Porosimeters

"Not Just Products ... Solutions"!

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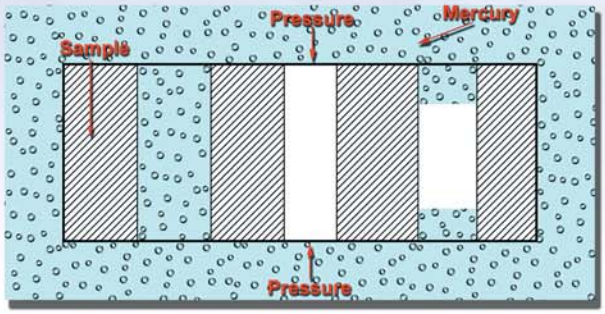
Principle

A nonwetting liquid like mercury does not spontaneously fill pores of a sample because the sample/nonwetting liquid surface free energy is greater than the sample/gas surface free energy. However, application of pressure can force a nonwetting liquid into the pores of a sample. The differential pressure required to force the nonwetting liquid into a pore is given by:

$$P = - 4 \gamma \cos \theta / D$$

P = differential pressure
γ = surface tension of nonwetting liquid
θ = contact angle of the nonwetting liquid with the sample
D = pore diameter

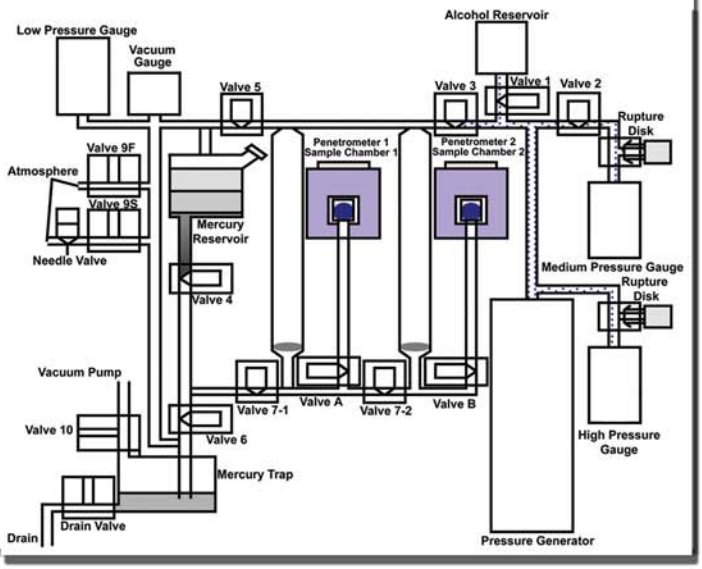


The pore diameter at any location in a pore is defined such that (dS/dV) = (4/D), where (dS/dV) is the ratio of small increase in surface area due to a small increase in volume of nonwetting liquid in the pore.In this technique the pressure and the volume of intruded nonwetting liquid are accurately measured. Combining these data with the surface tension and the contact angle of the liquid, pore diameter, pore volume, pore volume distribution and pore surface area are computed.

Operating Procedure

The PMI Porosimeter consists of a low pressure section, a high pressure section, and penetrometers connected to the sample chambers. The low pressure section includes the mercury reservoir and the mercury trap, and can be opened to the atmosphere or evacuated. The high pressure section includes a pressure generator that uses isopropyl alcohol as the hydraulic fluid to pressurize mercury. The sample is placed inside a stainless steel cell that has a hole on one of its sides for evacuation and entry of mercury into the cell. The cell is closed with a lid and placed in the sample chamber. Intrusion volume of mercury is measured by noting changes in mercury level in the penetrometer with a magnetic sensor. Exposure to mercury is negligible due to the unique design of the instrument. The unique design also permits use of nonmercury nonwetting liquids for intrusion.

Another instrument design uses water as the nonwetting liquid and uses absolutely no mercury. The instrument is known as the Aquapore.



Unique Features

Hardware

- Mercury and Nonmercury (other nonwetting liquid) Porosimetry can be performed in the Porosimeter.
- A unique system design allows use of the sample chamber to be connected to or isolated from the low pressure and high pressure sections of the instrument. This allows fully automated testing with no requirement to transfer samples from a low pressure station to a high pressure station in the middle of the test.
- Presence of three pressure transducers (low, medium, high) ensures maximum resolution and accuracy.
- Equipment design allows extrusion to be performed even at subatmospheric pressures.
- Operator involvement is minimum.
- Automatic clean-out routines after test limit the amount of mercury exposure.
- Samples can be tested at elevated or sub-ambient temperatures.
- Sample chamber and penetrometer are made out of stainless steel.
- Novel design enables sample chamber and penetrometer tube to be changed individually and independently.
- Simultaneous testing of multiple samples in a multiple sample chamber instrument is possible.
- In-situ pretreatment of samples is preformed to avoid contamination.

Safety

- A mercury level sensor warns the user to empty the mercury trap (where used mercury is collected).
- Doors of sample chamber area and the mercury drain reservoir area minimize operator exposure to mercury.
- The instrument cabinet has its own internal ventilation system. Mercury vapors within the cabinet are captured by an activated carbon filter.
- Burst pressure disks and relief valves prevent over pressurization of the system.
- The internal computer monitors the position of all of the valves and does not allow valve combinations that could impede the operation of the instrument. (The low and high pressure sections can not both be connected to the sample chamber, the mercury fill and drain valves can not be open at the same time, etc....)
- The hydraulic pressure generator system (in medium and high pressure instruments) has built-in limit switches that prevent mechanical damage that could be caused by over travel of the piston.
- Both the user software and the internal computer system monitor the pressure and do not allow the pressure generator to operate beyond the maximum pressure specifications of the instrument. In case of an over pressure situation, the internal computer will automatically retract the piston to relieve the pressure in a controlled and safe manner.

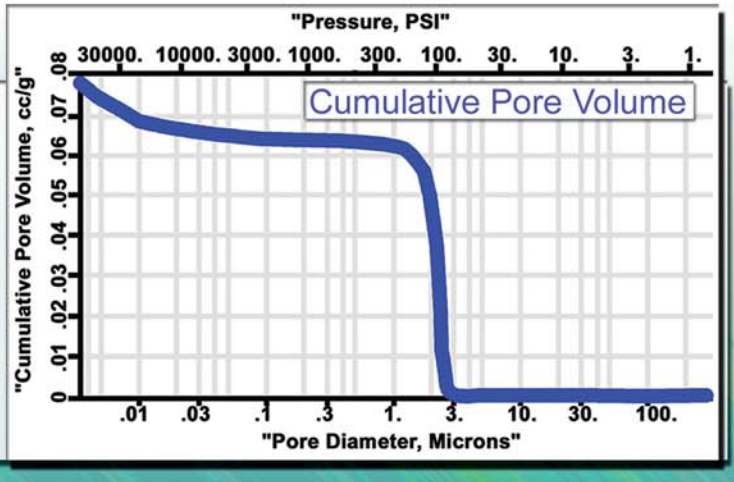
Software

- Windows 98/NT/2000/XP compatible software enables convenient use of the instrument.
- User defined contact angle and surface tension can be entered.
- Data can be collected either at user-specified pressure values, at user-specified intrusion volume values, or at evenly spaced user-specified number of data points. Equilibrium conditions are also user adjustable.
- Multiple users and/or sample types can be given their own software “group”. The software settings are remembered independently for each “group”.
- Outgassing and pretreatment performed either automatically or manually. The user can skip the normal automatic outgassing procedure.
- Testing can be paused at any time. While a test is paused, the software allows full manual operation of the instrument while retaining the ability to continue the test when manual control is completed.
- Separate software for report generation enables the user to plot up to seven test results on the same graph. Report generation does not have to be performed on the same computer that controls the instrument.
- The report software allows data to be converted directly into an excel spreadsheet or exported to standard text or tab-delimited database or other programs.
- The software features curve fitting and interpolation routines that help in better analysis of the data.
- Desired pressure units can be selected for the reports independent of the units in test.
- Report software enables user to view reports, modify scales in graphs and print reports.

Applications

Mercury Porosimetry

PMI Porosimeters are capable of measuring properties such as pore volume, surface area, pore volume distribution, percent porosity and density. Pore volume is determined by the total volume of mercury that intrudes into the pores. Measured pore volume is the volume of all pores accessible to the mercury. The percent porosity can also be obtained as a function of pressure (pore size).



Pore volume distribution is presented in terms of a distribution function, Fv.

$$Fv = -\frac{dV}{d \log D}$$

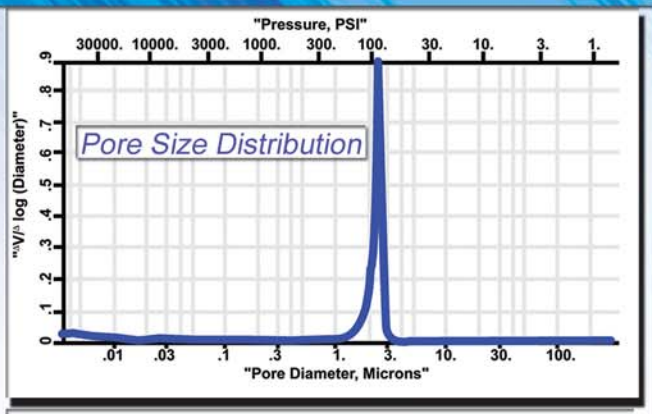
V = cumulative pore volume

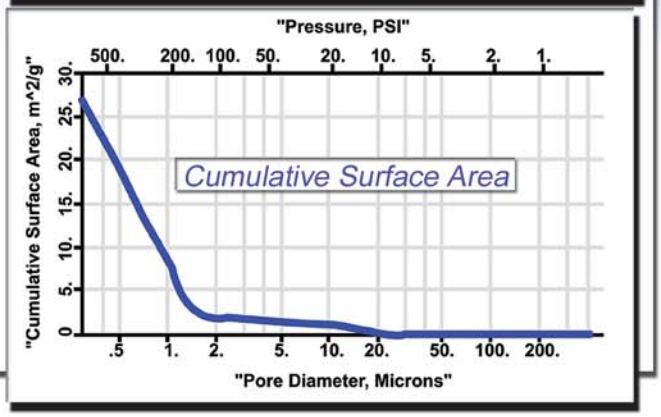
The distribution function is such that area under the function in any pore diameter range yields pore volume of pores in that range.

Measurement of surface area, through porosimetry gives a quick estimate of the surface area. Surface area is computed from volume measured as a function of pressure through the use of the following relation derived from the equations given earlier.

$$S = \int PdV/(-\gamma \cos \theta)$$

S = surface area

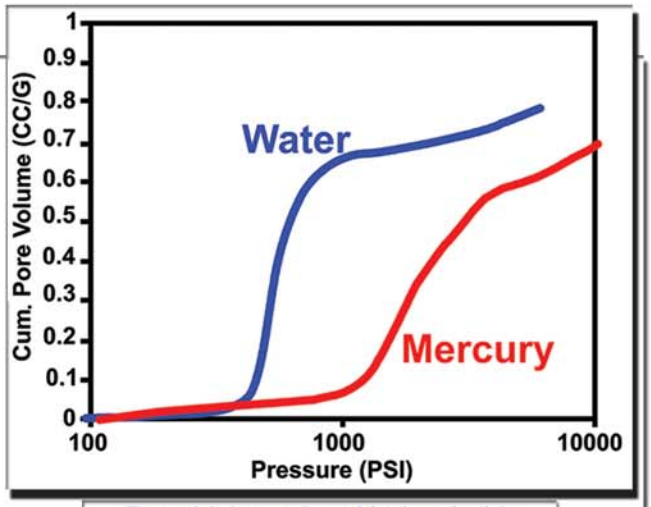




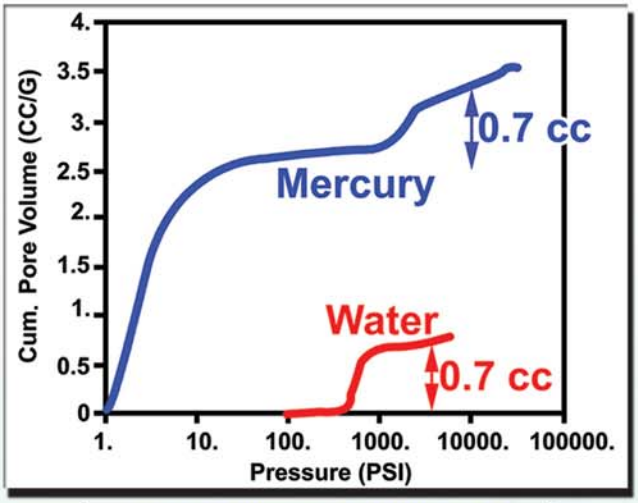
Nonmercury Porosimetry

PMI Porosimeters are unique in providing the dual capability to perform nonmercury porosimetry as well as conventional mercury porosimetry in the same instrument. The nonmercury porosimetry uses a fluid that is nonwetting to the material being tested. The use of nonmercury porosimetry over mercury porosimetry for certain materials may be advantageous due to the following reasons:

- Lower intrusion pressures: Most liquids have much lower surface tension values than mercury. The intrusion into pores can be accomplished at much lower pressures than those required with mercury. This greatly reduces the risk of the sample crushing at high pressures.



Pore Volume in a Hydrophobic Material as a Function of Pressure



Pore Volume of a Mixture of Hydrophobic & Hydrophilic materials as a Function of Pressure

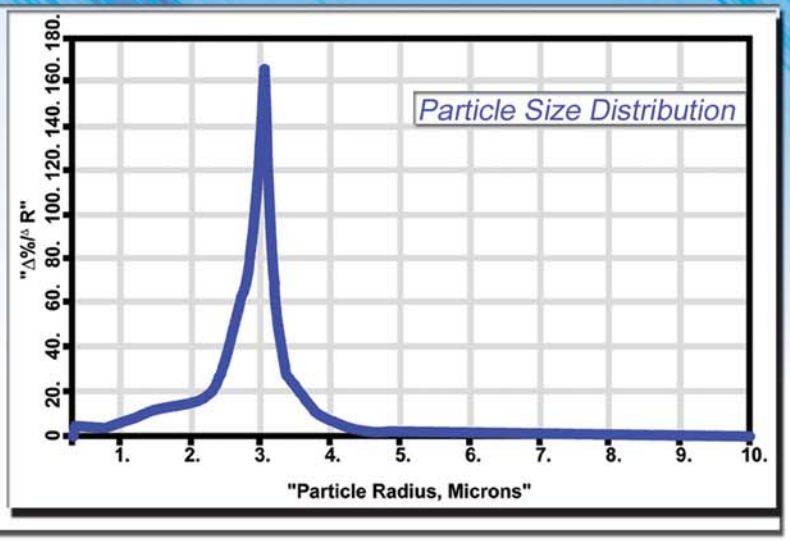
- Use of application liquid: Many applications require that the porous material be in constant contact with certain fluid. If this fluid is nonwetting to the material, it can be used for testing.
- Lower pore sizes: The pore sizes that can be measured in nonmercury porosimetry are much lower than those measured by mercury porosimetry.

For hydrophobic materials the use of water as the intruding fluid allows the measurement of very small pores that are beyond the scope of measurement by mercury intrusion method. When testing a sample that is a mixture of hydrophobic and hydrophilic materials, the combination of mercury intrusion and water porosimetry can give a comprehensive picture of the characteristics of both the hydrophobic and hydrophilic pores of the sample. The PMI **Aquapore** has the ability to perform water intrusion test without using any mercury.

Testing of Powders

Porosimetry of powders often results in bimodal distributions. Mercury enters the large interparticle voids at low pressures and intraparticle pores at high pressures.

The PMI Porosimeter uses a sample cell specifically designed for powders. This prevents the powder from contaminating other parts of the system. A slow evacuation routine prevents the powder from being pulled out of the sample cell. The particle size characterization is an important aspect in the manufacture of many powders including pharmaceutical powders.



Specifications

PMI's Automated Porosimeters are capable of measuring pore volume, surface area, pore size distribution, particle size distribution and percent porosity of porous materials based on intrusion and extrusion (below atmospheric pressure) of mercury or another nonwetting fluid.

Pore diameter range: 0.0035 – 400 microns (mercury)
Smaller pores with other liquids

Sample volume: Up to 10 cc for sample cells (others available)

Pressure transducer: Three separate transducers, low, medium and high, for each instrument (some low pressure instruments have only two transducers)

Test pressure: 0 – 60,000 psi (instruments available with different pressure ranges)

Vacuum gauge: 0 – 1000 microns (0 – 0.1 cm) Hg

Penetrometer: Up to 1.5 cc (nominal) with unlimited refill capability

Resolution: 0.001 cc

Accuracy: 0.005 cc

Pressurizing fluid: Air/ Isopropyl Alcohol/Water

Power requirements: 110/220 Vac, 50/60 Hz / 15 Amps.

Size: 72" H, 30" W, 30"D, (approximately)

Weight: 400 lbs (approximately)

Software: For Windows 98//NT/XP

Data Report

- Pore Volume versus diameter
- Delta volume histogram
- Pore distribution (histogram)
- Percent total pore volume
- Pore distribution (pressure and diameter)
- Particle size distribution (histogram)
- Percent porosity and density
- Cumulative surface area
- Porosimetry text data
- Summary sheet (reporting median pore sizes based on volume and surface area along with standard deviations)

Mercury Porosimetry Analysis
for
PMI Standard

on
07-11-2004
by
POROUS MATERIALS, INC. ANALYTICAL SERVICES DIVISION
20 DUTCH MILL ROAD
ITHACA, NY 14850 USA
PHONE (607)-257-5544 or 1-800-TALK-PMI

File: \\Fileserver1\porlab\Test-server\test_ser\
Sample ID: Glass Bubbles .153
Operator: S Cotton
Lot Number:
PMI Test Number: T101
Purchase Order Number:

Sample Weight = 0.2000 g
Liquid = MERCURY
Mercury Contact Angle = 140 Degrees
Mercury Surface Tension = 480 Dynes/cm

Cumulative pore volume in cc/g of sample
% total pore volume = % of total cumulative pore volume belonging to pores of diameter > D
Average pressure = square root of P(I)*P(I-1)
Pore size distribution function is equal to dV/dlog P
Surface area assumes cylindrical pores

Pressure PSIA	Pore Diameter Microns	Cumulative Pore Vol. cc/g	% of total Pore Vol.	Average Pressure PSIA	dV ----- dlogP	Cumulative Surf. area m2/g
107.495	1.9837	0.0	0.0	107.495	0.176	0.0
122.97	1.734	0.0103	0.433	114.972	0.128	0.0221
141.151	1.5107	0.0179	0.756	131.747	0.136	0.041

PMI Analytical Services

PMI has over two decades of experience in analyzing porous materials using mercury/nonmercury intrusion porosimetry and other techniques. The analytical services division of PMI is well known for providing timely, accurate and reliable contract testing services. Contact PMI for details.

Models

Model: AMP–200–A–1

This is a fully automated unit capable of pressures up to 200 psi. It uses mercury as the penetration liquid and is able to detect pores >1 micron. It has one sample chamber. This is an economical unit good for regular testing of small volume of samples with large pores. Samples that are sensitive to pressure can be tested in this porosimeter. The unit is robust and does not require much maintenance.

Model: AMP–200–A–1–NM

This model incorporates special design features to permit use of nonmercury nonwetting liquid for intrusion. Otherwise it is identical with Model: AMP–200–A–1. Measurement of smaller pores (>0.04 microns with water) become possible. All advantages of using a nonmercury intrusion liquid are available to the user.

Model: AMP–200–A–2

This model has two sample chambers. In all other respects this model and the Model: AMP–200–A–1 are identical. Two samples can be tested simultaneously. This feature provides the capability of testing a sample of a material and a standard under identical conditions, so that the material may be compared with the standard and decisions on the selection or rejection of the material, quality control concerns and process control may be taken intelligently.

Model: AMP–200–A–2–NM

The porosimeter has all the features of Model: AMP–200–A–1 and the optional feature to test two samples in its two sample chambers with any nonmercury nonwetting liquid. Small pores are easily detectable (>0.04 microns with water). The porosimeter has all the advantages of a two sample chamber unit (see Model: AMP–200–A–2) and those associated with the use of nonmercury

Model: AMP–200–A–3

This model is identical with Model: AMP–200–A–1 except that it has three sample chambers permitting three samples to be tested simultaneously. This is an inexpensive model to test a large volume of samples with larger pores (>1 microns).

Model: AMP–200–A–3–NM

This Model has three sample chambers and is designed to work with any nonmercury nonwetting liquid. It has all the features of Model: AMP–200–A–1. This instrument has the advantages of using nonmercury nonwetting liquid, permits detection of smaller pores (<0.04 microns with water) and can test a large volume of samples.

Model: AMP–2K–A–1

This is a fully automated low pressure model with maximum pressure capability of 2000 psi. Mercury is used as the intrusion liquid and the detectable pore size is 0.1 micron. The unit has one sample chamber and is good for testing a small volume of samples with pores >0.1 micron. Because of low pressures, the effect of pressure on the structure of the sample is not expected to be appreciable. This is also a robust model that requires very little maintenance.

Model: AMP–2K–A–1–NM

This model is identical with the Model: AMP–2K–A–1 except that it has features that permit any nonmercury nonwetting liquid to be used. Pores less than 0.015 micron in size are measurable. It gives all the advantages associated with its ability to use any liquid.

Model: AMP–2K–A–2

This model has two sample chambers. All other features of the model are identical with those of Model: AMP–2K–A–1. The instrument is capable of testing two samples simultaneously and has all the advantages associated with the use of two sample chambers (see Model: AMP–200–A–2).

Model: AMP–2K–A–2–NM

This low pressure model has been designed to use nonmercury liquid for intrusion into samples in two sample chambers and has the features of Model: AMP–2K–A–1. The porosimeter has all the advantages associated with use of any nonmercury intrusion liquid and two sample chambers (See Model: AMP–200–A–2).

Model: AMP–2K–A–3

This low pressure porosimeter has three sample chambers other features of the porosimeter are identical with those of Model: AMP–2K–A–1. Three samples can be tested simultaneously. The porosimeter is very useful for testing large volume of samples with large pores (>0.1 micron) on a regular basis.

Model: AMP–2K–A–3–NM

This model is identical with Model: AMP–2K–A–1, except that it has three sample chambers and it is capable of using any nonmercury nonwetting liquid. The instrument has all the advantages of using any nonmercury intrusion liquid and can test a large volume of samples with pores less than 0.1 microns in size.

Model: AMP–10K–A–1

This is a completely automated medium pressure model capable of going up to 10,000 psi. It has one sample chamber. It uses mercury as the intrusion liquid and measures pores with diameters down to 0.02 microns. The effect of pressure on pore structure is normally not appreciable. The instrument is suitable for testing small volume of samples with not very small pore diameters.

Model: AMP–10K–A–1–NM

This Model can use any nonmercury intrusion liquid and has all the features of Model: AMP–10K–A–1. The pore size detectable can be less than 0.02 microns. It has all the advantages of using any nonmercury liquid.

Model: AMP–10K–A–2

This Model has two sample chambers. Other features of this model are the same as those of Model: AMP–10K–A–1. Simultaneous testing capability for two samples gives the instrument all the advantages of a two–sample chamber instrument (See Model: AMP–200–A–2).

Model: AMP–10K–A–2–NM

This unit has the ability to test with any nonmercury intrusion liquid and has two sample chambers. In all other respects it is identical with Model: AMP–10K–A–1. The pore size detectable can be less than 0.02 microns. The porosimeter has all the advantages of a machine, that has two sample chambers (See Model: AMP–200–2) and has the ability to use any nonmercury nonwetting liquid.

Model: AMP–10K–A–3

This model contains three sample chambers and has all the features of Model: AMP–10K–A–1. Simultaneous testing of three samples is possible. The instrument is good for testing a large volume of samples containing not too small pores (> 0.02 microns).

Model: AMP–10K–A–3–NM

This medium pressure model is identical with Model: AMP–10K–A–1 and has additional features that permit use of any nonmercury intrusion liquid and simultaneous testing of three samples. Pores less than 0.02 microns may be detected. It has the advantages due to the use of any nomercury liquid. It is ideal for testing a large volume of samples with smaller pore sizes (0.02 microns).

Model: AMP–15K–A–1

This is another completely automated medium pressure unit in which the intrusion pressure can go up to 15,000 psi. Using mercury as the intrusion liquid, pores down to 0.015 microns can be measured. The Instrument has one sample chamber. The effect of pressure on pore structure is usually small. The instrument is suitable for testing a small volume of samples.

Model: AMP–15K–A–1–NM

This instrument can use any nonmercury intrusion liquid. In all other respects it is identical with Model: AMP–15K–A–1. Measurable pore size can be less than 0.015 microns, depending on the fluid. The instrument has all the advantages associated with the use of a nonmercury liquid.

Model: AMP–15K–A–2

This is a two-sample chamber model with all the capabilities of Model: AMP–15K–A–1. Two samples can be tested simultaneously. This capability gives the instrument all the advantages of a two sample chamber instrument. (See Model: AMP–200–A–2).

Model: AMP–15K–A–2–NM

This two–sample chamber model is capable of using any nonmercury intrusion liquid. Other features of the instrument are identical with the Model: AMP–15K–A–1. The porosimeter has the advantages of a two sample chamber machine that is capable of using any nonmercury liquid.

Model: AMP–15K–A–3

This is a three–sample chamber model that has all the features of Model: AMP–15K–A–1. Three samples can be tested simultaneously. The instrument can handle a large volume of samples with pores greater than 0.015 microns.

Model: AMP–15K–A–3–NM

This porosimeter is identical with Model: AMP-15K-A-1 except that it has three sample chambers and it can use any nonmercury intrusion liquid. The instrument has all the advantages due to the use of nonmercury liquid. It is ideal for testing a large volume of samples with pores which can be less than 0.015 microns.

Model: AMP–30K–A–1

This is a sophisticated and fully automated high pressure model. It is capable of attaining pressures up to 30,000 psi. The smallest pore size measurable is about 0.007 microns with mercury as the intrusion liquid. This is a single sample chamber unit, which is cost effective when a small number of samples need to be tested or when testing is infrequent. The high pressure attainable by this instrument can cause crushing of porous particles of powders so that the total surface area including surface area of internal pores in the particles could be measured.

Model: AMP–30K–A–1–NM

This model is identical with Model: AMP–30K–A–1 in addition to having the ability to use any nonmercury intrusion liquid. Pore sizes less than 0.007 microns can be measured, depending on the fluid used. The instrument has all the advantages associated with the use of nonmercury intrusion liquid.

Model: AMP–30K–A–2

This high pressure model has two sample chambers and all of the capabilities of Model: AMP–30K–A–1. Simultaneous testing of two samples is possible. The instrument has all the advantages of a two-sample chamber instrument (See Model: AMP–200–A–2).

Model: AMP–30K–A–2–NM

The model has the ability to use any nonmercury intrusion liquid and two samples in its two sample chambers. In all other respects it is identical with Model: AMP–30K–A–1. It has all the advantages due to the use of nonmercury liquid and simultaneous testing of two samples (See Model: AMP–200–A–2).

Model: AMP–30K–A–3

This sophisticated model is capable of testing three samples simultaneously in its three sample chambers. All other features of this model are the same as those of Model: AMP–30K–A–1. The porosimeter is suitable for testing a large volume of samples with pores > 0.007 microns.

Model: AMP–30K–A–3–NM

This is a model whose features are the same as those of Model: AMP–30K–A–1, but it has three sample chambers and is capable of using any nonmercury intrusion liquid. It has all the advantages of using nonmercury intrusion liquid. The instrument is suitable for testing a large volume of samples and for detecting < 0.007 micron pores.

Model: AMP–60K–A–1

This unit is sophisticated and fully automated. It is capable of attaining 60,000 psi intrusion pressure. Using mercury as the intrusion liquid, the minimum pore size detectable is 0.0035 microns. It has one sample chamber. It is the most economical unit for testing a small volume of samples with small pores. The high pressure can crush most powders with internal pores. Therefore, it is possible to determine areas of external surfaces of powders and surfaces of pores in powders.

Model: AMP–60K–A–1–NM

This model has all the features of Model: AMP–60K–A–1 and the ability to use nonmercury intrusion liquid. The instrument has all the advantages associated with the use of nonmercury intrusion liquid.

Model: AMP–60K–A–2

This porosimeter has two sample chambers. Otherwise it is identical with Model: AMP–60K–A–1. Two samples can be simultaneously tested. This unit has all the advantages of two-sample chamber model (See Model: AMP–200–A–2).

Model: AMP–60K–A–2–NM

This model is identical with Model: AMP–60K–A–1 and has the ability to test samples using mercury or any other nonmercury nonwetting intrusion liquid. The instrument can measure small pores and has all the advantages due to the use of nonmercury nonwetting liquid and two sample chambers (See Model: AMP–200–A–2).

Model: AMP–60K–A–3

This porosimeter model has all the features of Model: AMP–60K–A–1 and three sample chambers so that three samples can be tested simultaneously at high pressures. This instrument is ideal for testing of large volume of samples with small pores.

Model: AMP–60K–A–3–NM

This is a high pressure three–sample chamber porosimeter that has been designed to use any nonmercury intrusion liquid. All other features of the model are identical with those of Model: AMP–60K–A–1. The porosimeter is suitable for testing a large volume of samples with small pores and has all the special advantages due to the use of nonmercury intrusion liquid.