QUADRASORB™ SI

SURFACE AREA & PORE SIZE ANALYZER

QUANTACHROME

Quantachrome
INSTRUMENTS

Windows® - based performance

The **QUADRASORB™ *SI*** analyzer is microprocessor controlled, and communicates with a Windows® 2000 or XP-based PC utilizing Quantachrome's state-of-the-art, data acquisition and data reduction software, QUADRAWin™. A 21CFR Part 11 compliant software version is available for the pharmaceutical industry.

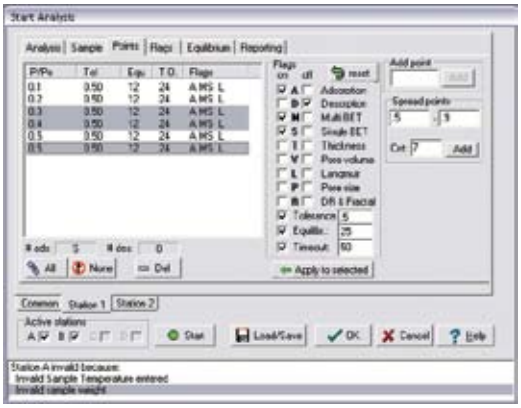
Comprehensive software to meet modern needs

The QUADRAWin™ software is highly functional and user friendly. QUADRAWin 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.

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.



▲ Analysis parameters in QUADRAWin software

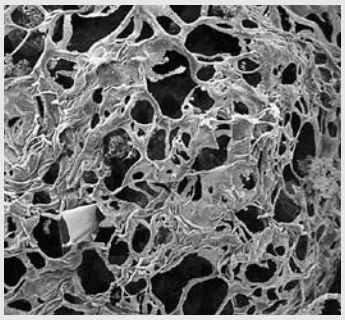
▲ Mutiple graphs to view isotherm and related plots

Data presentation

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

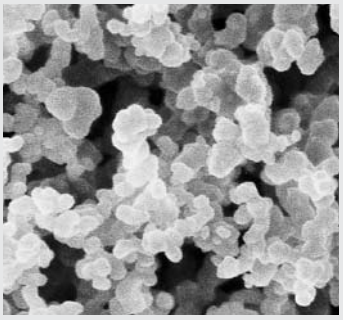
- Adsorption and desorption isotherms.
- Multi- and single point BET surface area (including C constant and correlation coefficient).
- Langmuir surface area.
- Mesopore volume and area distribution (BJH and DH methods).
- Standard micropore size distribution (MP method) and t-method by deBoer, Halsey or carbon black (STSA).
- Total pore volume, and average pore size.
- Dubinin-Radushkevich micropore surface area.
- Horvath-Kawazoe, Dubinin-Astakhov and Saito-Foley micropore distributions.
- Extensive 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 (see below).
- Monte Carlo based pore size model.
- Fractal dimension by FHH or Neimark-Kiselev models.

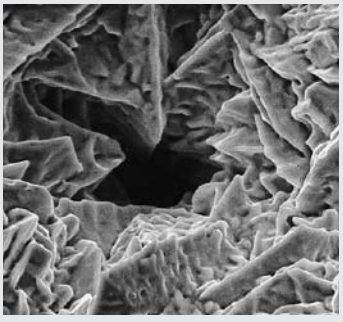
▼ Coal ash



▲ Natural zeolite

▼ Carbon black





▲ Prickly gold

NLDFT / GCMC Kernels Available in QUADRAWin Software

NLDFT / GCMC Kernel File	Applicable Pore Diameter Range
NLDFT - N_2 - Silica at 77 K Based on a cylindrical pore model. In case of sorption hysteresis pore size analysis is possible from both adsorption and desorption branches of the hysteresis loop.	0.35 nm - 100 nm
NLDFT - N_2 - Carbon at 77 K Based on a slit-pore model	0.35 nm - 30 nm
NLDFT - Ar-zeolite/Silica at 87 K Based on a cylindrical pore model. In case of sorption hysteresis pore size analysis is possible from both adsorption and desorption branches of the hysteresis loop.	0.35 nm - 100 nm
NLDFT – Ar-zeolite/Silica Based on a spherical pore model (pore diameter < 2 nm) and cylindrical pore model (pore diameter > 0.35 nm)	0.35 nm - 100 nm
NLDFT - Ar - Carbon at 77 K Based on a slit-pore model	0.35 nm - 8 nm
NLDFT - CO_2 - Carbon at 273 K Based on a slit-pore model	0.35 nm - 1.5 nm
GCMC - CO_2 - Carbon at 273 K Based on a slit-pore model	0.35 nm - 1.5 nm

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.

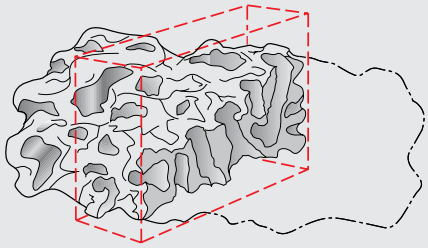
Once clean, the sample is brought to a constant temperature by means of an external bath. Then, small amounts of a gas (the adsorbate) are admitted in steps into the evacuated sample chamber. Gas molecules that stick to the surface of the solid (adsorbent) are said to be adsorbed and tend to form a thin 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.

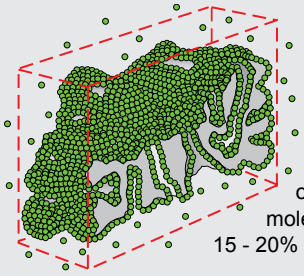
Methods such as the one by Barrett, Joyner and Halenda (B.J.H.) allow the computation of pore sizes from equilibrium gas pressures. One can therefore generate experimental curves (or isotherms) linking adsorbed gas volumes with relative saturation pressures at equilibrium, and convert them to cumulative 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.

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

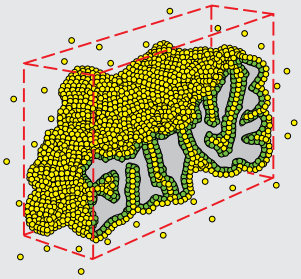


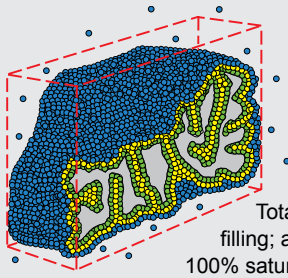
① A section of one greatly enlarged particle of a solid.



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.

Quantachrome INSTRUMENTS

QUADRAS

QUALITY CONTRO

Transparent blue cover protects sample stations and dewars during analysis operation.

Opens vertically for easy access to analysis stations.

Lower front panel opens to provide unrestricted access for Dewar placement, maintenance and clean-up.

Analysis Specifications

Transducer Accuracy:	0.11% full scale (1000 torr transc 0.15% reading (10 torr transduce
Pressure Resolution:	0.015 torr (1000 torr range) 0.00015 torr (optional 10 torr ran
Sensitivity:	<1 x 10^{-7} moles adsorbed/deso
Ultimate Vacuum:	1x10^{-3} torr achieved by dedicate or 1x10^{-9} torr achieved by oil-free t - KR/MP
Adsorbates:	Nitrogen and any other non-corro
Surface Area Range:	<0.01 m^2/g to no known upper li
Pore volume:	Detectable limit less than 0.0001
Pore Size:	Diameter range 3.5 to >4000Å /
Coolant Level:	Automatic compensation by RTD
Minimum P/P_0 (N_2):	1 x 10^{-3} QUADRASORB *SI* 4 x 10^{-5} QUADRASORB *SI* -KR/

** Pressure transducer and vacuum pump specifications from their re*

Physical Specifications

Dimensions:	Height: 29.0 inches (73.6 cm) Width: 25.1 inches (63.7 cm) Depth: 21.0 inches (53.3 cm)
Weight:	54.4 kg (120 pounds)
Electrical:	100 - 240 VAC, 50/60 Hz
Temperature:	10 - 38°C operating range at 90%

SORB™ *SI*

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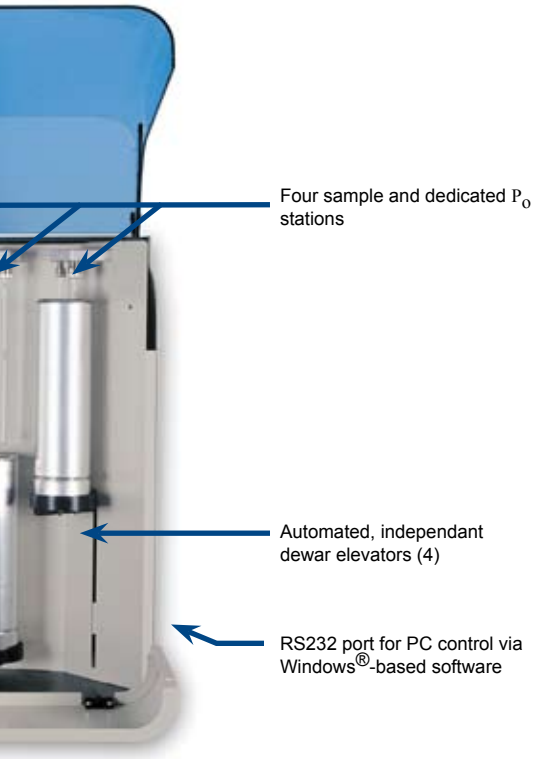

ducer) Four manifold/sample stations
er - optional) manifold

ge)

bed gas

ed 2 stage rotary, direct drive pump
turbomolecular vacuum pump in QUADRASORB *SI*

osive gas with appropriate coolant

mit

cc/g

0.35 - 400 nm

) coolant level sensor

MP

spective manufacturers.

Height Open: 44.0 inches (111.6 cm)

Depth Open: 26.2 inches (66.5 cm)

% maximum relative humidity

QUADRASORB *SI* Overview

Quantachrome's **QUADRASORB *SI*** Surface Area and Pore Size Analyzer was designed to satisfy busy laboratory needs for high analytical throughput , without sacrificing precision, flexibility or cost-effectiveness. Four simultaneous and independent (SI) analysis ports remove the limitations of single dewar systems allowing samples to be started as soon as previous measurements are completed. This measurement flexibility has never before been available in such a compact and cost-effective package.

The QUADRASORB *SI* for standard applications using a variety of-gases

- Fully automated, four-sample port analyzer for surface area, pore volume and pore size measurements.
- High resolution adsorption and desorption isotherms to detect fine pore structure detail.
- Each of the four analysis ports includes separate and independent Dewar (coolant flask),1000 torr pressure sensor and P_o (adsorbate saturated vapor pressure) measurement station for simultaneous measurements yielding maximum throughput and flexibility.
- Each of the four analysis ports includes RTD coolant level sensor to maintain constant small cold-zone for maximum sensitivity
- Each analysis port can be independently programmed with different analyses and measurement conditions.
- New samples can be started on each port as prior measurements are completed with little, or no, delay to other samples already in progress.
- Long life Dewar flasks for measurements exceeding 20 hours.
- User can choose from two measurement techniques: Patented NOVA helium-free method and classical helium void volume method.
- Multiple gas dosing methods to optimize analysis time and resolution: 1. MaxiDose™ automatically adjusts dose size in response to sample demand; 2. Constant Dose volume (0.1 - 10 cc per data point); and 3. Delta Volume which adds data points in regions of large uptake so critical pore filling is never missed.
- Low maintenance, vacuum volumetric system with temperature monitored dosing manifold.
- Windows®-based QUADRAWin™ software provides for instrument control and a comprehensive range of classical and modern models for reporting surface area and pore size.
- Designed for operation with many gases including nitrogen, argon, carbon dioxide, krypton, etc.
- Wide range of separately supplied sample preparation devices (Degassers) to meet the need of any laboratory.

Krypton/Micro pore option for low surface area and micropore measurements

- All of the functions of the standard model **QUADRASORB** SI plus low surface area measurement via krypton adsorption or low pressure micropore characterization.
- Includes low pressure (10 torr) sensor and patented oil-free turbomolecular vacuum system.
- Performs krypton gas sorption measurements for very low surface area determination, eg. pharmaceutical actives, powdered metals, etc.
- Provides low pressure adsorption data (as low as 4×10^{-5} P/P_o) necessary for more complete characterization of microporous

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 QUADRASORB *SI* Surface Area and Pore Size Analyzer.

Feature	MasterPrep™	Flow Degasser	FloVac™ Degasser	Autosorb® Degasser
Number of sample ports:	6	6	6	6
Independant 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
Heating Timer	Yes	No	No	No
Windows®-based PC programmable temperature ramping (digital):	Yes	No	No	No
Vacuum degas mode:	Yes	Not applicable	Yes	Yes - analog
Vacuum display:	Yes - digital	Yes	Yes - analog	No
Flow degas mode:	Yes	400°C	Yes	350°C; 450°C with optional quartz mantles
Maximum temperature:	425°C		400°C	
Cold Trap	No	N/A	No	Yes
Vacuum pump:	Sold separately	N/A	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.

The Autosorb Degasser

- 60 hour cold trap protects vacuum pump from vapor.

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^{0}C in steps of 1^{0}C monitored continuously on the digital display.

The FloVac Degasser set up for vacuum degassing

The 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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Needs Since 1968

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