Particulate Systems

HPVA II

High-Pressure Volumetric Analyzer

A MICROMERITICS BRAND

High-Pressure Volumetric Analysis

HPVA II Benefits

- Dual free-space measurement for accurate isotherm data
- Free space can be measured or entered
- Correction for non-ideality of analysis gas using NIST REFPROP compressibility factors calculated from multiple equations of state
- Reports provided as interactive spreadsheets
- Isotherm and weight percentage plots created automatically
- Tables of raw data used for report calculations
- Real-time charts for Pressure vs. Time and Temperature vs. Time
- Gas mixtures with up to three components can be used
- Kinetic data provided for rate of adsorpotion calculations
- Langmuir equation used to model Type I isotherms
- High-precision, solid-state design high-pressure transducer provides a reading accuracy of ±0.04% full scale with a stablility of ±0.1%
- Low-pressure pressure transducer provides a reading accuracy of ±0.15% of value
- System can attain a maximum pressure of 200 bar
- Hydrogen gas sensor automatically shuts down the system should a hydrogen leak occur
- BET surface area, Langmuir surface area, and total pore volume calculations included

High-Pressure Speciality Applications

The HPVA II Series of adsorption analyzers from Particulate Systems uses the static volumetric method to obtain high-pressure adsorption and desorption isotherms utilizing gases such as hydrogen, methane, and carbon dioxide.

The volumetric technique consists of introducing [dosing] a known amount of gas [adsorptive] into the chamber containing the sample to be analyzed. When the sample reaches equilibrium with the adsorbate gas, the final equilibrium pressure is recorded. These data are then used to calculate the quantity of gas adsorbed by the sample.

This process is repeated at given pressure intervals until the maximum preselected pressure is reached. Then the pressure can be decreased to provide a desorption isotherm. Each of the resulting equilibrium points [volume adsorbed and equilibrium pressure] is plotted to provide an isotherm.

Excellent reproducibility and accuracy are obtained by using separate transducers for monitoring low and high pressures.

Typical HPVA II Applications

Carbon Dioxide Sequestration

Evaluating the quantity of carbon dioxide that can be adsorbed by carbons and other materials is important in the ongoing study of carbon dioxide sequestration. High pressures obtained with the HPVA II can simulate the underground conditions of sites where CO_2 is to be injected. Configuring the HPVA II with a chiller/heater bath allows the user to evaluate the CO_2 uptake at a range of stable temperatures, providing data that can be used to calculate heats of adsorption. These isotherms are typically analyzed up to approximately 50 bar at near ambient temperatures due to CO_2 condensation at higher pressures.

Coal-Bed Methane

Porous coal samples from underground beds can be analyzed with the HPVA II to determine their methane capacity at high pressures. This allows the user to find the methane adsorption and desorption properities of the underground coal beds, which is useful in determining approximate amounts of hydrocarbons available in coal-bed reserves. Kinetic data from the experiments can also show the rate of metane adsorption and desorption on these porous carbon samples at specific pressures and temperatures.

Hydrogen Storage

Determining the hydrogen storage capacity of materials such as porous carbons and metal organic frameworks (MOFs) is pivotal in the modern demand for clean energy sources. These materials are ideally suited for storage because they allow you to safely adsorb and desorb the hydrogen. Stored adsorbed hydrogen in MOFs has a higher energy density by volume than a gaseous hydrogen and does not require the cryogenic temperatures needed to maintain hydrogen in a liquid state. The HPVA II software provides a weight percentage plot that illustrates the amount of gas adsorbed at a given pressure as a function of the sample mass — the standard method for reviewing a sample's hydrogen storage capacity.

Shale Gas

High-pressure methane can be dosed onto shale samples to generate adsorption and desorption isotherms. This provides the methane capacity of the shale at specific pressures and temperatures. The adsorption isotherm can be used to calculate the Langmuir surface area and volume of the shale. The Langmuir surface area is the surface area of the shale assuming that the adsorbate gas forms a single layer of molecules. The Langmuir volume is the uptake of methane at infinite pressure — the maximum possible volume of methane that can be adsorbed to the surface of the sample.

HPVA II Features

- Wide Operating Pressure range: High Vacuum to 100 or 200 bar
- Broad Temperature Capability: From cryogenic to 500 °C
- Excellent control of sample temperature by means of a recirculating temperature bath, cryogen dewar, or furnace
- Manifold temperature controlled with heater for stability and accuracy
- Fully automated analysis using interactive software
- Excellent data reproducibility
- Handles typical adsorbates such as nitrogen, hydrogen, methane, argon, oxygen, and carbon dioxide
- Comprehensive Data Analysis package using Microsoft® Excel® macros for data processing and graphing
- Software includes NIST REFPROP

HPVA II System

Four Methods of Sample Temperature Control

- Refrigerated/heated recirculation vessel [customer provides temperature control bath]
- Four-liter, stainless-steel dewar for liquid cryogen
- Furnance allows for experiments ranging up to 500 °C
- Cryostat can precisely control sample temperatures from ambient conditions to 30 K

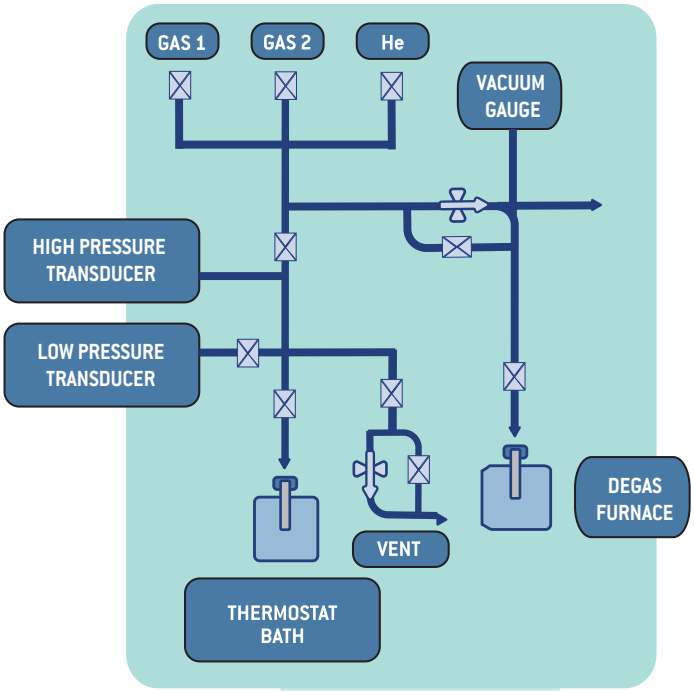
Manifold

All the valves in the manifold are pneumatically operated, high-pressure valves with Kel-F® seats. Valve tubing is constructed with heavy wall, 316L stainless steel and is attached via a VCR connection or welded. The temperature of the insulated manifold region is stabilized using a heater controlled by an adjustable PID controller.

Pressure Transducers

Two transducers are used to precisely measure the system pressure. A 1000-torr transducer is used to accurately monitor pressures below 1 atmosphere and is protected from high pressure with an isolation valve and a cracking valve that relieves to the vent.

SYSTEM SCHEMATIC



Servo Valves

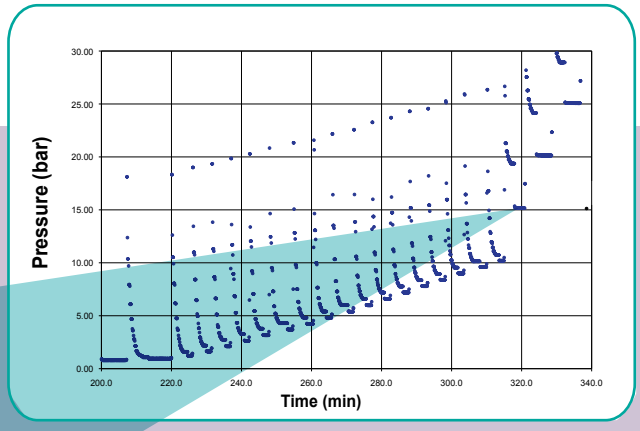
The servo valves are used to automatically regulate flow of the gas in the manifold to the vent and vacuum.

Vacuum System

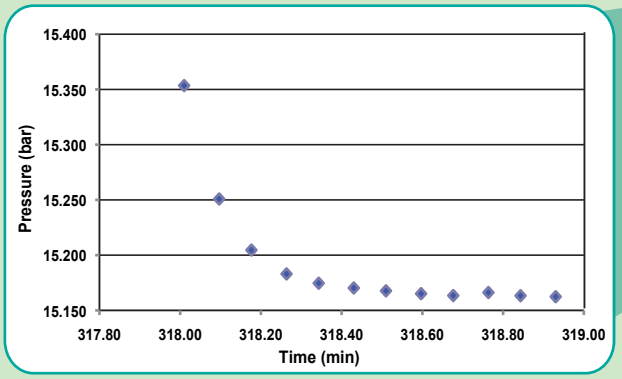
Consists of a mechanical pump and internal Pirani vacuum gauge. User can provide their own pump or purchase the high-vacuum turbo pump package.

A MICROMERITICS BRAND

Supplemental HPVA II Reporting

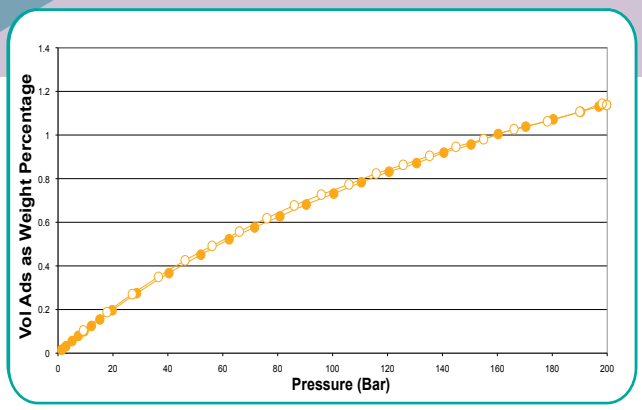


H_2 Uptake on Cu(BTC) at 100 K, Kinetic Data

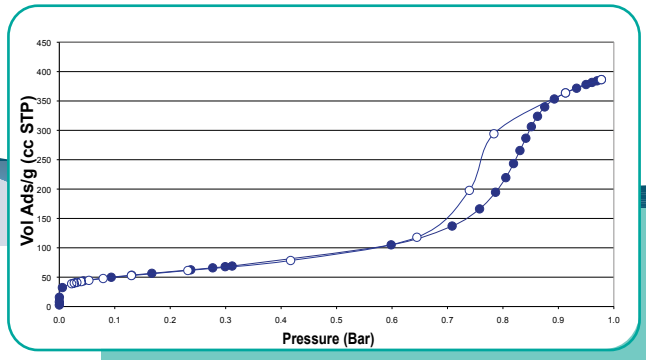


H_2 Uptake on Cu(BTC) at 100 K, Kinetic Data

The decrease in pressure indicates the adsorption of the gas.



Weight Percentage Uptake of H_2 on MOF (CuBTC)

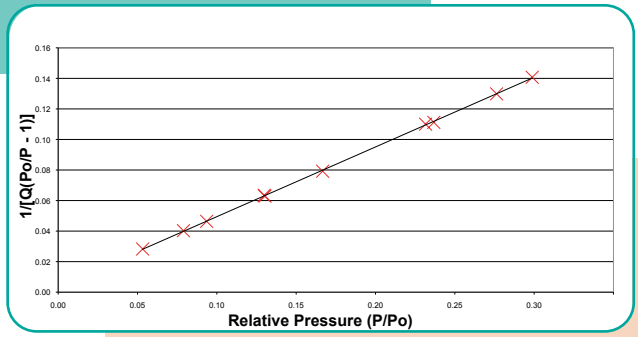


Nitrogen on Silica Alumina

Data Reduction

The HPVA II software uses a National Instruments data acquisition interface to communicate with the analyzer. The data acquired during analyses are written to files that are read by a macro written in Microsoft Excel.

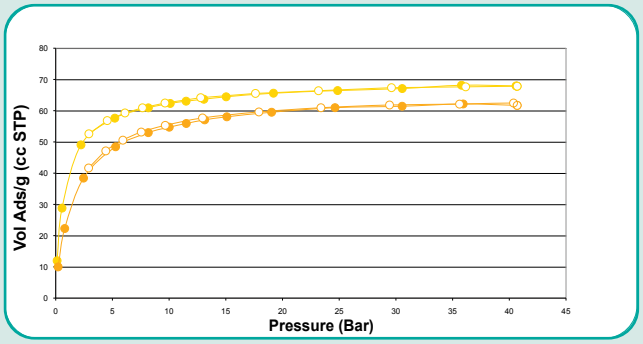
The macro uses the temperature and pressure data to obtain the corresponding compressibility factors from NIST REFPROP software to correct for the non-ideality of the high-pressure gases. Data reduction using the Excel macro provides reports as interactive spreadsheets which list the temperature and pressure data used for volume adsorbed calculations as well as excess isotherm, weight percentage, Langmuir theory, kinetic data plots, BET surface area, and total pore volume.



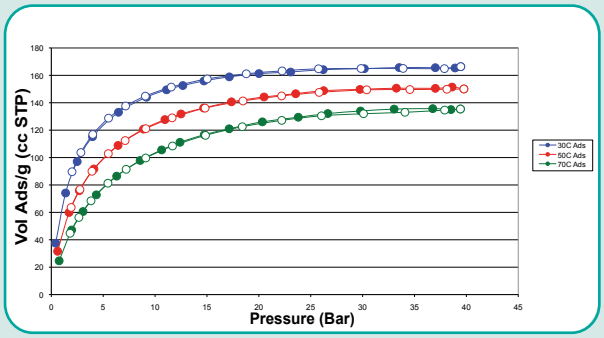
BET Surface Area Plot, SiAl

HPVA II Reporting

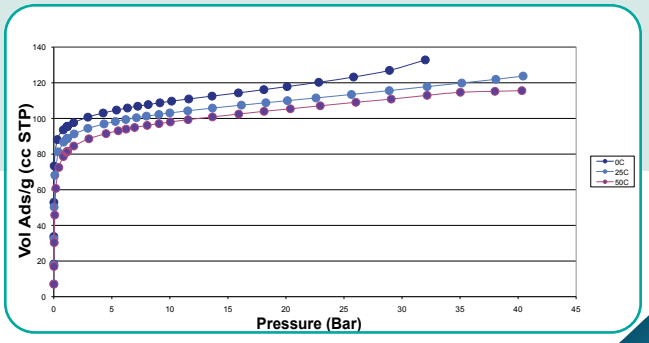
CO_2



Two Molecular Sieves at 25 °C

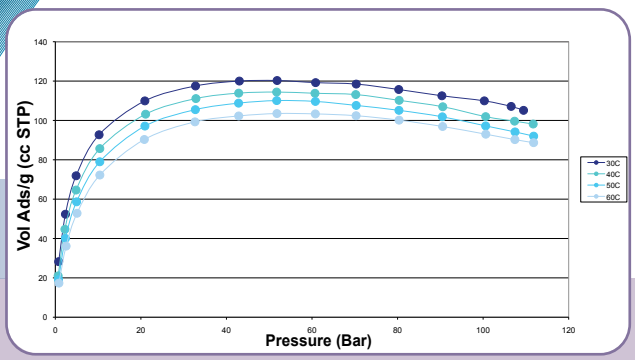


Microporous Carbon at Various Temperatures

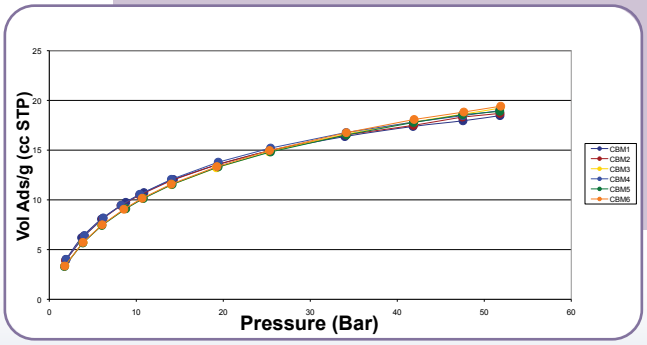


Zeolite 5A at Various Temperatures

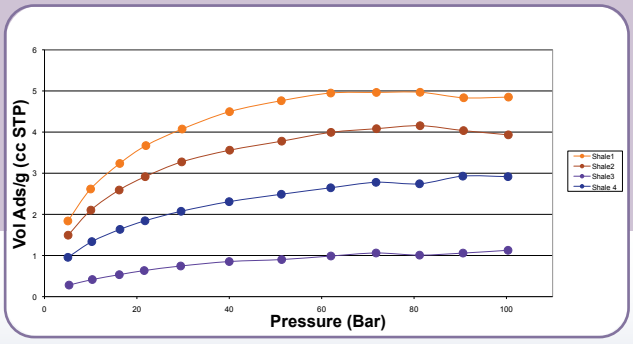
CH_4



S-III Microporous Carbon at Various Temperatures



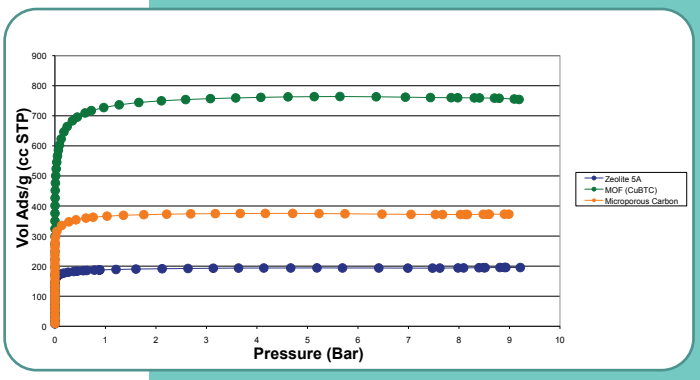
Six Different Coal Bed Samples at 30 °C



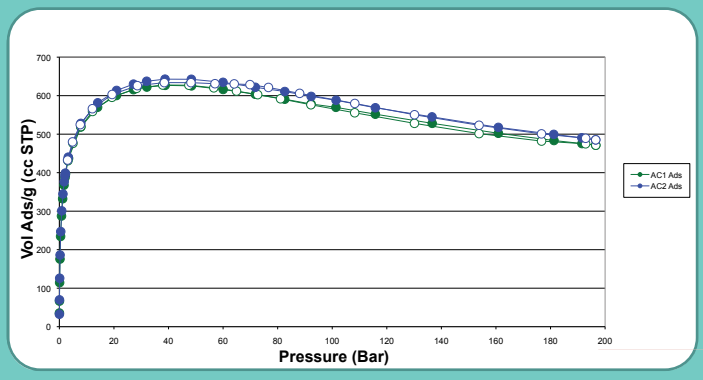
Four Different Shales at 25 °C

Additional HPVA II Reporting

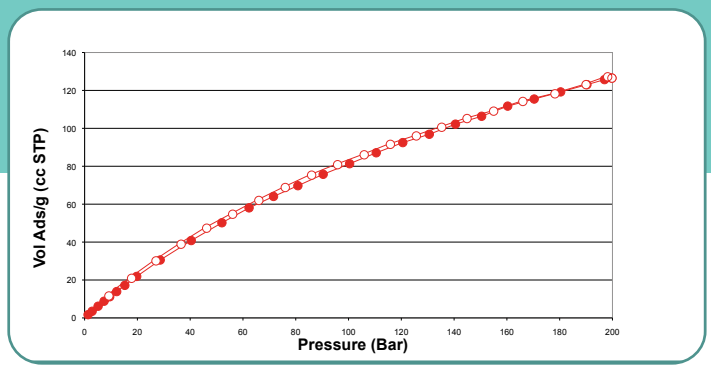
H_2



Various Materials at 30 K



Two Activated Carbons at 77 K



MOF (CuBTC) at O °C

Cryostat

For extensive hydrogen storage studies at high pressures and low temperatures, the HPVA II can be interfaced with a cryostat to control analysis temperatures down to 30 K with a stability of ±0.003 K. The cryostat does not require liquid cryogens for operation; instead, it utilizes the Gifford-McMahon refrigeration cycle where pressurized helium is supplied from a compressor to produce cold temperatures. The HPVA II software communicates directly with the cryostat temperature controller allowing for precise temperature measurements recorded over the duration of the adsorption experiment. Generating hydrogen adsorption isotherms at multiple cryogenic temperatures presents researchers the advantage of more accurately calculating the isosteric heats of adsorption of hydrogen on their materials of study.

Particulate Systems

Specifications

Physical	Height:	88.9 cm (35 in.)
	Width:	50.8 cm (20 in.)
	Depth:	50.8 cm (20 in.)
	Weight:	27.2 kg (60 lbs)
Electrical	Voltage:	100 - 240 VAC
	Frequency:	50 to 60 Hz
Environment	Temperature:	10 to 45 °C (50 to 113 °F), operating; -10 to 55 °C (14 to 131 °F), non-operating
Computer	Processor:	Pentium IV 2.8 GHz processor
	Memory:	512 MB RAM
	Hard Disk Space:	80 gigabytes
	Monitor:	SVGA
	Media Drive:	Writable CD or DVD
	Interface:	Available PCI Slot
	Software:	Windows® XP Professional Windows 7 Microsoft Excel® - 2002 or higher

Particulate Systems
4356 Communications Drive
Norcross, GA 30093
USA
Telephones:
Sales 770.662.3636
Fax 770.662.3696

For additional product and materials analysis service details visit:

www.particulatesystems.com

P02/42700/00