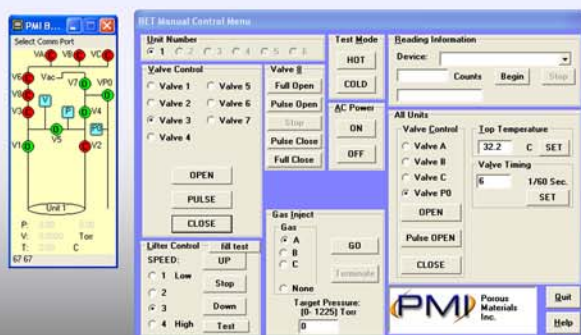


# SORPTOMETERS



*"Not Just Products... Solutions"!*



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## Principle

When a clean surface is exposed to a gas, an adsorbed film forms on the surface. Adsorbed films also form on the surface of pores within a material and vapor can condense in the pores. At a constant temperature, the amount of adsorbed/condensed gas on a surface depends on the pressure of the gas. Measurement of the amount of adsorption/condensation as a function of pressure can give information on the pore structure. The PMI Sorptometers use gas adsorption/condensation to analyze pore characteristics.

### Physical Adsorption

Weak van der Waal's type interaction of molecules with a pore surface leads to physical adsorption. The Brunauer, Emmett and Teller (BET) theory of physical adsorption is normally used for analysis of adsorption data to compute surface area.

$$\frac{P}{W(P_o - P)} = \frac{1}{CW_m} + \frac{C-1}{CW_m} \times \frac{P}{P_o}$$

where:

W = amount of adsorbed gas

$W_m$  = amount of gas adsorbed in a monolayer

P = gas pressure

$P_o$  = equilibrium (saturation) vapor pressure at the test temperature

C = dimensionless constant that depends on the temperature and the gas/solid system

When vapor pressure, P, is low compared with  $P_o$  ( $0.05 < P/P_o < 0.3$ ), the plot of  $[P/W (P_o - P)]$  versus  $[P/P_o]$  is linear and the plot yields the magnitudes of C and  $W_m$ . The surface area S per unit mass, m, of the sample is computed using the cross-sectional area of the adsorbed gas molecule:

$$S = \frac{W_m N_o \alpha}{m}$$

where:

$N_o$  = Avogadro's number

$\alpha$  = cross-sectional area of the adsorbed gas molecule

$W_m$  = amount of gas absorbed in moles.

### Vapor Condensation

As the relative vapor pressure ( $P/P_o$ ) increases, vapor eventually condenses in the pores utilizing the surface free energy available due to replacement of the solid/vapor interface by solid/liquid interface. The amount of vapor condensed in pores gives the pore volume, and the Kelvin equation gives the pore diameter.

$$\ln\left(\frac{P}{P_o}\right) = -\frac{4 \gamma \underline{V} \cos \theta}{D R T}$$

where:

$\gamma$  = surface tension of condensed liquid

$\underline{V}$  = molar volume of condensed liquid

$\theta$  = contact angle

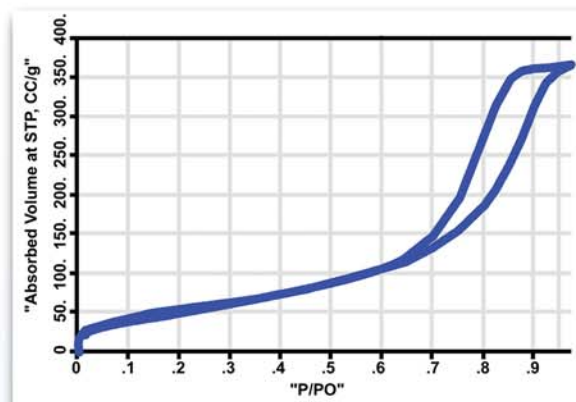
D = pore diameter

R = gas constant

T = absolute test temperature

Adsorbed layers of molecules form on the pore walls before condensation fills the pores. Therefore, the actual pore diameters are computed by adding two times the thickness of the adsorbed gas layer to D.

A complete adsorption isotherm is determined by measuring the amount of vapor adsorbed as a function of increasing pressure. A desorption isotherm is determined by measuring the amount of adsorption as a function of decreasing pressure. Based on this technique, characteristics of materials related to adsorption, desorption, surface area and pore volume can be determined.



Adsorption and Desorption Isotherms at Liquid  $N_2$  temperature

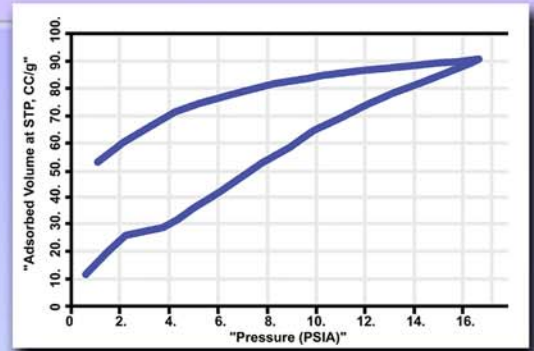
## Chemisorption

Chemical reaction between a gas and a surface accompanied by a high heat of adsorption results in chemisorption. Chemisorption data can be analyzed using various theories. Langmuir equation yields:

$$\frac{W}{W_m} = \frac{KP}{1 + KP}$$

where:

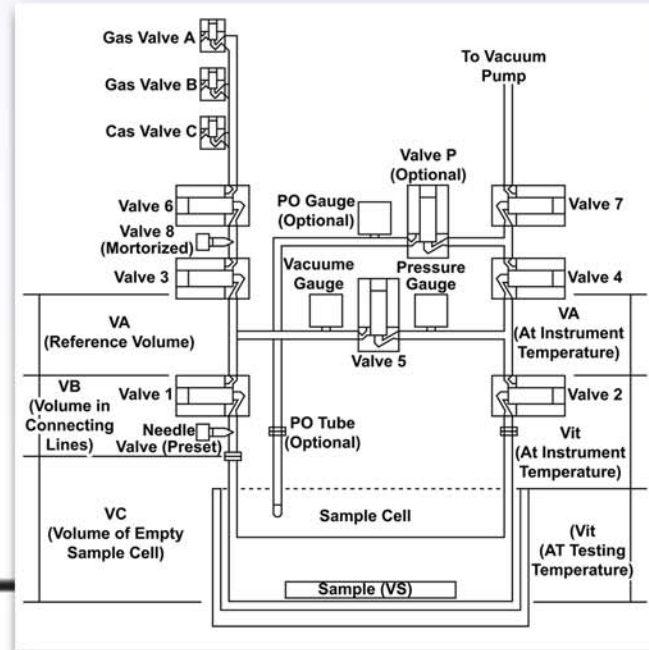
W = amount of adsorbed gas  
 $W_m$  = amount of gas adsorbed in a monolayer  
 K = equilibrium constant  
 P = gas pressure



Ammonia Adsorption and Desorption Isotherms at 0°C

## Equipment

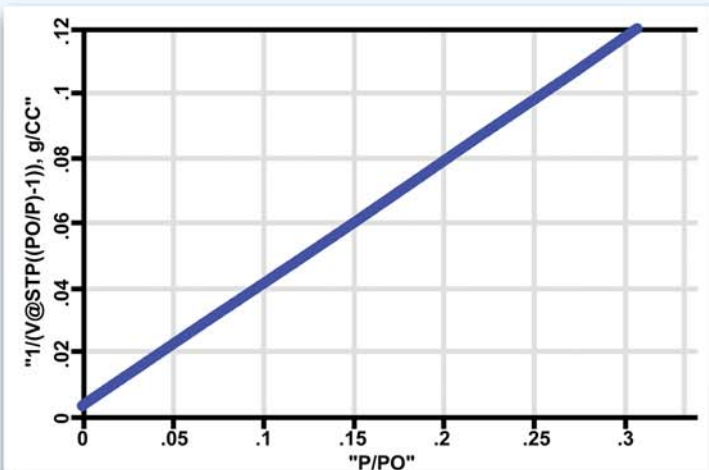
The PMI Sorptometer uses volumetric method to compute the amount of gas. The sample chamber is connected through valves to the reference volume, gas supply, & vacuum line. For a test, a weighed sample is placed in the sample chamber. The sample chamber of known volume is heated and evacuated to remove moisture and adsorbed gases. The desired adsorption temperature is then established in the sample chamber using a constant temperature bath, and the chamber is isolated. The reference volume is pressurized with adsorbate gas, and isolated. The pressure of reference volume is measured. The gas is allowed to expand into the sample chamber. After equilibration the gas pressure is measured. The amount of gas adsorbed by the material is calculated by making use of the gas law.



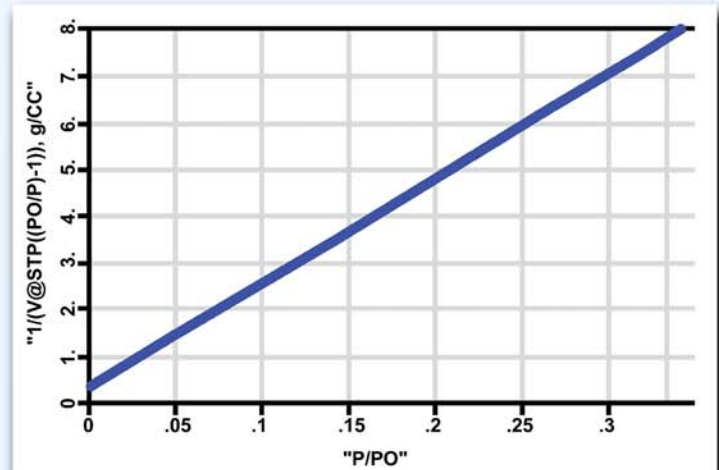
## Applications

### Surface Area:

PMI BET Sorptometers are capable of testing samples with moderate to high surface areas by using nitrogen gas. For samples with very low surface area, a larger amount of sample or krypton gas can be used for greater accuracy. The number and spacing of data points in multipoint surface area measurement are user adjustable.



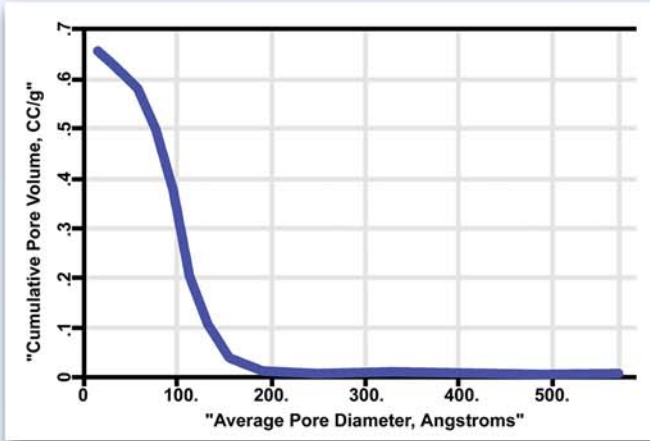
Surface Area Analysis — Nitrogen



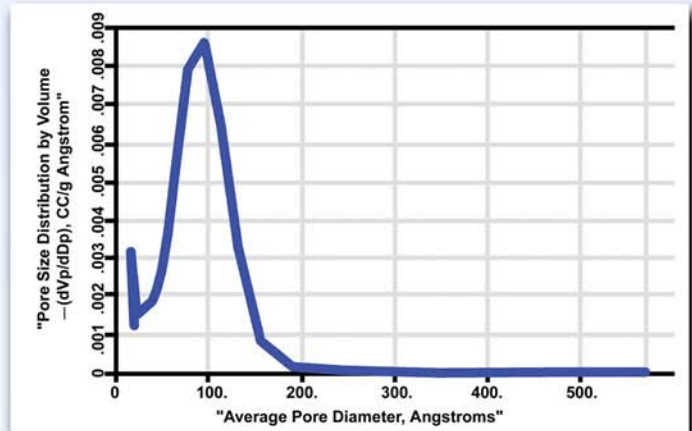
Surface Area Analysis — Krypton

## Pore Volume and Pore Diameter

Pore volume, pore diameter and pore volume distribution can be determined accurately by the PMI Sorptometer. The distribution function is such that area under the function in any pore diameter range is the volume of pore in that range.



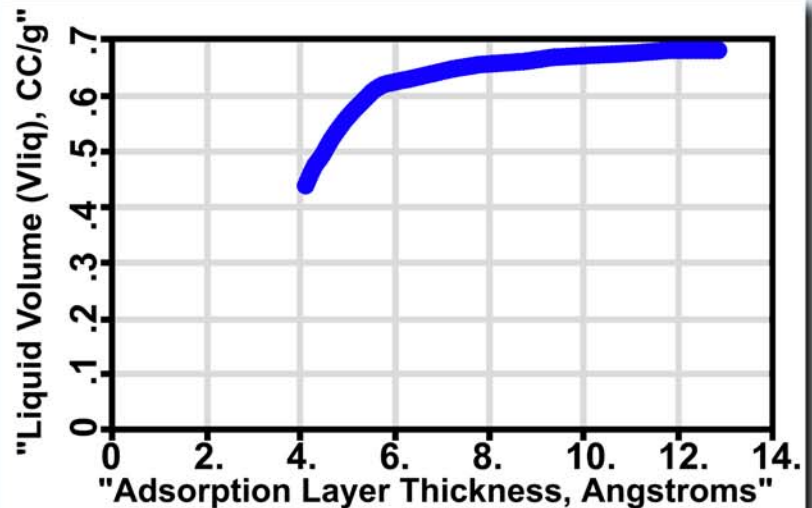
Cumulative Pore Volume



Pore Volume Distribution

## Adsorption and Desorption Isotherms:

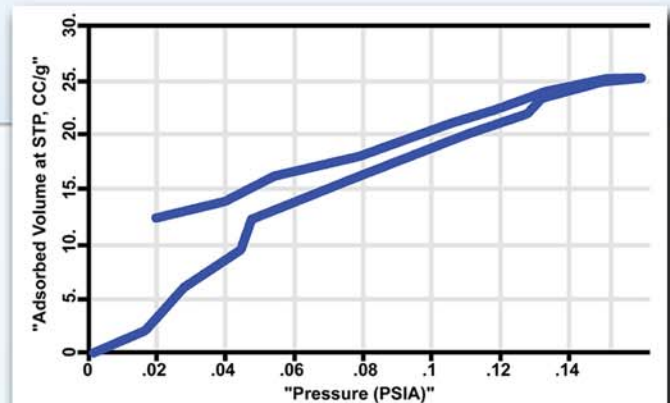
Adsorption and desorption of gasses on samples can be accurately measured using PMI BET sorptometers. The user has independent control over the quantity and spacing of pressures used in both adsorption and desorption testing. Many different kinds of analyses are available to interpret data using the supplied report generation software. Pore size calculation for adsorption and desorption include Pierce, BJK, and DH models. Microporous solids can be analyzed using T-Plot and H-K analysis.



T - Plot Method — Micropore Volume Analysis

## Chemisorption:

PMI BET Sorptometers can use specialty gasses like  $\text{NH}_3$  and  $\text{H}_2\text{O}$  for measuring chemisorption. Other gasses can be used by creating additional gas specification files. The user can specify any temperature and pressure of gas, limited only by the capabilities of the instrument and the equilibrium vapor pressure of the gas at the temperature selected.



Adsorption and Desorption Isotherms –  
Water Vapor at 0 °C

## Specifications

**Surface Area range:** 0.01 m<sup>2</sup> and higher.

**Pore diameter range:** Micropore to 2000Å.

**Sample volume:** Up to 10 cc (others available).

**Pressure gauge:** 10 torr to 500 psi.

**Resolution:** 1 part in 20,000

**Accuracy:** 0.15% of reading

**Adsorption temperature:** -195.6 deg C (liquid nitrogen) to 300 deg C. (higher temperature available)

**Adsorbate:** Any noncorrosive gas including N<sub>2</sub>, H<sub>2</sub>, CO, CO<sub>2</sub>, H<sub>2</sub>O, Kr, Ar.

**Degassing system:** Heater oven up to 500 deg C (Higher temperatures available).

Degassing and testing performed in-situ.

**Power requirements:** 110/220 Vac, 50/60 Hz

## Unique Features

- ◆ Adsorption of chemically active substances like NH<sub>3</sub>, CO & CO<sub>2</sub>
- ◆ Adsorption of vapors of liquids like water, benzene and alcohol
- ◆ Adsorption studies using H<sub>2</sub>, N<sub>2</sub>, Ar & Kr
- ◆ Adsorption under pressures up to 500 psi
- ◆ Adsorption under extra low pressures down to 10<sup>-5</sup> psi
- ◆ Adsorption studies at temperatures up to 300°C & higher
- ◆ Use of flow method by QBET series permits fast and reproducible surface area measurements
- ◆ Volumetric method employed measures equilibrium amount of adsorbed gas precisely without the possibility of any contamination
- ◆ Design features modifiable to satisfy your special requirement
- ◆ Any number of user specified data points between specified pressure limits
- ◆ Automated calibration routine for different sample chambers
- ◆ Continuous status display
- ◆ Continuous recording of equilibrium (saturation) pressure
- ◆ Data reduction software allows data analysis by many available procedures
  - ✓ Surface area: Single point, Multipoint
  - ✓ Pore Size: Pierce, NJH and DH models
  - ✓ Micropore: t-plot, Langmuir, D-R and H-K model
- ◆ Software enables curvefitting and interpolation of data, output to be written in text and Excel files, and multigraph analysis that can analyze up to seven graphs
- ◆ Fully automated and minimal operator involvement
- ◆ Windows based menu-driven procedure makes test execution, data acquisition, and data reduction very simple
- ◆ In-situ outgassing of samples at temperatures up to 800°C
- ◆ No need for transfer from outgassing station to test station and increase possibility of contamination
- ◆ Multiple sample chambers with provision to use different gases in different sample chambers, and simultaneous testing of multiple samples
- ◆ Unique design permits measurement of very low and high surface areas in the same instrument

## PMI Analytical Services

PMI has many years of experience in analyzing porous materials using the gas adsorption technique. The analytical services division of PMI is well known for providing timely, accurate and reliable contract testing services. Contact PMI for details.

## Models

### QBET Series

Units in this series are basic simple instruments dedicated to quick generation of highly reproducible data on surface area using nitrogen or argon. Each unit has one sample chamber. Liquid nitrogen is the only accessory required to perform the test. Various models in this series are capable of measuring single point or multipoint surface area and pore volume. These units are least expensive, physically small, robust and require very little maintenance. Model Number: QBET-G-X-Y-A

### CBET Series

Instruments in this series are volumetric compact units, but the capabilities are much better than those of the QBET series. These instruments can measure surface area, adsorption and desorption isotherms, pore volume and pore volume distribution using nitrogen, argon and krypton. The results are reproducible and accurate. Models in this series are inexpensive, and robust. These models require very little maintenance. Model Number: CBET-G-D-Y-A

### ABET Series

Models in this series are the most advanced instruments in which many novel and advanced designs are incorporated. These instruments are capable of measuring very low adsorption, chemical adsorption and water vapor adsorption. The results are highly accurate and reproducible. Multiple sample chambers permit testing of as many as six samples at a time. A variety of gases may be used. The advanced series provides many options to the user. Model Number: ABET-G-D-Y-A-L-C-W

### Brief Descriptions of Selected Models

**Model: QBET-N-V-1-A**

This fully automated sorptometer is a single-chamber instrument that can determine single point surface area, multipoint surface area and pore volume using nitrogen as the adsorption gas. The instrument is small, robust and inexpensive. It requires very little maintenance. The sorptometer is ideal for applications such as quality control requiring rapid generation of reproducible data.

**Model: CBET-N / Kr/Ar-D-2-A**

This table top compact model can measure single point surface area, multipoint surface area and pore volume using N<sub>2</sub>, Kr or Ar as the adsorption gas. The instrument is fully automated and has two sample chambers. Two samples can be tested simultaneously. This option gives the opportunity to compare samples with the standard. The model is also robust and inexpensive.

**Model: ABET-G-D-6-A-L-C-W**

This is a sophisticated unit with many capabilities. It uses gases like N<sub>2</sub>, Ar, Kr, H<sub>2</sub> and CO<sub>2</sub> for adsorption and measures single point surface area and pore volume distribution. Chemical adsorption of substances like ammonia, alcohol and benzene is measurable. The results are accurate and reproducible. The instrument is fully automated and requires very little operator involvement. The six sample chambers of the instrument permit simultaneous testing of six samples. The sorptometers are ideal for determination of a variety of physical and chemical characteristics.

#### Symbols

A = fully automated

C = chemical adsorption of many chemical species

D = surface area, pore volume and pore distribution

G = adsorption gas that could be N<sub>2</sub>, Ar, Kr, H<sub>2</sub> or others

L = very low adsorption pressure

V = single point and multipoint surface area and pore volume

W = water vapor adsorption capability

X = type of test

Y = number of sample chambers

## Highlights of PMI BET Sorptometer Models

<b>FEATURES</b>	<b>QBETSERIES</b>	<b>CBET SERIES</b>	<b>ABET SERIES</b>
<b>Principle:</b>	Flow	Volumetric	Volumetric
<b>Design:</b>	Simple	Moderately complex	Complex
<b>Gases:</b>	N <sub>2</sub> /Ar	N <sub>2</sub> /Ar/Kr	N <sub>2</sub> /Ar/Kr/CO <sub>2</sub> / H <sub>2</sub> /NH <sub>3</sub> /Others
<b>Liquid N<sub>2</sub> Bath:</b>	Container with cover	Container with Cu-block and cover	Refill system
<b>Measurable Properties:</b>	Single point and multipoint surface area. Pore volume	Single point & multipoint surface area. Pore volume. Pore distribution. Desorption.	Single point & multipoint surface area. Pore volume. Pore distribution. Desorption. Very low adsorption. Chemisorption. Water vapor adsorption.
<b>Reliability:</b>	Highly reproducible	Highly reproducible and accurate	Highly reproducible and very accurate
<b>Duration of Test:</b>	Short	Moderate	Long
<b>Size:</b>	Small	Table top	Floor model
<b>Maintenance:</b>	Very little	Very little	Regular
<b>Cost:</b>	Low	Medium	High

# A Selection of Other PMI Products



**Capillary Flow Porometer**  
Pore Throat Diameter & Distribution



**Envelope Surface Area Analyzer**  
Surface Area & Average Fiber Diameter



**Liquid Extrusion Porosimeter**  
Through Pore Volume & Diameter, Liquid Flow Rate



**Multihead Bubble Point Tester**  
The Largest Pore Diameter



**Compression Porometer**  
Effects of Compressive Stress on Pore Characteristics



**QC Porometer**  
Rapid Acquisition of Data On Pore Characteristics



**Automated Perm Porometer**  
Liquid Permeability, Pore Diameter & Distribution

**Integrity Tester**  
Filter Integrity



**Permeameter**  
High and Low Gas Permeability



**Clamp-On Porometer**  
Clamp-On Testing For Pore Characteristics

**Mercury / Non-Mercury Pycnometer**  
True Density & Bulk Density



**Mercury / Non-Mercury Porosimeter**  
Pore Volume & Distribution