QUANTACHROME

QUADRASORB™ *SI*

SURFACE AREA & PORE SIZE ANALYZER

Quantachrome
INSTRUMENTS

 0312 07160 Rev.E

QUADRASORB™ *SI*

Quantachrome's ***QUADRASORB SI*** Surface Area and Pore Size Analyzer is designed to satisfy busy laboratory needs for high analytical throughput , without sacrificing precision, flexibility or cost-effectiveness. Up to 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.

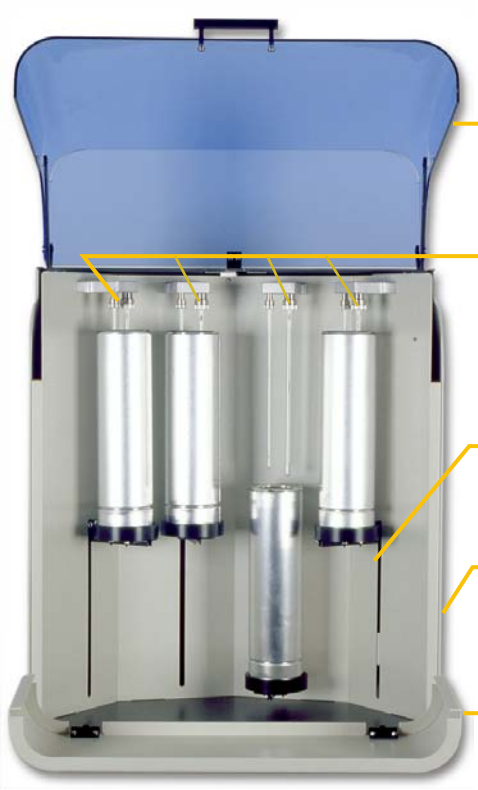
QUADRASORB™ *SI* for standard applications

- Fully automated, multi-sample port analyzer for surface area, pore volume and pore size measurements. Available with four, three or two sample stations.
- Each of the 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 analysis ports includes RTD coolant level sensor to maintain a constant, small cold-zone for maximum sensitivity. Long life Dewar flasks for measurements exceeding 30 hours.
- Each analysis port can be independently programmed with different analyses and measurement conditions and new samples can be started on each port as prior measurements are completed with little, or no, delay to other samples already in progress.
- Choose from two measurement techniques: Patented NOVA helium-free method and classical helium void-volume method. Designed for analyses using many different gases, including nitrogen, argon, carbon dioxide, krypton, etc.
- 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. High vacuum/low pressure version available (see below).
- Windows®-based QuadraWin™ software provides for instrument control and a comprehensive range of classical and modern models for reporting surface area and pore size.
- Wide range of separately supplied sample preparation devices (Degassers) to meet the need of any laboratory (See page 4).

Krypton/Micropore option for low surface area and micropore measurements

- All of the functions of the standard model QUADRASORB *SI;* Plus low pressure (1 torr) sensor and turbo-molecular vacuum pump. Available with optional oil-free roughing pump.
- Performs krypton gas sorption measurements for very low surface area determination, eg. pharmaceutical actives, powdered metals, etc. on any or all samples stations.
- Capable of low pressure adsorption data (as low as 4×10^{-5} P/P_O) necessary for more complete characterization of microporous materials, eg. zeolites, activated carbons, molecular sieves, etc.

QUADRASORB™ *SI*



Analysis Specifications

Transducer Accuracy: 0.1% span (1000 torr transducer) each manifold/sample station
0.15% reading (1 torr transducer - KR/MP model) manifold

Pressure Resolution: 0.016 torr (1000 torr range)
0.000016 torr (1 torr range)

Ultimate Vacuum: <$1x10^{-2}$ torr achieved by dedicated 2 stage rotary, direct drive pump
$1x10^{-9}$ torr achieved by turbo-molecular vacuum pump in QUADRASORB *SI* - KR/MP

Adsorbate: Nitrogen or any other non-corrosive gas with appropriate coolant

Surface Area Range: 0.01m^2/g to no known upper limit (nitrogen)
0.0005 m^2/g to no known upper limit (krypton)KR/MP-model only

Minimum Pore Volume (liquid): (STP): $2x10^{-6}$ cc/g
0.0001cc/g

Pore Size Range: 3.5 - 4000Å / 0.35 - 400 nm

Coolant Level: Automatic compensation by RTD coolant level sensor

Minimum P/P_0 (N_2): 1 x 10^{-3} QUADRASORB *SI*
4 x 10^{-5} QUADRASORB *SI* -KR/MP

* Pressure transducer and vacuum pump specifications from their respective manufacturers.

QuadraWin™ - Software

The QUADRASORB™ *SI* analyzer is microprocessor controlled, and communicates with a Windows® Vista, 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.

State-of-the-art

The *QuadraWin* software is powerful and user friendly. *QuadraWin* is superior for data reduction, incorporating the latest DFT (Density Functional Theory) models. The software guides you through analysis setup, 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 you 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 show 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.
- Langmuir surface area.
- Mesopore size distributions (BJH and DH methods).
- t-method by deBoer, Halsey, carbon black (STSA).
- Alpha-s and MP micropore methods.
- Total pore volume, and average pore size.
- Dubinin-Radushkevich (DR) micropore surface area.
- Horvath-Kawazoe,(HK) Dubinin-Astakhov(DA) and Saito-Foley (SF) micropore methods.
- Extensive Density Functional Theory (DFT) library for unified micro- and mesopore analysis using N_2, Ar and CO_2.
- Fractal dimension by Frenkel-Halsey-Hill(FHH) or Neimark-Kiselev (NK) models.

QUADRASORB™ *SI*

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 QUADRASORB *SI* 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	FloVac 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)

QUADRASORB™ SI

Physical Specifications

Dimensions: Height: 29.0 inches (73.6 cm)
Height Open: 44.0 inches (111.6 cm)
Width: 25.25 inches (63.7 cm)
Depth: 21.0 inches (53.3 cm)
Depth Open: 26.2 inches (66.5 cm)

Weight: 54.4 kg (120 lbs.)

Electrical: 100 - 240 VAC, 50/60 Hz

Environmental: 10 - 38°C operating range at 90% maximum relative humidity

Accessories

Regulator Assembly

Proper Quadrasorb functioning is assured when high-quality gas regulators are used. Quantachrome supplies complete assemblies which include two-stage regulators with dual gauges, cylinder connector, isolation valve and 1/8" gas line connector. The regulators feature stainless steel, non-venting diaphragms and the appropriate CGA fitting for specific gases. Different assemblies are available for nitrogen (and other inerts including helium), hydrogen, carbon monoxide, oxidizing gases etc.

Vacuum Pump (standard applications).

All vacuum-volumetric gas sorption analyzers require a good vacuum pump and the Quadrasorb-SI is no exception. The pump shall have the capability to pull an ultimate vacuum of 10 millitorr or below. Quantachrome can supply the correctly sized pump complete with oil, hoses and fittings. You are not required to purchase the necessary vacuum pump from Quantachrome, but if you do the entire system will have been qualified in our factory as a set, ensuring consistent performance.

Vacuum Pumps (krypton/micropore model).

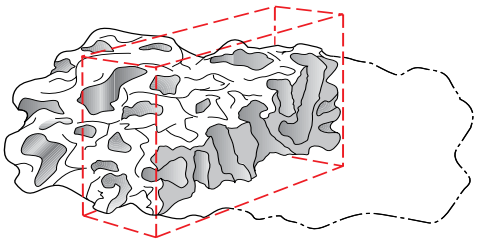
Low pressure applications require a deeper vacuum that can only be achieved by a turbo-molecular pump. The turbo pump is included, and the correct backing pump can be supplied by Quantachrome either as a rotary oil pump or as an oil-free, dry diaphragm (membrane) pump.

Storage Dewars

For the convenience of having larger quantities of liquefied gases on hand, Quantachrome offers storage dewars in sizes ranging from 5 liters to 30 liters, plus a transfer device and trolley for the largest size.

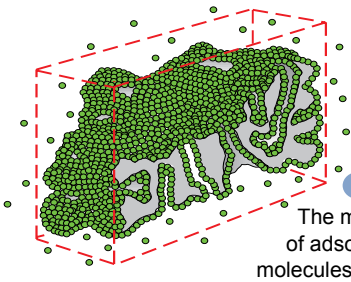
The Gas Sorption Process

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.



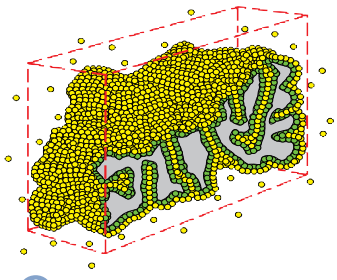
1 A section of one greatly enlarged particle of a solid.

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.



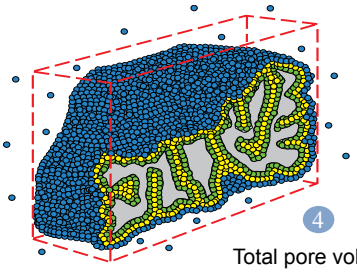
2 The monolayer of adsorbed molecules; typically 15 - 20% saturation.

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.



3 The multilayer capillary condensation stage approximately 70% saturation.

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.



4 Total pore volume filling; approximately 100% saturation.

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.

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.

Quantachrome Instruments' corporate headquarters in Boynton Beach, Florida.

Quantachrome®

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

For 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.

Quantachrome Instruments Application Laboratory.

CORPORATE HEADQUARTERS
Quantachrome Instruments

1900 Corporate Drive
Boynton Beach, FL 33426 USA
Phone: +1 (561) 731-4999
Fax: +1 (561) 732-9888
E-mail: qc.sales@quantachrome.com
www.quantachrome.com

CHINA
Quantachrome Representative Office

M806. Jingbao Garden
183 Andingmenwai Street
Beijing 100011, China
Tel/Fax: +86 10 64400892
www.quantachrome-china.com

EUROPE
Quantachrome UK Limited

Pale Lane Farm, Pale Lane
Hartley Wintney
Hook RG27 8BA, UK
Phone: +44 (0) 1252819719
Fax: +44 (0) 1252819901
www.quantachrome.co.uk

EUROPE
Quantachrome GmbH & Co. KG

Rudolf-Diesel Str. 12
85235 Odelzhausen, Germany
Phone: +49 (0) 8134/93240
Fax: +49 (0) 8134/932425
www.quantachrome.de

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