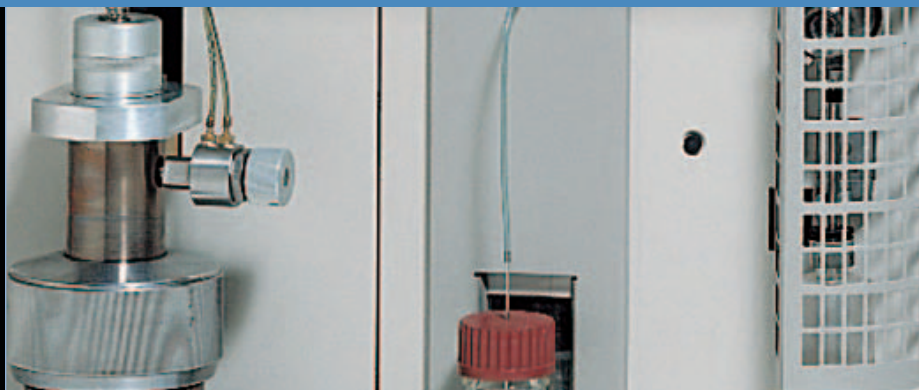


Pascal Series Mercury Intrusion Porosimeters



Maximum resolution in minimum time!



Reservoir rocks



Ceramic materials



Building materials



Polymers and resins



Pharmaceuticals



Restoration and conservation



Geology



Mining

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 Electron Corporation 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.

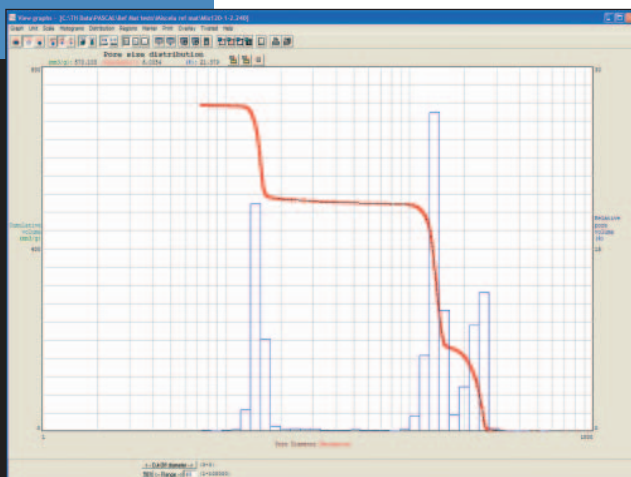
Pascal does it faster than ever!

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.



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.

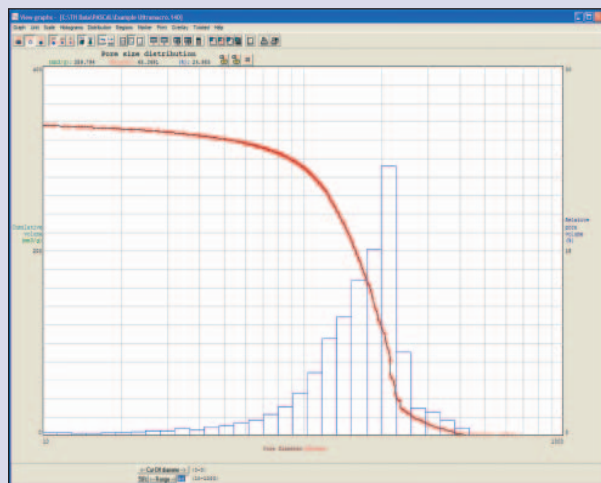


Dilatometer in vertical position

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 (µm)	Specific Volume (cm³/g)	Relative Volume (cm³/g)	Relative Volume (%)	Specific Surface (m²/g)	Relative Surface (m²/g)
435.6508-435.036	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
335.254-294.286	12.73	6.46	1.93	.0001	.0001
294.286-258.328	20.27	7.54	2.25	.0003	.0001
258.328-226.763	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	7.68	.0059	.0008
118.1868-103.7454	285.69	17.29	5.16	.0065	.0006
103.7454-91.0694	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
12.8932-11.3179	334.63	0.81	.24	.0105	.0003
11.3179-9.9348	335.35	0.72	.22	.0107	.0003

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.

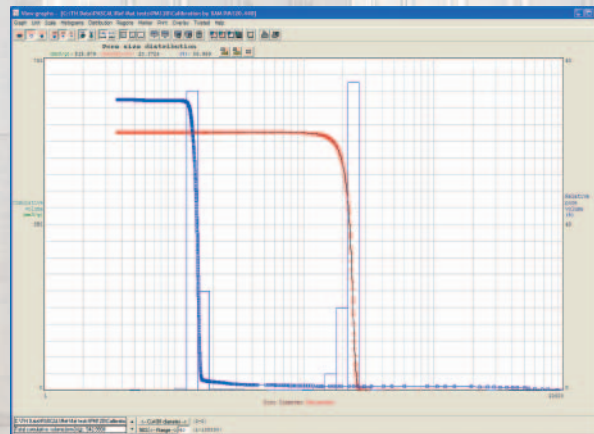
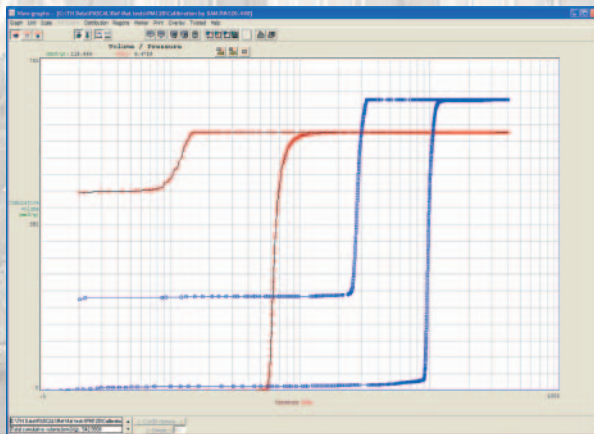


Pascal 240 accepts all type dilatometers

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.



Pascal 140/240/440 Ver. 1.04B1 - [C:\VTH Data\PASCAL\Ref_Mat tests\Nist-Bam1 - ...]

File View Report Graphs Instruments serial port Edit data points Help

Pascal 440

Sample information | Acquisition parameters | Calculation parameters | Blank parameters (7) | Report selection

Company Name: Thermo
Operator: P R Date: 16-10-2001

Sample name: 1 NIST_BAM Sample mass (g): 332
Preparation: vuoto fino 10 Pascal Sample dens. (g/cm3): 1

Vol. at run (mm3): 461 Mercury height (mm): 104.218
Volume (mm3) at: Capillary mercury height (mm): 95.218
pressure (kPa): 100 Weight dilat.+Hg+Snip (g): 226.435 --> Correct (g) --> 226.584

Number of points increase: 844 Number of points decrease: 772 Number of points macropores: 0

Report Options

Particle size distribution | Data Report | Custom | Graphs | Preview | Zoom + | Next | Back

Tab | Parameters | Results | Pore size distribution

Logo Load...

Instrument type ...
 Sample name
 Filename
 Software version
 Date
 Comment

Sheet Font... Print Exit Help

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Many ways to look at the data

Calculations

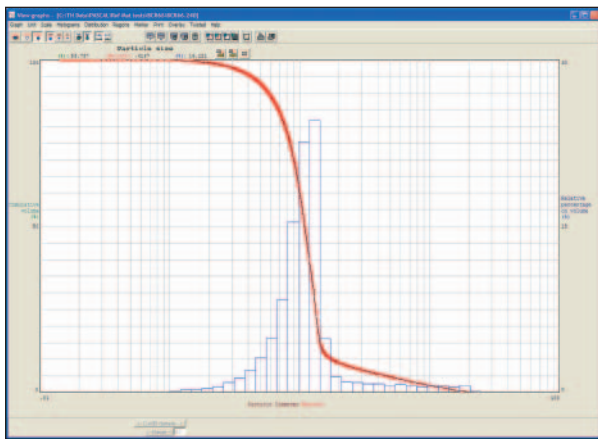
Bulk and apparent density, total and incremental pore volume, porosity, incremental specific surface area (four models), average pore size (two models), pore size distribution (derivative and histograms), particle size distribution (Mayer-Stowe model), pore and particle surface area distinction.

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 information		Acquisition parameters		Calculation parameters	
Company Name :	Thermo	Max pressure :	400	Contact 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	.85	9.6117	.0889
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	0.84	.12	9.7269	.0237
104.7-96.2	541.40	0.32	.06	9.7394	.0126
96.2-88.3	542.04	0.84	.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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