

AUTOSORB-6B[®]

SURFACE AREA & PORE SIZE ANALYZER



Quantachrome
INSTRUMENTS

Windows® based performance

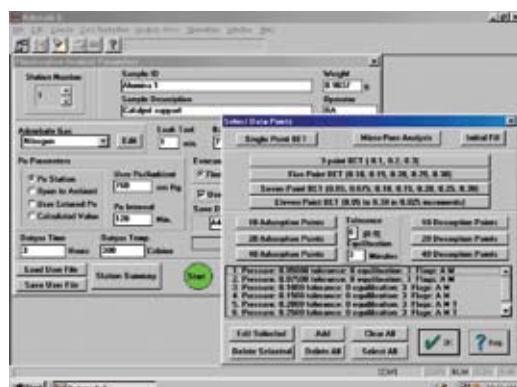
The AUTOSORB-6B analyzer is microprocessor controlled, and communicates with a Windows® 95, 98, 2000, XP based PC utilizing Quantachrome's state-of-the-art, data acquisition and data reduction software.

Comprehensive software to meet modern needs

The Autosorb software is highly functional and user friendly. The software can be utilized to control multiple analyzers or accessed via network by multiple users for data reduction. The software incorporates an advanced built-in database that allows users to quickly search accumulated data files by specific ID, description, operator, comment or range of dates. The Autosorb software is superior for data reduction, incorporating classical methods and the latest DFT (Density Functional Theory) and Monte Carlo models.

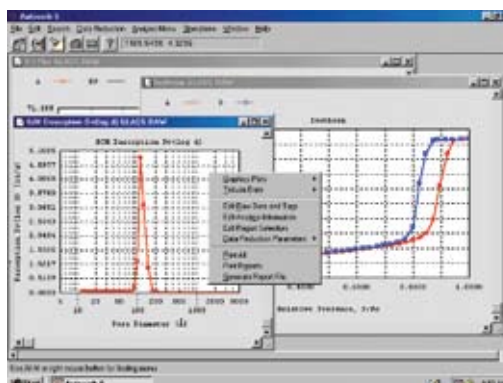
The user-friendly software guides you through analysis setup, preprogrammed parameter recall or making settings for operations, data reduction, graphs and report printouts.

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



▲ Analysis parameters in Autosorb software

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 print out.



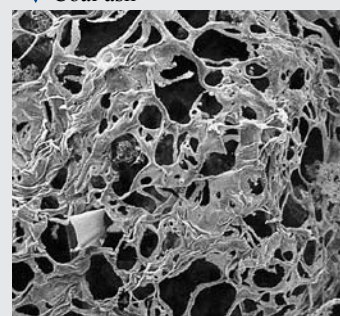
▲ Multiple graphs to view isotherm and related plots

Data presentation

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

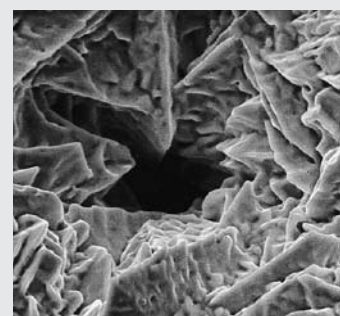
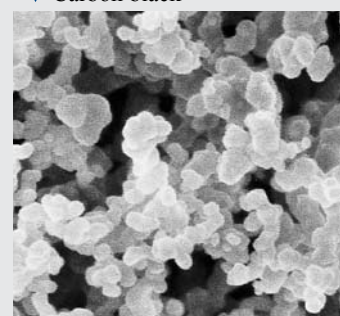
- Adsorption and desorption isotherms.
- Multi and single point BET surface area (including C constant and correlation coefficient).
- Langmuir surface area.
- Mesopore volume and area distribution (BJH and DH methods).
- Standard micropore size distribution (MP method) and t-method by deBoer, Halsey 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 N₂, Ar and CO₂ on materials such as zeolites, MCM-41, carbons and silicas.
- Monte Carlo based pore size model.
- Fractal dimension by FHH or Neimark-Kiselev models.

Photomicrograph:
▼ Coal ash



▲ Photomicrograph:
Natural zeolite

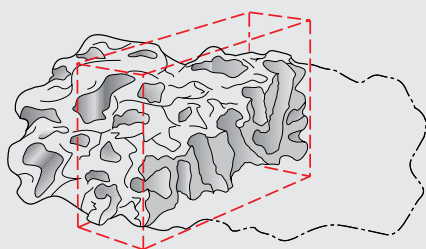
Photomicrograph:
▼ Carbon black



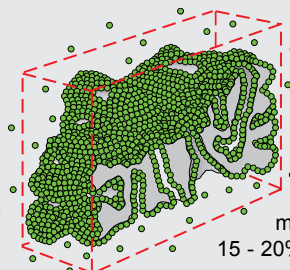
▲ Photomicrograph:
Prickly gold

The Gas Sorption Process

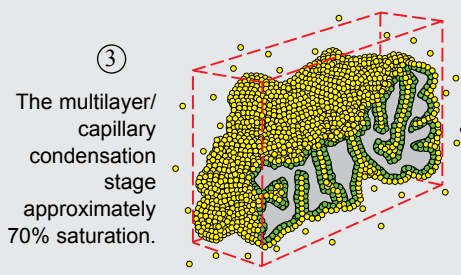
Before performing gas sorption experiments, solid surfaces must be freed from contaminants such as water and oils. Surface cleaning (degassing) is most often carried out by placing a sample of the solid in a glass cell and heating it under vacuum or flowing gas. Figure 1 illustrates how a solid particle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



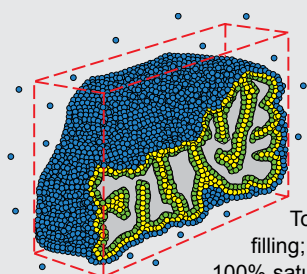
① A section of one greatly enlarged particle of a solid.



② The monolayer of adsorbed molecules; typically 15 - 20% saturation.



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



④ Total pore volume filling; approximately 100% saturation.

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. Gas molecules that stick to the surface of the solid (adsorbent) are said to be adsorbed and tend to form a thin layer that covers the entire adsorbent surface. Based on the well-known Brunauer, Emmett and Teller (B.E.T.) theory, one can estimate the number of molecules required to cover the adsorbent surface with a monolayer of adsorbed 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). The formation 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.

Methods such as the one by Barrett, Joyner and Halenda (B.J.H.) 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. The resulting hysteresis leads to isotherm shapes that can be mechanistically related to those expected from particular pore-shapes.

Quantachrome
INSTRUMENTS

AUTOSORB

QUALITY CONTROL

Multifunction temperature/pressure meter displays:

- manifold pressure
- manifold temperatures
- station pressure
- saturated vapor pressure, P_0

Front panel LEDs display continuous system status information

Six coolant level sensors (one per station)

Optional rolling cart (not shown)



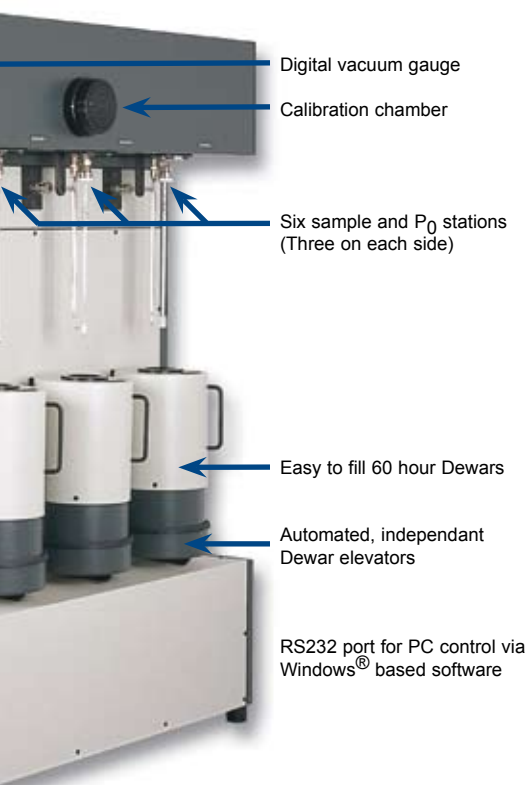
Feature highlights of

Analysis Specifications

Transducer Accuracy:	0.11% full scale (1000 torr transducer) 0.15% reading (10 torr transducer)
A/D Converter:	22-bit (1 part in 4,190,000)
Sensitivity:	$<2 \times 10^{-8}$ moles adsorbed/desorbed
Ultimate Vacuum:	1×10^{-3} torr achieved by dedicated turbomolecular pump 1×10^{-4} torr achieved by turbomolecular pump
Adsorbates:	Nitrogen and any other non-corrosive gas
Surface Area Range:	0.01 m ² /g to no known upper limit
Pore volume:	Detectable limit less than 0.0001 cc/g
Pore size Range:	3.5 to $>4000\text{\AA}$ / 0.35 to $>400\text{nm}$
Coolant level:	Controlled to ± 0.5 mm with RTD sensors
Minimum P/P₀:	1×10^{-3} AUTOSORB 6-B 1×10^{-5} AUTOSORB 6-B-KR/M

Physical Specifications

Dimensions:	Height 40.0 inches (101.6 cm) Width 25.5 inches (64.8 cm) Depth 29.0 inches (73.7 cm)
Weight:	380 Pounds (172 kg)
Electrical:	100 - 240 VAC, 50/60 Hz
Temperature:	10 - 38°C operating range at 90% RH



Digital vacuum gauge

Calibration chamber

Six sample and P_0 stations
(Three on each side)

Easy to fill 60 hour Dewars

Automated, independent
Dewar elevators

RS232 port for PC control via
Windows[®] based software

the AUTOSORB-6B

ducer)
cer - optional)

orbed gas

ted 2 stage rotary, direct drive pump or 1×10^{-7}
r vacuum pump in AUTOSORB-6B-KR/MP

rosive gas with appropriate coolant

mit

1 cc/g

n

D sensor

IP

0% maximum relative humidity

AUTOSORB[®]-6B Overview

Quantachrome's AUTOSORB-6B is designed to satisfy the needs of laboratories with high analytical throughput for rapid and accurate surface area and pore size data.

The AUTOSORB-6B for standard applications using a variety of-gasses

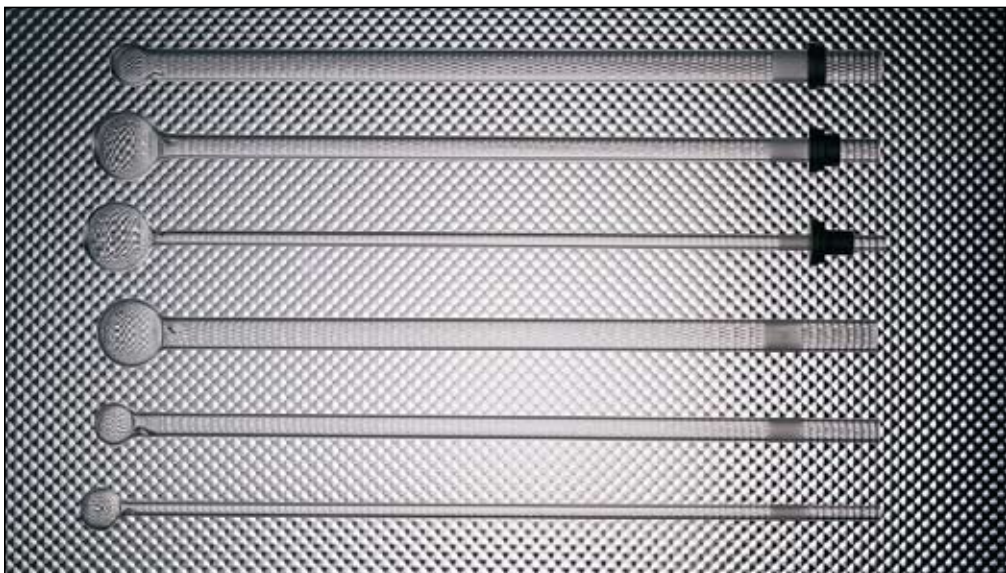
- Fully automated analyzer for surface area, pore volume and pore size measurements.
- High resolution adsorption and desorption isotherms defined with up to 200 equilibrium data points.
- Six analysis ports, each with its own dewar (coolant flask) and pressure sensor, provides independent and simultaneous analyses for the highest analytical throughput.
- Dedicated station pressure transducers ensure equilibration is monitored in smallest enclosed volume in addition to allowing simultaneous operation.
- Dedicated P_0 cells ensure accurate P/P_0 values. Coolant does not have to be of same age, or quality, in all dewars.
- Independent coolant level sensors ensure minimum cold zones to enhance sensitivity and accuracy.
- Fresh samples can be started while other analyses are already in process.
- Low maintenance, vacuum volumetric system with stainless steel manifold construction.
- Speed and precision ensured by MAXIDOSE[™], a proprietary dosing algorithm that responds and adjusts to the adsorption demand of the sample.
- Windows[®] based software provides for instrument control and a comprehensive range of classical and modern models for reporting surface area and pore size.

- May be operated on the benchtop or on a rolling cart to fit any laboratory.
- Wide range of sample preparation degassers available.
- System supplied complete and ready for operation.

AUTOSORB-6B-KR/MP for low surface area and micropore measurement

- All of the functions of the standard model AUTOSORB-6B plus low surface area and micropore capability.
- Includes low pressure (10 torr) sensor and turbomolecular vacuum system.
- Performs krypton gas adsorption measurements for very low surface area determination, ie. pharmaceutical actives, powdered metals, etc.
- Provides low pressure adsorption data (as low as 1×10^{-5} P/P_0) necessary for more complete characterization of microporous materials, eg. zeolites, activated carbons, molecular sieves, etc.

Sample Cell Options



- ▲ Sample cells of 6, 9, or 12 mm may be used with standard cell adapters. (Not all styles shown. Others available for many sample geometries.)

Sample Preparation: Degassers

Consistent and reliable surface area results depend upon proper sample preparation procedures. In terms of B.E.T. analysis, the limiting step in rate of throughput is often sample preparation. The complete degassing

of samples can often require several hours, while surface area measurements may require as little as 8 minutes.

Quantachrome manufactures several models

of degassers to fulfill your sample preparation needs. These degassers provide a virtually continuous supply of properly prepared samples for the Autosorb-6B Surface Area and Pore Size Analyzer.

Feature	MasterPrep™	NOVA® Flow Degasser	NOVA® FloVac Degasser	Autosorb® Degasser
Number of sample ports:	6	6	6	6
Independent temperature control for each sample port:	Yes - with independent ovens	No - all ports at same temperature	No - all ports at same temperature	Yes - with heating mantles
Temperature ramping:	Yes - up to 20 steps, independent for each sample port	No	No	Yes - with manual adjustment
Windows® based PC programmable temperature ramping (digital):	Yes	No	No	No
Vacuum degas mode:	Yes	No	Yes	Yes
Vacuum display:	Yes - digital	Not applicable	Yes - analog	Yes - analog
Flow degas mode:	Yes	Yes	Yes	No
Maximum temperature:	425°C	400°C	400°C	350°C; 450°C with optional quartz mantles
Vacuum pump:	Sold separately	Not applicable	Sold separately	Included (turbo pump optional)

AUTOSORB Degasser

- Six side-by-side sample preparation ports for easy access.
- Optional turbomolecular vacuum system for high vacuum sample preparation.
- Individual digital heat settings and temperature metering.
- Individual temperature “ramping” capability increases heat at selected rate.
- Digital timer for precise preparation times.
- 60 hour cold trap protects vacuum pump from vapor.



The Autosorb Degasser

MasterPrep™ Degasser

- Each of six samples may be simultaneously prepared under different temperature conditions.
- Flow and evacuation rates are operator selectable.
- A six-channel digital temperature controller allows for independent temperature programming (up to 20 steps) on each sample preparation port.
- Windows® based setup software provided.



The MasterPrep Degasser set up for vacuum degassing

The FloVac Degasser & Flow Degasser

For additional flow and vacuum degassing, Quantachrome offers two affordable options: the FloVac Degasser for combined flow or vacuum degassing, and the Flow Degasser offering the flow method only.

- Six sample stations
- Individual control valves to allow the addition or removal of individual sample cells without interrupting the other samples in process.
- Built-in heating mantle provides user-selectable degas temperature to 400°C in steps of 1°C monitored continuously on the digital display.



The FloVac Degasser set up for vacuum degassing



The NOVA Flow Degasser for flow degassing of up to six samples



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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Quantachrome Instruments' quality management system is certified to be in accordance with ISO9001:2000.

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Materials and Powder
Characterization
Needs Since 1968



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