required to reach the maximum pressure makes the Pascal 240 and Pascal 440 particularly suited for QC applications where short analysis times and high productivity are most important.



# Many ways to look at the data

## Calculations

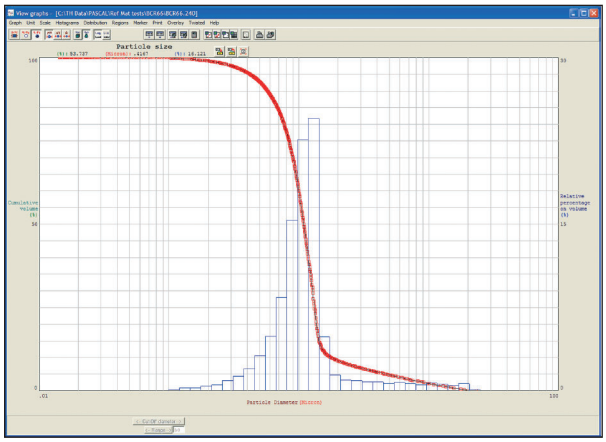
Bulk and apparent density, total and incremental pore volume, porosity, incremental specific surface area (four models), average pore size (two models), pore size distribution (derivative and histograms), particle size distribution (Mayer-Stowe model), pore and particle surface area distinction.

## Available graphs

Intrusion/extrusion, pore size distribution (cumulative and derivative), particle size distribution (cumulative and percentage), raw data plot, overlay of multiple curves.

## Data printout and reporting

Pore volume (raw, normalized, incremental and relative), intrusion/extrusion pressure (corrected), pore size (choice of three measuring units), surface area (normalized, incremental and relative), pore and particle percentage fraction in user defined intervals. Reporting can be customized by user and printed or saved in different electronic formats (i.e. Excel, text, etc.)




Sample name : BAM-PM-120
Instrument type : Pascal 440

| *** Sample information | *** | *** Acquisition parameters | *** | *** Calculation parameters | *** |
|---|---|---|---|---|---|
| Company Name : | Thermo | Max pressure : | 400 | Conctact angle (Deg) : | 140 |
| Operator : | P R | Pump speed increase : | 8 | Hg surface tension (Dyne/cm) : | 480 |
| Date : | 26/04/01 | Pump speed decrease : | 8 | Hg density (g/cm3) : | 13.533 |
| Sample name : | BAM-PM-120 | Pump speed inc. compensation : | Continuous | Temperature Hg (°C): | 25.4 |
| Preparation : | vuoto fino 10 Pascal | Pump speed dec. compensation : | Continuous | Pores model : | Cylindrical |
| Sample mass (g) : | .314 | Dilatometer number : | 1 | Sample volume correction : | Yes |
| Sample density (g) : | 1 | Dilatometer type : | Normal | Region start : | 5 |
| Mercury height (mm) : | 105.765 | * | * | Region stop : | 50000 |
| Vol. at run (mm3) : | 479 | * | * | Region number of intervals : | 40 |
| Weight dilatometer+mercury+sample (g) : | 235.168 | * | * | Pores average mode : | 50 % |
| Weight dil.+mercury+sample correct(g) : | 234.951 | * | * | Particle size distribution : | No |
| Capillary mercury height (mm) : | 67.765 | * | * | Kp factor : | 0 |
| *** | *** | *** | *** | Cut off radius : | 0-0 |

RESULTS

| | |
|---|---|
| Total cumulative volume (mm3/g) : | 542.9936 |
| Total specific surface area (m²/g) : | 9.807 |
| Average pore diameter (Nanometer) : | 231.889557 |
| Total porosity (%) : | 70.9738 |
| Bulk density (g/cm3) : | 1.30708 |
| Apparent density (g/cm3) : | 4.50313 |
| Sample volume correction : | .9832295 |

PORE SIZE DISTRIBUTION (custom ranges)

| Pore diameter ranges (nM) | Specific Volume (mm3/g) | Relative Volume (mm3/g) | Relative Volume (%) | Specific Surface (m²/g) | Relative Surface (m²/g) |
|---|---|---|---|---|---|
| 376.7-345.9 | 0.00 | 0.00 | 0 | 0 | 0 |
| 345.9-317.6 | 0.00 | 0.00 | 0 | 0 | 0 |
| 317.6-291.6 | 0.32 | 0.32 | .06 | .0042 | .0042 |
| 291.6-267.8 | 1.59 | 1.27 | .23 | .0226 | .0184 |
| 267.8-245.9 | 72.93 | 71.34 | 13.15 | 1.1516 | 1.129 |
| 245.9-225.7 | 329.62 | 256.69 | 47.33 | 5.4922 | 4.3405 |
| 225.7-207.3 | 444.59 | 114.97 | 21.2 | 7.5988 | 2.1067 |
| 207.3-190.3 | 491.40 | 46.82 | 8.63 | 8.5351 | .9362 |
| 190.3-174.7 | 515.92 | 24.52 | 4.52 | 9.068 | .5329 |
| 174.7-160.5 | 527.07 | 11.15 | 2.06 | 9.3317 | .2637 |
| 160.5-147.3 | 534.08 | 7.01 | 1.29 | 9.5128 | .181 |
| 147.3-135.3 | 537.58 | 3.50 | .65 | 9.6117 | .0989 |
| 135.3-124.2 | 539.49 | 1.91 | .35 | 9.6711 | .0594 |
| 124.2-114 | 540.45 | 0.96 | .18 | 9.7032 | .032 |
| 114-104.7 | 541.08 | 0.64 | .12 | 9.7269 | .0237 |
| 104.7-96.2 | 541.40 | 0.32 | .06 | 9.7394 | .0126 |
| 96.2-88.3 | 542.04 | 0.64 | .12 | 9.767 | .0276 |
| 88.3-81.1 | 542.04 | 0.00 | 0 | 9.767 | 0 |
| 81.1-74.4 | 542.04 | 0.00 | 0 | 9.767 | 0 |
| 74.4-68.3 | 542.36 | 0.32 | .06 | 9.7845 | .0175 |

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