AUTOSORB®-1 SERIES

SURFACE AREA, PORE SIZE AND CHEMISORPTION

QUANTACHROME

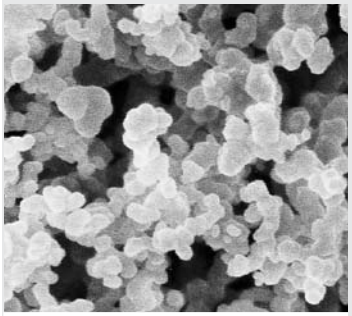
Quantachrome
INSTRUMENTS

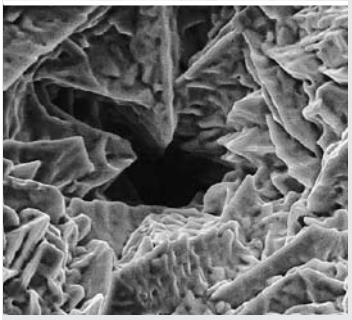
Industries Served

Particle characterization technology serves a wide variety of industries, including:

Aerospace
Agriculture
Automotive
Aviation
Building Materials
Ceramics
Chemicals
Communications Equipment
Construction
Consumer Goods
Cosmetics
Electrical & Electronics
Environmental Services
Foods
Food Processing
Manufacturing
Marine
Medical Devices
Metals
Mining & Minerals
Munitions
Oil Exploration
Optics
Paints & Coatings
Paper & Packaging
Petrochemicals
Pharmaceuticals
Plastics
Rubber
Textiles
Water Treatment

Photomicrograph:
▼ Carbon black





▲ Photomicrograph:
Prickly gold

Quantachrome
INSTRUMENTS

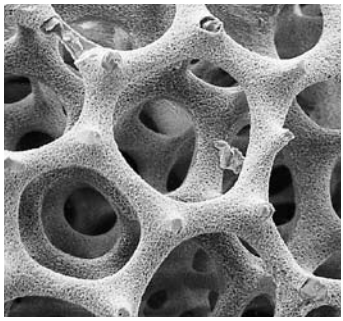
The

The structure and reactivity of so
molecules. Quantifying gas–soli
routine evaluation of properties t
of p

All matter is made up of atoms. In gases, atoms and molecules are free to move about in space. In contrast, atoms in solids are located in fixed positions by electrical forces of attraction among neighboring atoms. But the outermost (or *surface*) atoms in the solid have fewer neighbors than the atoms beneath them. To compensate for their electrical force imbalance, surface atoms seek to attract surrounding gas molecules. The tendency of all solid surfaces to attract surrounding gas molecules gives rise to a process called gas sorption. As-illustrated by the ensuing examples, monitoring the gas sorption process provides a wealth of useful information about the characteristics of-solids.

Before performing gas sorption experiments, solid surfaces must be freed from contaminants such as water and oils. Surface cleaning (degassing) is most often carried out by placing a sample of-the solid in a glass cell and-heating it under vacuum. Figure 1 illustrates how a solid particle containing cracks and orifices (*pores*) of different sizes and shapes may look after its pretreatment.

Once clean, the sample is brought to a constant temperature by means of an external bath. Then, small amounts of a gas (the *adsorbate*) are admitted in steps into the evacuated sample chamber. Adsorbate molecules quickly find their way to the surface of every pore in the solid (the *adsorbent*). These molecules can either bounce off or stick to the surface. Gas molecules that stick to the surface are said to be *adsorbed*. The strength with which adsorbed molecules interact with the surface determines if the adsorption process is to be considered physical (weak) or chemical (strong) in nature.



▲ Photomicrograph:
Radiolaria

Physical adsorption (*physisorption*) is the most common type of adsorption. Physisorbed molecules are fairly free to move around the surface of the sample. As more gas molecules are introduced into the system, the adsorbate molecules tend to form a thin layer that covers the entire adsorbent surface. Based on the well-known Brunauer, Emmett and Teller (BET) theory, one can estimate the number of molecules required to cover the adsorbent surface with a *monolayer* of adsorbed

Quantachrome has fostered

Gas Sorption Process

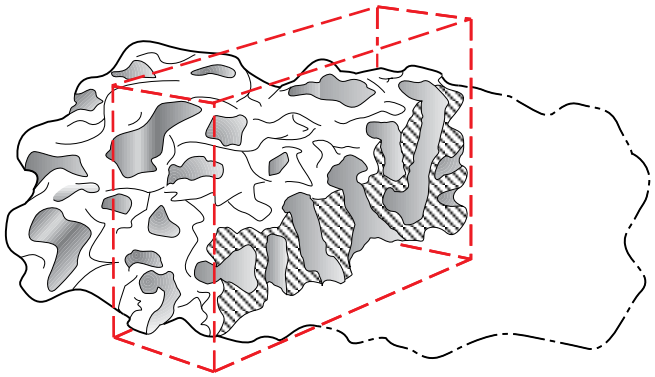
olid materials determine the manner in which their surfaces interact with gas
d interactions through the generation of gas sorption isotherms permits the
hat control the performance of solids, such as surface area, size and shape
ores, chemically active sites, and many others.

molecules, N_m (see Figure 2). Multiplying N_m by the cross-sectional area of an adsorbate molecule yields the sample's ***surface area***.

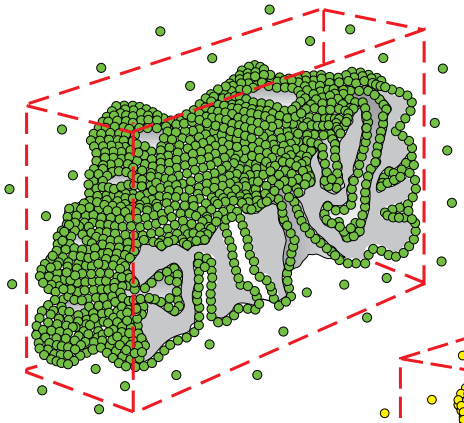
Continued addition of gas molecules beyond monolayer formation leads to the gradual stacking of multiple layers (or multilayers) on top of each other. The formation of multilayers occurs in parallel to capillary condensation (see Figure 3). The latter process is adequately described by the Kelvin equation, which quantifies the proportionality between residual (or equilibrium) gas pressure and the size of capillaries capable of condensing gas within them. Computational methods such as the one by Barrett, Joyner and Halenda (BJH) allow the computation of pore sizes from equilibrium gas pressures. One can therefore generate experimental curves (or *isotherms*) linking adsorbed gas volumes with relative saturation pressures at equilibrium, and convert them to cumulative or differential ***pore size distributions***.

As the equilibrium adsorbate pressures approach saturation, the pores become completely filled with adsorbate (see Figure 4). Knowing the density of the adsorbate, one can calculate the volume it occupies and, consequently, the ***total pore volume*** of the sample. If at this stage one reverses the adsorption process by withdrawing known amounts of gas from the system in steps, one can also generate *desorption* isotherms. Since adsorption and desorption mechanisms differ, adsorption and desorption isotherms rarely overlay each other. The resulting *hysteresis* leads to isotherm shapes that can be mechanistically related to those expected from particular ***pore-shapes***.

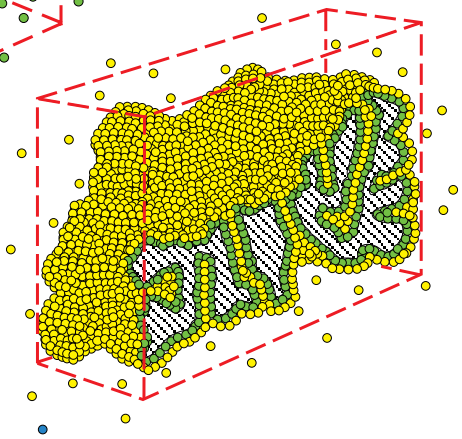
In contrast to physisorption, **chemical adsorption** (*chemisorption*) involves the formation of strong chemical bonds between adsorbate molecules and specific surface locations known as ***chemically active sites***. Chemisorption is thus used primarily to count the number of surface active sites which are likely to promote chemical and catalytic reactions.



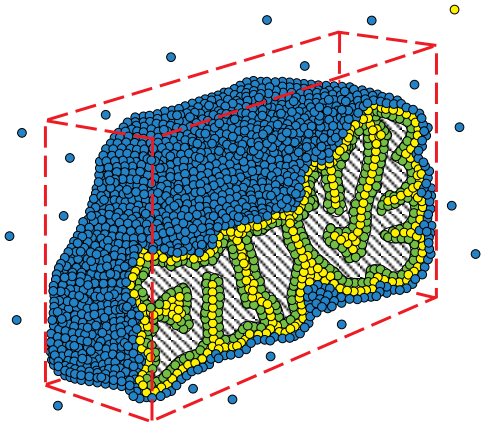
① A section of one greatly enlarged particle of a solid.



② The monolayer of adsorbed molecules; approximately 15% saturation.



③ The multilayer/capillary condensation stage; approximately 70% saturation.



④ Total pore volume filling; approximately 100% saturation.

e's pioneering development of gas sorption instruments
worldwide R&D and quality control efforts for decades.

AUTOSO

PRECISE • RELIABLE • FAST

Windows® based performance

The **AUTOSORB-1** analyzer is microprocessor controlled, and communicates with a Windows 95, 98, 2000 based PC utilizing Quantachrome's state-of-the-art, data acquisition and data reduction software.

Comprehensive software to meet modern needs

Whether a laboratory's need is for wide ranging physisorption studies or complete automation of the catalyst treatment and multiple pass chemisorption process, the **AUTOSORB** software serves admirably. It also serves two or more analyzers... and two or more users connected via a network. And, after the accumulation of many data files, a built-in database engine quickly locates desired files by searching for a specific ID, description, operator, comment or range of dates.

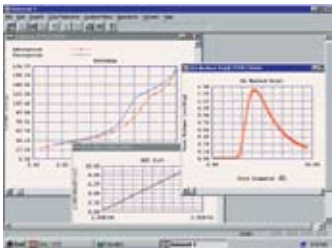
The user-friendly software

▲ Analysis parameters in Autosorb software

guides you through sample preparation, preprogrammed parameter recall or making settings for operations, data reduction, graphs and report printouts.

During operation one can view the accumulated data, the isotherm and all associated graphs and analytical results up to that point.

After a run, reports and graphs are printed automatically or the operator can use the software to determine the best fitting method, to compare data by overlaying curves or to adjust graph, size, scaling, titles, plot markers and line colors for best



▲ Mutiple graphs to view isotherm and related plots

print out.

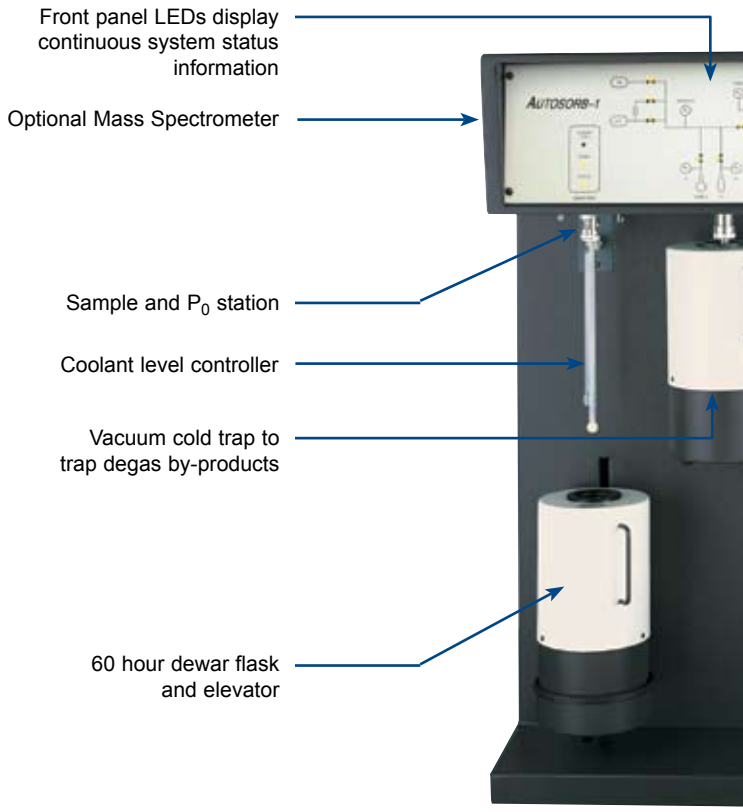
Data presentation

A comprehensive range of surface area and pore size methods is available:

- Adsorption and desorption isotherms.
- Multi and single point BET surface area (including C constant and correlation coefficient).
- Langmuir surface area
- Mesopore volume and area distribution (BJH and DH methods).
- Standard micropore size distribution (MP method) and t-method by deBoer, Halsey or carbon black (STSA).
- Total pore volume, average pore size and sample density.
- Dubinin-Radushkevich micropore surface area
- Horvath-Kawazoe, Dubinin-Astakhov and Saito-Foley micropore distribution.
- Full Density Functional Theory library for unified micro- and mesopore analysis using N_2, Ar and CO_2 on materials such as zeolites, MCM-41, carbons and silicas.
- Monte Carlo based pore size model.

For chemisorption analysis, standard calculations include:

- Combined, strong and weak, (reversible and irreversible) isotherms.
- Monolayer coverage by extrapolation or bracketing of isotherm data and Temkin or Freundlich plots.



Functional Capability by Model	AUTOSORB -1	AUTOSORB -1-MP	AUTOSORB -1-C
Surface area analysis	✓	✓	✓
Mesopore size distribution	✓	✓	✓
Standard micropore methods	✓	✓	✓
Advanced, low pressure micropore analysis	–	✓	✓
Chemisorption methods	–	–	✓

▲ A variety of sample cells are available for all applications.

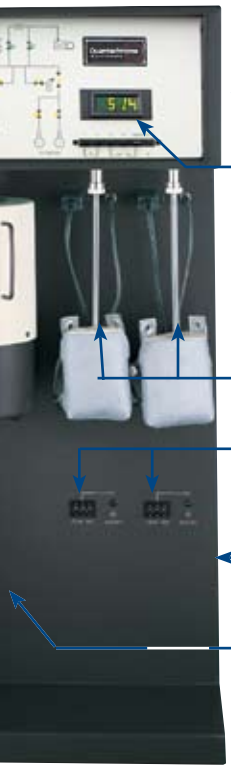
- Active metal area, degree of metal dispersion and average crystallite size.
- Heats of adsorption.
- Fractal dimension.

Sample preparation

Sample surface contaminants are removed by heating under vacuum. Two automated degassing stations may operate concurrently with the analysis station. PASS/FAIL degas test with digital display of degas pressure permits user to set and monitor sample preparation more precisely.

RB®-1 Series

• VERSATILE • ECONOMICAL



Vapor Sorption option w/ heated manifold and vapor generator

Multifunction temperature/ pressure meter displays:

- manifold pressure
- manifold and degas temperatures
- station pressure
- saturated vapor pressure

Sample preparation stations (four with Degas Doubler)

Heating mantle digital temperature controls

Gas inlet ports *located on rear panel*

Vacuum pump for analysis and degassing stations *(inside cabinet)*

- Oil-free turbomolecular vacuum pump (-MP and -C models)

Functional Capability by Model	AUTOSORB -1	AUTOSORB -1-MP	AUTOSORB -1-C
Number of pressure transducers	3	5	5
Very low surface area analysis with krypton	–	✓	✓
Sample preparation by high vacuum	–	✓	✓
Automatic gas switching (5 ports)	–	–	✓
Sample preparation/activation by gas flow	–	–	✓
Available vapor sorption option	–	✓	✓

AUTOSORB-1-MP and -C models provide sample preparation in an oil-free vacuum at the same high vacuum as required for analysis. The special needs of chemisorption preparation are achieved with multiple gas inputs featuring automatic switching. Flow rates for preparation are user selectable for each gas using the built-in flow meter. A programmable temperature controller provides linear heating rates for the furnace

▲ The AUTOSORB-1-C has automatic valves for active and treatment gas selection, built-in gas flow meter and temperature programmer.

AUTOSORB-1 Series Benefits

Superior engineering delivers years of trouble-free operation. All process control features are self-contained, enabling the instrument to function independently without operator intervention or external computer support.

Designed for operator convenience and ease of use

With minimal operator training, the AUTOSORB-1 produces the results you need.

- Store a limitless number of user-defined analyses for fast and simple measurement initialization.
- Automatic error checking ensures reliable data.
- Front panel LED display of-valve and dewar status provides "at-a-glance" confirmation of system operation.
- Easy-fill dewar flasks offer safe and simple handling of cryogenic liquids.
- Benchtop and floor standing units satisfy any laboratory space requirement.
- Excellent graphical interface offers manual control of all instrument functions. It also aids in maintenance and system diagnostics.

Meets the most rigorous technical demands

- Separate sample cell transducers monitor equilibration in the smallest possible volume. This gives maximum sensitivity while a sample cell is isolated from the dosing manifold.
- Dedicated P_o transducer updates each data point for maximum accuracy of surface area and pore size measurements.
- Sample preparation under high vacuum of turbomolecular pump offers a more complete degassing.
- Welded, stainless steel dosing manifold ensures compatibility with a wide range of gases and contamination-free operation.
- Thermistor sensor coolant level control with TempComp™ for improved void volume accuracy and sensitivity.
- Built-in, automated calibration maintains optimum performance consistent with ISO 9000 requirements.
- Onboard computer continuously monitors the dosing manifold temperature and automatically compensates for environmental temperature changes.

Feature-rich, extra touches define quality

- Oil-free turbomolecular drag pump eliminates oil backstreaming (-MP and -C models).
- Cold trap eliminates degas byproducts.
- Tandem manifolds for low maintenance, high efficiency single vacuum system.

Comprehensive, advanced chemisorption capabilities

- Fully integrated chemisorption option with small bench-space requirements.
- PC programmable temperature controller with auto-tuning functions offers limitless number of preparation routines with guaranteed temperature accuracy.
- Automatic repeat measurement of isotherm following evacuation provides complete weak and strong chemisorption data without operator intervention.
- Multiple, sequential unattended measurements at operator selected temperatures.

SPECIFICATIONS

	AUTOSORB-1 General Physisorption Measurement	AUTOSORB-1-MP Detailed Micropore Characterization
Pressure Transducers *(per manufacturer's specifications)*		
0 to 1,000 torr	yes	yes
Accuracy: ±0.11% full scale		
Minimum resolvable pressure: 2.5 x10^{-4} torr		
Minimum resolvable relative pressure (P/P_o): 3 x 10^{-7} (nitrogen)		
0 to 10 torr [Optional 0 -1 torr]	no	yes [yes]
Accuracy: ±0.15 % reading		
Minimum resolvable pressure: 2.5 x 10^{-6} torr [2.5 x 10^{-7} torr]		
MInimum resolvable relative pressure (P/P_o): 3 x 10^{-9} [3 x 10^{-10}] (nitrogen), 7.6 x 10^{-5} (krypton)		
Ultimate Vacuum *(per manufacturer's specifications)*		
Vacuum Pump: 1.1 x 10^{-3} torr	yes	yes
Turbo Molecular Pump: <5 x 10^{-9} torr	no	yes
Adsorbates		
Non-corrosive gas (N_2, Ar, CO_2, C_4H_{10}, etc.)	yes	yes
Krypton gas	no	yes
Corrosive vapors (NH_3, cyclohexane, etc.)	no	optional
Programmable 5-gas switching	no	no
Temperature Capabilities		
Degas Heating Mantles (2 stations)	yes	yes
Heating up to 400°C in steps of 1°C		
Accuracy within 5°C of set point		
Optional quartz mantles for higher temperatures		
Analysis Heating Furnace	no	no
Heating to 1,100°C with automatic calibration		
Programmed ramp rates from 1 to 50°C/min.		
Unlimited number of heating/cooling programs		
Auto-Leveling Cooling Bath	yes	yes
Two liter standard (60 hours)		
One liter, wide mouth (optional)		

Physical

Operating Temperature
10°C to 38°C, non-condensing

Electrical
100 to 240 V, 50/60 Hz
750 VA without furnace
1200 VA with furnace

Dimensions
Width : 25.5 in. (63.5 cm)
Depth: 29.0 in. (72.2 cm)
Height: 40.0 in. (99.6 cm)

Weight
Installed: 335 lbs. (161 kg)
Includes internal vacuum pump(s)

Steel Construction
Cabinet and optional rolling cart

Performance

Surface Area Analysis
Nitrogen range:
0.01 m^2/g to
no known upper limit
Krypton range:
0.0005 m^2/g to
no known upper limit

Pore Analysis
Detectable volume limit:
<0.0001 cc/g
Pore size range:
3.5 to >4000 ångströms

Analysis Station Components

1 Sample cell with dedicated pressure transducer(s)
1 Saturated vapor pressure (P_o) cell with dedicated transducer
1 Dewar bath level sensor for liquid coolant or slurries

Sample Cell Volumes

	Stem OD		
	6 mm	9 mm	12 mm
Small Pellet cm^3	1.40 cm^3	1.80 cm^3	2.40
Large Pellet cm^3	5.50 cm^3	5.50 cm^3	5.50
Micro-			

Special cells available upon request.

AUTOSORB-1-C Chemisorption and Physisorption Capability

yes

yes [yes]

yes
yes

yes
yes
yes
yes

yes

yes

yes

System Requirements

Computer, Recommended:
Pentium, 16Mb RAM, mouse, color printer

Operating System:
Standard Windows 3.1 or higher

Prompt, Dependable Service… Worldwide

At Quantachrome, reliability means more than product performance...it means responsiveness.

You can depend on our staff for on-site installation and service, prompt factory repairs, and telephone troubleshooting support.

The purchase of a Quantachrome product signifies the beginning of a long term relationship, with a-goal-to ensure the-maximum return-on your investment.

Global Network of Distributors

With more than 50 offices worldwide, Quantachrome's distribution network delivers products and support on a global basis...truly a single source for all your particle characterization instruments.

Other Fine Products from Quantachrome

Quantachrome markets a-full range of particle technology characterization instruments, including:

- **AUTOSORB® 6B & 3B** for automated, independent multiple sample sorption measurements.
- **CHEMBET™-3000 TPR/TPD** for chemisorption studies and catalyst characterization.
- **NOVA® Series** for simultaneous gas sorption analysis of 1, 2, 3 or 4 samples
- **MONOSORB®** for automated single point B.E.T. surface area determinations.
- **HYDROSORB™** for water vapor sorption analysis.
- **THERMOFLOW** degassing-instruments.
- **FLOW CONTROLLER** for gas mixing.
- **POREMASTER® Series** Mercury Porosimeters (60,000 psi and 33,000 psi) for a-full range of pore size distribution.
- **ULTRAPYCNOMETER 1000 & MICRO-ULTRAPYCNOMETER, PENTAPYCNOMETER, & ULTRAFOAM PYCNOMETER** for automatic volume and true density measurements.
- **MULTI & STEREOPYCNOMETERS®** for-manual volume and true density measurements.
- **AUTOTAP & DUAL AUTOTAP** for tap volume-and tap density measurements.
- **SIEVING & ROTARY MICRO RIFFLERS** for accurate representative sampling.
- **REFERENCE MATERIALS** for surface area, metal dispersion, pore volume and pore size.

AUTOSORB-1 Series Product Overview

Quantachrome's AUTOSORB-1 Series offers a full line of high quality, high performance Surface Area and Pore Size Analyzers with three fully automatic models to meet the-needs of any research or quality assurance laboratory.

The AUTOSORB-1 for standard applications using a variety of-adsorbates

- Fully automated analyzer for surface area and pore size measurements.
- Simultaneous analysis and sample preparation.
- System supplied complete and ready for operation.
- Low maintenance, vacuum volumetric system with stainless steel manifold construction.
- Speed and precision ensured by MAXIDOSE™, a proprietary operating algorithm.
- DoseWizard™ learn mode and InitialFill™ for analysis time savings up to 60%.
- High sensitivity ensured by-multiple pressure transducers, minimum void volume and accurate coolant-level control.
- Operates with any non-corrosive adsorbate and a wide variety of coolants, including slurries.

AUTOSORB-1-MP for low surface area and micropore measurement

- Allows adsorption data acquisition at relative pressures below 1-x-10^{-7} utilizing 1 torr transducers.
- Patented oil-free turbomolecular vacuum system eliminates any risk of oil backstreaming.
- Enhanced 22 bit A/D converter and micropore dosing algorithm allow precise data-acquisition of up to 200 data points, sub-ångström resolution and automatic correction for low pressure thermal transpiration.
- Ideal for detailed micropore-size measurements of zeolites, carbons, molecular sieves, etc.
- Many modern data reduction models and dosing algorithms for maximum flexibility.
- Well suited for low surface area analysis with krypton gas.

AUTOSORB-1-C for total precise, chemisorption/ physisorption measurement

- Unattended chemisorption measurement with automatic transfer from *in-situ* sample preparation to analysis.
- Patented oil-free turbomolecular vacuum system eliminates any risk of oil backstreaming.
- Sample preparation with flow, vacuum or static conditions under programmable heating rates at temperatures up to 1,100°C. Prep gas flows through powder bed for thorough reduction.
- Built-in, five port gas input manifold with automatic gas switching.
- Applications include characterization of metal dispersion, acid site distribution, crystallite size, heats of adsorption and much more.
- Compatible with broad range of gases and vapors including hydrogen, carbon monoxide, ammonia, and cyclohexane (optional seals available).
- Optional interface for mass spec or GC.

VAPOR SORPTION option for AUTOSORB -1-C and AUTOSORB-1-MP

- Thermostatted dosing manifold to reduce vapor condensation.
- Built-in, heated vapor generator.
- Specially designed vapor dosing software for precise vapor isotherms.

Vapor generation controls.
(Vapor source visible through transparent cover)

Applications for Quality Control and Research

The list of materials and products-employing Quantachrome's particle characterization technology is-as-diverse as industry itself:

Carbon for rubber, adsorbents (gas separation and water purification), gas masks, inks, laser printers and-copiers

Catalysts for the automotive, fertilizer and petrochemical industries

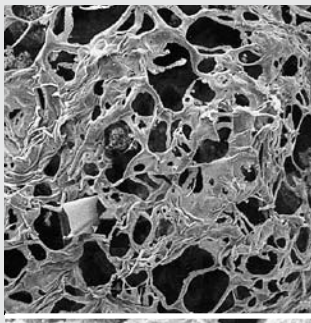
Organic materials for adhesives, chromatography, cosmetics, detergents, foodstuffs, explosives, ion exchange resins, pharmaceuticals and plastics

Minerals such as alumina, clays,-hydroxyapatite, pigments, phosphates, silicas, zirconia, etc., used for abrasives, adsorbents, biomaterials, ceramics, cements, desiccants, fillers, papers and-paints

Powdered metals and ferrites for batteries, pressure formed/ sintered products, electronics, magnets and magnetic tape

Other applications related to bone, composite materials, fibers, rigid foams, soil, sludge, slurries, suspensions and well cores

Photomicrograph:
▼ Coal ash



▲ Photomicrograph:
Natural zeolite

optimizing PARTICLE PERFORMANCE

Quantachrome Instruments' corporate headquarters in Boynton Beach, Florida.

Quantachrome®

Renowned innovator of ideas for today's porous materials community.

For almost 40 years, Quantachrome's scientists and engineers have revolutionized measurement techniques and designed instrumentation to enable the accurate, precise, and reliable characterization of powdered and porous materials:

- Adsorption/Desorption Isotherms
- Surface Area Measurement
- Pore Size Distribution
- Chemisorption Studies
- Water Sorption Behavior
- Mercury Porosimetry
- True Solid Density
- Tapped Density

Not only are Quantachrome products the instruments of choice in academia, but the technology conceived and developed by our expert staff is applied in industrial laboratories worldwide, where research and engineering of new and improved porous materials is ongoing. Manufacturers also rely on porous materials characterization technology to more precisely specify bulk materials, to control quality, and to isolate the source of production problems with greater efficiency.

Quantachrome is also recognized as an excellent resource for authoritative analysis of your samples in our fully equipped, state-of-the-art powder characterization laboratory.

Quantachrome Instruments Application Laboratory.

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