QUANTACHROME

AUTOSORB®-6B
SURFACE AREA & PORE SIZE ANALYZER

Quantachrome
I N S T R U M E N T S

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 gases.

- 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 Po cells ensure accurate P/Po values. Coolant does not have to be of same age, or quality, in all dewars.

- Independant 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.

- System supplied complete and ready for operation - includes vacuum pump, dewar flasks and sample cells.

- Wide range of sample preparation degassers available.

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, orifices and pores of different sizes and shapes might look after pretreatment.
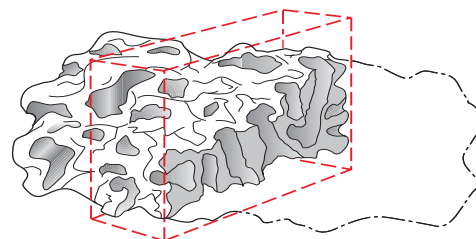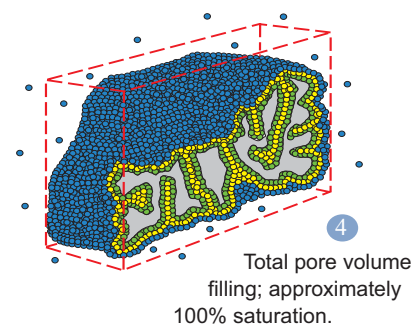
Once clean, the sample is brought to a constant temperature by means of an external bath. Then, small amounts of a gas (the adsorbate) are admitted in steps into the evacuated sample chamber. Gas molecules that stick to the surface of the solid (adsorbent) are said to be adsorbed and tend to form a thin 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 (Figure 2). Multiplying N_m by the cross-sectional area of an adsorbate molecule yields the sample's surface area.
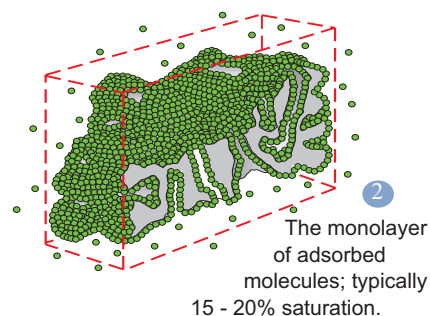
Continued addition of gas molecules beyond monolayer formation leads to the gradual stacking of multiple layers (multilayers). The formation occurs in parallel to capillary condensation (Figure 3). The latter process is approximately described by the Kelvin equation, which relates equilibrium gas pressure to the size of capillaries capable of condensing gas within them.
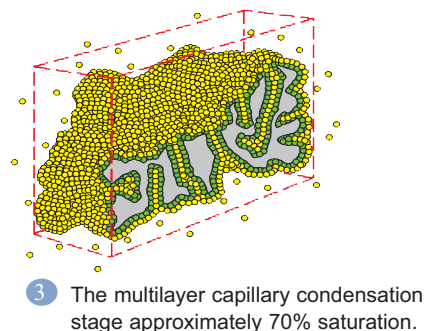
As the equilibrium gas pressure approaches saturation, the pores largely completely fill with adsorbate (Figure 4). Knowing the density of the adsorbate, one can calculate the volume it occupies and, consequently, the total pore volume of the sample. If at this stage one reverses the adsorption process by withdrawing known amounts of gas from the system in steps, desorption isotherms are generated. The resulting hysteresis leads to isotherm shapes that can be mechanistically related to those expected from particular pore-shapes.

Older calculation 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 take experimental curves (isotherms) of adsorbed gas volumes versus relative pressures and convert them to cumulative or differential pore size distributions.

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.

Modern pore size models are based on Non-local Density Functional Theory (DFT)- a statistical mechanics approach that allows one to describe the sorption of gas molecules in nanoporous materials at a molecular level. Hence, the application of such microscopic methods produces the most accurate surface area and pore size results.

Feature Highlights of the AUTOSORB®-6B

Multifunction temperature/
pressure meter displays:

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

Digital vacuum gauge

Calibration chamber

Front panel LEDs display
continuous system status
information

Six coolant level sensors
(one per station)

Six sample and P_O stations
(Three on each side)

Easy to fill 60 hour Dewars

Automated, independant
Dewar elevators

Optional rolling cart
(not shown)

RS232 port for PC control via
Windows® based software

Analysis Specifications

Transducer Accuracy: 0.11% full scale (1000 torr transducer)

A/D Converter: 22-bit (1 part in 4,190,000)

Ultimate Vacuum: 5×10^{-3} mbar achieved by dedicated 2 stage rotary, direct drive pump(included)

Adsorbates: Nitrogen and any other non-corrosive gas with appropriate coolant

Surface Area Range: 0.01 m^2/g to no known upper limit

Minimum Pore volume
(liquid): 1×10^{-6} cc/g
(STP): 5×10^{-5} cc/g

Pore size Range: 3.5 to >4000Å / 0.35 to >400nm

Coolant level: Controlled to ± 0.5 mm with RTD sensor

Minimum P/P_o: 1×10^{-3}

Physical Specifications

Dimensions: Height 40.0 inches (101.6 cm) • Width 25.5 inches (64.8 cm) • Depth 29.0 inches (73.7 cm)
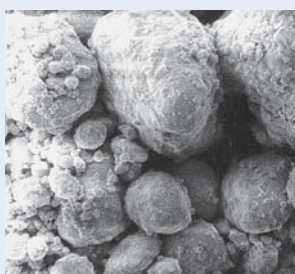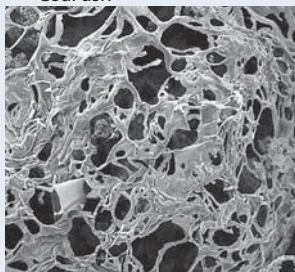
Weight: 380 Pounds (172 kg)

Electrical: 100 - 240 VAC, 50/60 Hz

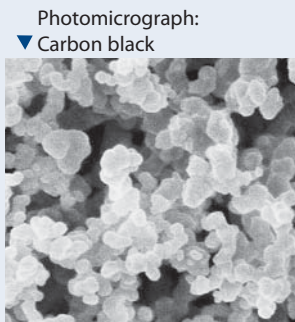**Ambient:** 10 - 38°C operating range - 90% maximum relative humidity

AUTOSORB®-6B

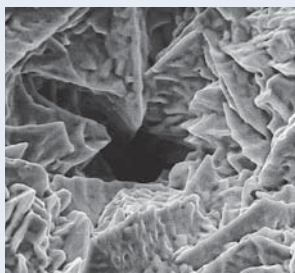## ASMultiwin Software

Photomicrograph:
▼ Coal ash

Photomicrograph:
▲ Natural zeolite

Photomicrograph:
▼ Carbon black


Photomicrograph:
▲ Prickly gold

Windows® based performance

The Autosorb-6B analyzer is microprocessor controlled, and communicates with a Windows® 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.
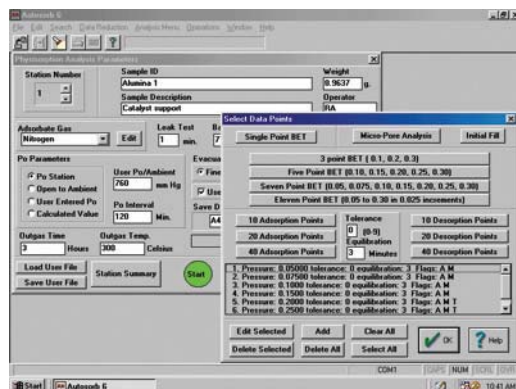
▲ Analysis parameters in Autosorb software

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 print out.
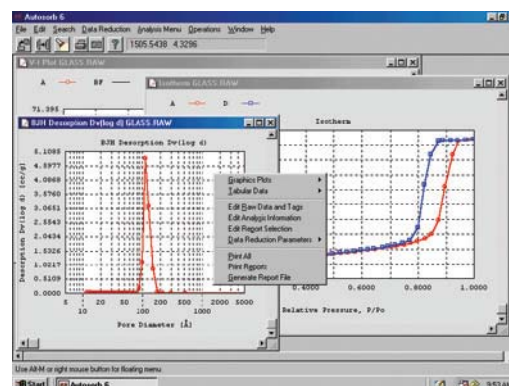
▲ Mutiple graphs to view isotherm and related plots

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 N_2, Ar and CO_2 on materials such as zeolites, MCM-41, carbons and silicas.

- Monte Carlo based pore size model.

- Fractal dimension by FHH or Neimark-Kiselev models.

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 15 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.

Autosorb Degasser

The gold-standard in sample preparation for physisorption analyzers. Individual temperature control and ramp rates for all six stations, plus optional use of heater timers. Built-in cold trap for efficient removal of condensables at low degassing temperatures and for clean vacuum. Vacuum pump included. Turbo- pumped version available for rapid high-vacuum degassing.

The FloVac™ Degasser

The FloVac provides cost effective vacuum degassing. Complete with single-zone heater (to 400°C), digital temperature controller and built-in digital vacuum gauge. Each sample station has its own adjustable evacuation/backfill rate control.Flow degassing is also possible and can be advantageous in removing large quantities of moisture prior to vacuum degassing. Individual cooling stations. Vacuum pump not included.

MasterPrep™ Degasser

Fully featured preparation unit. Individually heated stations with programmable heating profiles for R&D, commercial labs and multi-user laboratories. Dual modes (vacuum or flow) for further flexibility and optimization. Temperature control and logging via PC software (included). Integral cooling stations provide additional convenience. Vacuum pump not included.

Flow Degasser

The popular Flow Degasser has the same heating and cooling features as the FloVac but without vacuum capability. Ideal for quality control applications, teaching and start-up labs. Each sample station has its own adjustable flow rate control.

Features	Flow Degasser	FlowVac Degasser	MasterPrep	Autosorb Degasser
Number of sample ports:	6	6	6	6
Separate temperature control for each sample port:	All ports at same temperature	All ports at same temperature	Yes - with independent ovens	Yes - with independent heating mantles
Temperature ramping:	No	No	Yes - up to 20 steps, independent for each sample port	Yes - with manual adjustment, independent for each sample port
Heating timer:	No	No	Yes	Yes
Software ramp control:	No	No	Yes	No
Analog ramp control:	No	No	No	Yes
Vacuum degas mode:	N/A	Yes	Yes	Yes
Vacuum display:	N/A	Yes	Yes	Yes
Flow degas mode:	Yes	Yes	Yes	No
Maximum temperature:	400°C	400°C	425°C	350°C (450°C with optional quartz mantles)
Cold trap:	N/A	No	No	Yes
Vacuum pump:	N/A	Sold separately	Sold separately	Included (turbo pump optional)

Quantachrome Instruments' corporate headquarters in Boynton Beach, Florida.

Quantachrome®

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

For over 43 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, LabQMC.

Quantachrome Instruments Application Laboratory.

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Quantachrome Instruments' quality management system is certified to be in accordance with ISO9001:2008.

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Needs Since 1968

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