

surface area & pore size analyzer









Ceramics



Energy

Carbon



Pharma



evo=evolution

The ability to adapt to a changing environment over time. This imparts a higher survival rate compared to those species that do not evolve.



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Benefits - Speed, Accuracy & Flexibility

Quantachrome's QUADRASORB evo Surface Area and Pore Size Analyzer is designed to satisfy busy laboratory needs for high analytical throughput, without sacrificing precision, flexibility or cost-effectiveness. Four simultaneous and independent analysis ports remove the limitations of single dewar systems allowing samples to be started as soon as previous measurements are completed. A dedicated transducer has been provided for measurement of P_o (adsorbate saturated vapor pressure), so that continually measuring P_o does not slow down analysis capabilities. The new BET "QuickMode" provides high throughput industrial materials characterization labs the ability to process samples at dramatically improved speeds. This measurement flexibility has never before been available in such a compact and cost-effective package.

QUADRASORB evo

Features & Benefits

> BET QuickMode - For industrial high throughput settings - Time savings up to 50%.

• By passes most initialization, extended evacuation, and transducer zeroing sequences.

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• Samples can run synchronously.

Capable of running four (4) samples in as little as 25 minutes.*

- > Dedicated Po transducer and manifold provides uninterrupted dosing of samples during measurements.
 - Po can be continually measured.
 - No need to stop measurement to update Po.
 - No slow down in analysis to measure Po.
- > Flexible P_o Modes:
 - Po can be estimated from ambient atmospheric pressure.
 - Po can be entered as fixed value.
 - Po can be analytically measured.
- > Hardware Features:
 - Soft closing protective door with pneumatic hinges for operator safety.
 - Integrated, insulated dewar covers incorporated in lift mechanism to extend the life and insulating the dewar content.
 - Smooth belt drive dewar lift for precise control of Quantachrome's novel small cold zone.
 - 1 torr, low pressure transducer for krypton and micropore measurements.
 - Stainless steel gas paths including metal to metal seals for optimal vacuum performance.
- > New Dose Wizard Flexibility:
- Can use a previously measured sample as a dosing template for reduced analysis time.
- Can be used with or without QuickMode.

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The Newest Evolution in Gas Sorption Analysis

- Fully automated, four sample port analyzer.
- Each of the analysis ports includes separate and independent Dewar (coolant flask), 1000 torr pressure sensor and P_O (adsorbate saturated vapor pressure) cell.
- Each of the analysis ports includes coolant level sensor to maintain a constant, small cold-zone for maximum sensitivity.
- Communication port for PC control via Windows®-based software



- With soft close hinges.
- Up to four independent sample stations; each can accommodate a variety of cell sizes.
- Gravity Rings- extends dewar life by form fitting closure (up to 30 hours).
- Automated, independent dewar elevators.

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



Proven Features:

- 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 delay to other samples already in progress.
- Choose from two measurement techniques:
 - Patented NOVA helium-free method
 - Classical helium void-volume method.
- > Multiple gas dosing methods to optimize analysis time and resolution:
 - Standard- intelligently adjusts dose size in response to sample demand.
 - Vector Dosing- user selectable dose volumes from 0.25 to 10cc per point
 - Delta (volume) Max adds data points in regions of large uptake so critical pore filling is never missed.
 - Dose Wizard- can be used with or without QuickMode enabled. This allows the instrument to dose more aggressively, saving significant analysis time.
- > Low maintenance, vacuum-volumetric system with temperature monitored dosing manifold.

Krypton / Micropore Option for Low Surface Area and Micropore Measurements

- Includes 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, e.g. pharmaceutical actives, powdered metals, etc. on any or all sample stations.
- Capable of low pressure adsorption data (as low as 4 x 10⁻⁵ P/P₀) necessary for more complete characterization of microporous materials, e.g. zeolites, activated carbons, molecular sieves.



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Specifications / Accessories

Physical Specifications

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: 57.5 kg (127 lbs.)*

Electrical: 100 - 240 VAC, 50/60 Hz

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

* Four (4) station standard model not Kr/MP model.

Analysis Specifications

Pressure Transducers: Six 1000 torr transducers (one on each sample station, plus dosing manifold and P_O manifold) plus one[#] 1 torr transducer (dosing manifold)

Transducer Accuracy: 1000 torr: 0.11% full scale 1 torr: 0.15% reading

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

Ultimate Vacuum: <1x10⁻² torr achieved by dedicated two

(2) stage rotary, direct drive pump

1x10⁻⁹ torr achieved by turbomolecular vacuum pump in

QUADRASORB **evo**- Kr/MP

Adsorbate: Nitrogen or any other non-corrosive gas with appropriate coolant Surface Area Range: 0.01m²/g to no known upper limit (nitrogen)

0.0005 m²/g to no known upper limit (krypton) Kr/MP model only

Minimum Pore Volume: (liquid): 2x10⁻⁶ cc/g (STP): 0.0001cc/g

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

Minimum P/P_0 (N₂):

 1×10^{-3} QUADRASORB **evo** 4×10^{-5} QUADRASORB **evo**- Kr/MP

Coolant Level:

Automatically maintained at a level around the sample cell to minimize cold zone volume.

Kr/MP version only.

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Accelerated Analysis Times for Busy Labs

Is the QUADRASORB **CVO** "evolutionary" or "revolutionary"? You decide! Technical improvements have made it possible to conduct surface area analyse up to 50% faster than previously!



Designed to benefit customers in an industrial setting that require high throughput BET surface area measurements. This is ideal for customers doing initial screening of candidate materials as part of a material discovery group. Our QuickMode will provide scientifically accurate surface area data.

QuickMode Features:

- Shorter initialization cycle.
- All samples start at one time, running synchronously.
- When a sample finishes it is not immediately evacuated, but waits for the remaining samples in a batch to complete their runs unimpeded.
- Available in both classical helium void volume, helium free Nova mode, and with all of Po modes.

Dose Wizard

This unique proprietary method can yield even more time savings by using a previously measured isotherm as a dosing template. This provides the information of how much gas the sample is likely to adsorb at relative pressures utilizing the information from a previous analysis. In this mode, the unit does not have to learn and adjust how much gas to add in order to achieve a data point at each relative pressure. This results in significant time savings. The Dose Wizard can be used with or without QuickMode enabled. This allows the instrument to dose more aggressively, saving significant analysis time.



Software & Firmware Improvements for Speed

A new proprietary dosing algorithm, implemented in the QUADRASORB **evo** firmware increases speed and accuracy for reaching target pressure in the mesopore range. For repeat analysis a feature has been added in the software to allow the option of entering previous measurements of a cold and warm zone. This significantly saves time as well. The dosing software has optimized the calculation of the rate of adsorption required in each dose. This reduces the number of doses required, and also speeds up analysis times.



Compelling Impacts on Speed & Analysis Time

All of the various improvements made can speed up analysis times from 30% to as high as 50% faster than the predecessor Quadrasorb *Sl.* The geatest increases will be when using the QuickMode for standard BET analysis using the NOVA mode.

Up to 50% Faster Analysis Time*

*Than Quadrasorb SI using mesopore analysis in turbo mode.

Four Multi-Point BET -Measurements in 25 Minutes!*

*Multi-point BET



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Gas Sorption Overview

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 (multi layers). 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.

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.



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



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



Figure 3 : The multilayer capillary condensation stage approximately 70% saturation.



Figure 4: Total pore volume filling; approximately 100% saturation.

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Proprietary Technology for Enhanced Sensitivity



The transparent blue door encloses the physisorption station for additional safety and improved temperature control.

As the coolant evaporates, the level sensor signals the dewar drive to compensate for the change in level, thereby maintaining a constant and *small* cold



Small Cold Zone For Increased Sensitivity

The sensitivity of a manometric sorption analyzer depends on the internal volume of the measurement zone (free space) and how many adsorbate molecules remain unadsorbed. The goal always therefore is to minimize the amount of unadsorbed gas occupying the free space. For example, filler rods that occupy the stem portion of the sample cell are commonly employed. Additionally the bulbous portion of the sample cell can be selected to minimize the free space, consistent with the bulk volume of the sample and its adsorption capacity. Measurements at lower absolute pressures can also be very effective, and this is why krypton (whose saturation pressure at liquid nitrogen temperature is approximately 1/300th that of nitrogen at the same temperature) is often employed when measuring extremely low surface areas (less than one square meter total for example). The amount of unadsorbed gas is also a function of the temperature of the free space: the higher it is the fewer molecules it contains for a given pressure, and the lower it is the more molecules are present at the same pressure. In any manometric instrument part of the free space is "warm" (not in coolant), and part is "cold" (submerged in coolant). Therefore it is advantageous to minimize the volume of free space that is cold since every cm³ at liquid nitrogen temperature (77.4K) contains almost four times as many unadsorbed molecules as every cm³ does around room temperature (e.g. 298K). It is true that for any cell geometry more of it should be warm and less should be cold for maximum sensitivity.



Quadrasorb

other



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QuadraWin Data Reporting Capabilities

NLDFT-Methods

- N₂ at 77K on carbon (slit pore, equilibrium model)
- N₂ at 77K on carbon (cylindrical pore, equilibrium model)
- N₂ at 77K on carbon (slit & cylindrical pores, equilibrium model)
- Ar at 77K on carbon (slit pore, equilibrium model)
- Ar at 87K on carbon (slit pore, equilibrium model)
- Ar at 87K on carbon (cylindrical pore, equilibrium model)
- CO₂ at 273K on carbon (slit pore, equilibrium model)
- N₂ at 77K on silica (cylindrical pore, equilibrium model)
- N₂ at 77K on silica (cylindrical pore, adsorption branch model)
- N₂ at 77K on silica (cylindrical and spherical pores, adsorption branch model)
- Ar at 87K on zeolites/silica (spherical/cylindrical pore equilibrium model)
- Ar at 87K on zeolites/silica (spherical/cylindrical pore adsorption branch model)
- Ar at 87K on zeolites/silica (cylindrical pore, equilibrium model)
- Ar at 87K on zeolites/silica (cylindrical pore, adsorption branch model)

GCMC-Methods

• CO₂- carbon at 273K based on a slit-pore model

Fractal Dimension

• Neimark-Kiselev (NK), Frenkel-Halsey-Hill (FHH)

QSDFT

- QSDFT N₂ carbon equilibrium transition kernel at 77 K based on a slit-pore model
- QSDFT Ar carbon equilibrium transition kernel at 87 K based on a slit-pore model
- QSDFT N₂ carbon adsorption branch kernel at 77 K based on a cylindrical pore model
- QSDFT N₂ carbon adsorption branch kernel at 77 K based on a cylindrical pore model
- QSDFT N₂ carbon equilibrium transition kernel at 77 K based on a slitpore model
- QSDFT, N₂, carbon equilibrium transition kernel at 77 K based on a slit-pore model (pore diameter < 2nm) and a cylindrical pore model (pore diameter > 2 nm)
- QSDFT, N₂, carbon adsorption branch kernel at 77 K based on a slitpore model (pore diameter
 2 nm) and cylindrical pore model (pore diameter
 2 nm)
- QSDFT, N₂, carbon adsorption branch kernel at 77 K based on a cylindrical pore model (pore diameter < 5 nm) and spherical pore model (pore diameter > 5 nm)
- QSDFT, N₂, carbon adsorption branch kernel at 77 K based on a slitpore model (pore diameter
 <2 nm) and a cylindrical pore model (pore diameter 2-5 nm) and a spherical pore model (pore diameter > 5 nm)

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Software

The QUADRASORB **evo** analyzer is micro-processor controlled, and communicates with a Windows Vista, 2000, XP or Window 7 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.

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 N₂, Ar and CO₂.
- > Fractal dimension by Frenkel-Halsey-Hill (FHH) or Neimark-Kiselev (NK) models.



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21 CFR Part 11 Software Features

Includes many features that support these regulations and provide the necessary tools for customer compliance.

QuadraWin-CFR Software

Functions Relating to System Access, Electronic Signatures and Security.

Required login with unique user i.d. / full name combination.

- > Password aging and forced change.
- Automatic user account expiration and / or manual suspension.
- > Selectable minimum i.d. and password length.
- > Three user levels gives three privilege levels.
- Access level programmable by administrator level.
- Programmable session time-out (auto log-off through inactivity).
- Tamper resistant binary-encoded data files.
- Data security is established through the closed QUADRASORB / QuadraWin system.
- Data reduction parameters (metadata) used to calculate final results are included as part of the data file.
- "Operator" user level does not have the access privileges to change meta-data.
- Changes to meta-data are reflected in the audit trail.
- Data files acquired by means other than directly from the QUADRASORB instrument are flagged as such in the audit trail.

21 CFR Part 11

The QUADRASORB **evo**, when configured for security and used with its 21 CFR Part 11 version of QuadraWin software, is designed to allow the user to meet the regulatory requirements for electronic records within the pharmaceutical and allied industries as set forth by the US Food and Drug Administration (FDA). The FDA intends to enforce Part 11* compliance under FDA Regulations, the Federal Food, Drug, and Cosmetic Act and the Public Health Service Act as outlined in its 2003 Guidance for Industry "Part 11, Electronic Records; Electronic Signatures - Scope and Application" prepared by the Office of Compliance in the Center for Drug Evaluation and Research (CDER). This version of the QUADRASORB **evo** software adopts software design features to allow for easy integration into pharmaceutical and other GLP laboratories.

* Final Rule, Federal Register / Vol. 62, No. 54, pp13429-13466, 1997



►CFR Software- Audit Trail.

Audit Trail Functions & Reporting Features

The audit trail does not obscure previous entries; old and new values are both recorded and visible. The audit trail itself cannot be edited and is included as part of the securely encoded data file it cannot become disconnected. Changes to meta-data require that the user enter a reason, to be in accordance with the underlying predicate record rules, which is then retained as part of the audit trail. The audit trail is included in all human readable formats, screen display, print preview, PDF and hard copy formats. Multiple page report sets are linked by a unique report i.d. generated automatically by QuadraWin.

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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 metal, 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 volmetric gas sorption analyzers require a good vacuum pump and the QUADRASORB evo 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.

QUADRASORB <mark>evo</mark>



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.



You are responsible for purchasing an instrument to meet your lab's needs of today. Do you know what your lab's needs will be five years from now?

Only the **evo** provides you the ability to meet your needs of today with the assurance that you can adapt and evolve to meet your needs of tomorrow without having to purchase an entirely new analyzer.



Renowned innovator for today's porous materials community. The quality of Quantachrome's after sales service support is the reason we are proud to maintain life time relationships with our customers.

Field Service

Our global service staff assure you that Quantachrome Instruments will continue to be the reliable engines of material characterization laboratories. We offer you the flexibility of choosing from service contracts tailored to provide you with the response time, service package, and spare parts discounts that best fit your needs.

Spare Parts

Quantachrome spare parts are certified to work with our instruments. We provide rapid response spare parts orders, and keep large inventories of replacement parts and hardware available.

Application Lab

Our fully equipped, state-of-the-art powder characterization laboratory (email: application.qt@anton-paar.com), provides the option of contracting for expert testing services. Laboratory services are also available to validate the applicability of our products prior to your purchase using your actual samples.

Lifetime Application Support

We view the field support of our instruments as an essential component of our business strategy. Our expert scientists are always available to answer questions on applications, or the use of our instruments. We do this as a standard service regardless of whether you have a service contract with us or not.

Partners in Science

Quantachrome has a scientific research department consisting of world renowned experts in material characterization. Our staff, led by team conducts collaborative research projects with leading material research labs around the world. They regularly publish articles in leading peer reviewed journals, and speak at technical symposiums around the world. For almost half a century 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. We have an unwavering commitment to providing state of the art technology, along with superior and unparalleled customer service and support.

Our commitment to customers is to support you before, during, and after the sale throughout the lifetime of our instruments. This is a big commitment because our products are so robust and reliable that we regularly find many still in use for decades.

Corporate Headquarters-USA Quantachrome Instruments a brand of Anton-Paar 1900 Corporate Drive Boynton Beach, FL 33426

www.quantachrome.com

Serving Porous Materials and Powder Characterization Needs Since 1968



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