

autosorb[®] O Series

automated gas sorption analyzer surface area, pore size, and chemisorption







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Instrument appearance and colors subject to change.



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Superior Performance For Today, Combined With Future Flexibility

The Autosorb-iQ has been designed to be the most flexible, versatile, and customizable gas sorption analyzer on the market. You can purchase the requirements for your laboratory's needs today, but have the confidence that the instrument has the ability to expand to meet your laboratory's future analysis needs, without having to purchase an entirely new instrument.

Operator Convenience

All instrument operations are accessed through the Windows[®]-based software program. Fully customizable analysis parameters can be set up ahead of time and recalled as often as one likes. So too can degassing protocols. Reconfiguring the instrument, between physisorption and chemisorption modes, takes mere seconds. The unit is sized to comfortably fit on a standard laboratory bench with all electrical and gas connections to the side, rather than to the rear, for easier access. An optional rolling cart is available if greater mobility is desired or bench space is limited.

Oil Free Vacuum System*

High vacuum with no oil contamination or back streaming * MP/C-MP/XR/C-XR models

Analysis Flexibility

No two applications are the same and so a variety of physisorption measurement types are included to optimize data point spacing in the measured isotherm according to pore size and pore volume. And since some users prefer the classical helium void volume method and others helium-free analyses, both techniques are at the disposal of the operator. Advanced analytical techniques that require scanning the isotherm hysteresis loop, or pore size distribution of thin films using krypton gas, or the largest available library of density functional theory (DFT) calculations, are accommodated as standard features.



Oil free diaphragm pump Turbo Molecular

Technical Excellence

Electronic pressure transducers form the core of the instrument; among them, ceramic diaphragm capacitance types offer superior stability at lower pressures. The high vacuum system of the MP and XR micropore/ chemisorption models is mounted internally and features a 90,000 rpm turbo-molecular pump backed by a dry diaphragm pump for oil free analysis. A constant, small cold zone (physisorption mode) is achieved by using the proven coolant level sensing system. Interruptions to the analysis are minimized by virtue of a dedicated P₀ (saturation pressure) transducer on the Po cell. Metal-to-metal seals for critical fittings in the measurement zones ensure the best possible vacuum performance. The XR model's 0.1 torr transducer extends physisorption measurement capability into the 10^{-8} P/Po range (N₂ / 77K).

Customizable for Higher Throughput

By offering 2- and 3-station versions, the Autosorb-iQ represents a huge improvement in sample throughput. This is most evident when properly analyzing microporous materials in detail. These notoriously slow measurements (because of restricted diffusion of gases at the very low pressures required for analysis) have, in the past, been the cause of many laboratory bottle necks. Simultaneous analysis of up to three samples, each using their own dedicated sets of pressure transducers, effectively triples throughput and capacity without sacrificing the quality of the results. For busy industrial labs doing high throughput surface area and mesopore analyses, the economic benefits of the increased capacity are dramatic. Even the chemisorption models are available with up to three physisorption stations (not C-AG-ND).

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Intelligent By Design

- Temperature-monitored dosing manifold chamber houses precision pressure transducers
- Dedicated Po* transducer
- 0.1 torr transducer(s) (XR models)
- Sample and Po* stations
- Optional 2nd/3rd* physisorption stations
- Cold trap (chemisorption models with TCD)
- Coolant level
 controller
- *Physisorption Dewar and elevator (*90 + hrs with LN₂)
- High temperature (1100 °C) furnace (chemisorption models)
- Forced air furnace cooling (chemisorption models)



- Optional built-in and thermostatted vapor generator
- Multiple gas inputs for adsorbates, helium (void volume), and *backfill
- 24 bit A/D signal processing
- Four sample degassing stations* featuring PC-controlled ramp, hold and test protocols
 - Remote control via Ethernet communications
- 64 Bit compatible software

Instrument appearance and colors subject to change.

*Not C-AG-ND model

Highly Precise Sample Preparation

Quality data come from properly prepared samples. Four built-in degassing stations (with optionally independent turbo pump capabilities) offer the user flexible programming of automatic ramp, hold and test protocols, including a pressure rise limit method to minimize elutriation and steaming damage to susceptible samples. Chemisorption mode offers completely hands-free operation from in-situ preparation including gas switching, furnace control (heating and cooling), flow rate control (optionally using a mass flow controller) to isotherm acquisition without operator intervention.

Advanced Flow Methods

While the Autosorb-iQ is a state-of-the-art manometric (also called vacuum volumetric) gas sorption analyzer, the chemisorption models are also available with optional flow-based methods of analysis, such as temperature programmed desorption (TPD), oxidation (TPO), reduction and reaction (TPR), along with pulse titration (automatic loop injection) using a built-in TCD. A close-coupled mass spectrometer (no separate vacuum required) is also offered for detailed catalyst characterization requiring the identification of gaseous species evolving from the samples.





Modular Design To Meet Your Precise Need

The Autosorb iQ has the most versatile and modular performance available on the market. You can choose a tailored instrument designed to meet your specific application needs today. You have the confidence that as your needs change, the Autosorb iQ is designed on a revolutionary modular platform that allows its capabilities to be upgraded with your lab's future needs. You can satisfy your performance requirements of today, but have the flexibility to adapt to future increases in either throughput or desired capabilities.



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Autosorb® iQ-MP / iQ-XR

The gold standard in micropore analyzers. Patented, dry highvacuum system, with ultra-stable transducers covering multiple pressure ranges that seamlessly overlap. Available with up to three stations for enhanced analysis throughput. Turbo level degassing perfectly complements low starting pressures required for true micropore analysis. The iQ-MP model also enables analyses of extremely low surface area samples and porous thin films using krypton gas at low pressures. The iQ-XR models expand these capabilities further by substituting 0.1 torr transducers for ultra-low pressure (10⁻⁸ P/P₀) physisorption measurements.

Second and third stations are available in matching MP or XR configurations, or the simpler AG configuration.

Applications

Highly detailed, precise and accurate measurements to characterize the structure and improve the properties and performance of microporous materials such as zeolites, activated carbons, novel carbon structures (nanotubes, bulk graphenes), MOFs, carbon- and silica-based materials with hierarchical micro-mesopore structure (e.g., CMKs), molecular sieves, new materials for gas storage, improved fuel cells and batteries, membranes, nanofilters, etc., with Kr capability being ideally suited for APIs, thin films, nanosized ceramic and metal powders, and the like.

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Tailored Performance For Your Application

Autosorb[®] iQ-C-MP / C-XR

Ideal for breakthrough and pioneering research, where maximum analytical capability is essential, and bench space is at a premium. This is the only combined chemisorption physisorption manometric analyzer that features four built-in degassing stations and up to three physisorption stations for enhanced analysis throughput. The optional TCD gives this model unparalleled capabilities in the field. Indeed, this model is unrivaled in catalyst characterization capability: One moment it's a rapid surface area and pore size analyzer, the next it is measuring active metal area and dispersion, and even temperature programmed desorption (TPD), oxidation (TPO), reduction and / or reaction (TPR) analyses.

Second and third physisorption stations are available in matching MP or XR configurations, or the simpler AG configuration.

Applications

Ideally suited for the most comprehensive physiochemical characterization of the surface and porous structure of discrete solid materials. In particular, this fully integrated analyzer is extensively used in the fields of heterogeneous catalyst and catalyst support R&D and for high throughput and innovative analyses of novel battery and solar energy materials, chemicals and petrochemicals, gas storage, purification and sequestration, semiconductor and membrane development, and general industrial and academic research programs for which the most powerful yet simple instrumentation can simultaneously solve problems and uncover new opportunities for porous solid materials.

Autosorb® iQ-C-AG/C-AG-ND

Chemisorption and physisorption capable instrument providing versatility and flexibility of analysis. The iQ-C-AG series includes chemisorption hardware and software capabilities, such as a programmable high temperature (1100°C) furnace to enable more demanding sample treatments and analyses with more reactive gases (hydrogen, carbon monoxide, ammonia) and, optionally, condensible vapors.

Without on-board degassing capabilities the C-AG-ND (available in single station version only) provides a cost-effective alternative for users focused specifically on chemisorption applications.

Applications

The chemisorption capabilities expand the range of applications of the iQ-AG units to provide more detailed and key performance information about the activity, selectivity, stability, and regeneration of conventional heterogeneous catalysts and catalyst supports. This enables users to investigate ways to tailor the structure and properties, and thus optimize the performance and cost, of materials including transition and precious metal catalysts exhibiting strong, weak, dissociative and spillover adsorption modes on specific active surface sites, including acidic and basic sites on metal oxides.



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Measurement Capabilities & Applications

Gas Sorption

Physisorption, or physical adsorption, is the process by which gas and vapor atoms and molecules are adsorbed onto the surface of a solid and which relies on weak attractive forces, usually measured at cryogenic temperatures. It is also the experimental means by which one can quantify the surface area of a solid, its pore size and pore volume distribution. Chemisorption, or chemical adsorption, is the process of gas sorption to specific sites through the formation of chemical bonds. It is the experimental means by which one measures active metal area, and related properties, of catalysts, usually at much higher temperatures than physisorption measurements.



Measurement Sensitivity

The sensitivity of a physisorption analysis using the manometric technique, relying on pressure changes in the sample cell to define the sorbed amounts, is higher when the volume around the sample, the so called free-space or void volume, is as small as is practicably possible, most importantly the cold free space/void volume, since volume for volume it contains more (unadsorbed) gas molecules than the equivalent warm space. That is ensured by accurately controlling the level of cryogenic coolant around the sample cell so as to immerse as little as is necessary for analysis.



The highest quality chemisorption measurements are no less demanding, and are equivalently achieved by intelligent hardware integration.

State-of-the-Art

Building on the reputation of the Autosorb[®] name, the iQ represents a major leap forward in gas sorption measurement technology, providing materials science researchers with a highly sophisticated platform to confidently tackle any pore size, surface area or catalyst characterization problem encountered in QC and R&D applications alike.

For BET Surface Area: Physisorption

The Autosorb iQ can determine total BET surface area with remarkable sensitivity. Multi-point or single point measurements can be performed. Surface area of 0.01 m²/g and upwards can be accurately determined using nitrogen (at liquid nitrogen temperature) or argon (at liquid argon temperature) in MP & XR models. Using krypton at liquid nitrogen temperature, surface areas down to 0.0005 m²/g can be accurately measured due to the added sensitivity. The ASiQwin software automatically displays the BET plot and computes the BET "C" constant, yintercept, slope, and correlation coefficient of the least-squares best fit. In addition, the Micropore BET Assistant function enables the user with the click of a button to determine the correct linear BET range for a microporous material based on published criteria.

Chemisorption

Some surfaces, especially catalysts, are sufficiently reactive to form chemical bonds with certain gases. In contrast to physisorption, chemical adsorption (chemisorption) involves the formation of strong bonds between adsorbate molecules and specific surface locations known as active sites.

Chemisorption is thus used primarily to evaluate quantitatively the number of surface active sites which are likely to promote (catalyze) chemical reactions. Both static adsorption isotherms and dynamic pulse titrations yield monolayer uptake, metal area, nanocluster (crystallite) size and active metal area of heterogeneous catalysts. Hydrogen and carbon monoxide are the two most commonly employed gases. Oxygen or other gases are sometimes suitable. In an analogous manner, the amount of acidic or basic sites is determined from the adsorption of a basic or acidic gas, such as ammonia or carbon dioxide, respectively. Isothermal results can be used to map surface energetic heterogeneity via heat of adsorption calculations.

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iQ = Intelligence With Enhanced Physisorption Sensitivity



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.



Autosorb-iQ

Other instruments



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Flow Chemisorption For Advanced Catalyst Characterization (Autosorb iQ-C with TCD)

TPR: Temperature Programmed Reduction



Many heterogeneous catalysts are used as the zero-valent metal, but start life as the oxide. An important factor in catalyst design and use is the ease of reduction of the metal oxide and TPR is a direct measure of that. A reducing gas mixture, say 2%-5% H₂ in N₂, flowing over the oxide

will cause reduction at some point as the temperature is raised using a linear heating ramp. The signal caused by consumption of hydrogen represents the rate of reaction and goes through a maximum at a temperature that is characteristic of both the oxide and the heating rate.

Repeating the same analysis on a fresh sample at a different heating rate is the means by which activation energy for the process can be evaluated. Low loadings of metal oxides, especially surface oxides, generate little water and a successful analysis can be done without trapping it. Larger amounts of moisture generated by the reduction of bulk oxides can be trapped prior to reaching the detector to leave a clean signal based solely on the change in hydrogen concentration.

- Industrial Catalysts (eg., Hydrocracking, Hydrodesulfurization, Hydrodenitrogenation and Fischer-Tropsch)

TPD: Temperature Programmed Desorption

Species previously adsorbed can be desorbed into a stream of pure carrier gas to generate a characteristic fingerprint. One very common application is ammonia TPD, by which one can evaluate relative acid site strength of, for example, zeolites. Basic sites can similarly be evaluated by TPD of carbon dioxide.

Some materials may be characterized by decomposition, or dissociation, of the bulk solid, not merely by desorption from the surface. Such examples include carbonates resulting from CO_2 removal studies, hydrides used as potential hydrogen storage materials, etc.

- Zeolites (eg., FCC, Isomerization)

TPO: Temperature Programmed Oxidation



Carbons and carbides are amenable to evaluation by careful oxidation while being heated. A stream of diluted oxygen (e.g. 2-10% O_2 in He) directed over the sample during a linear heating ramp generates a signal due to the loss of O_2 from the gas stream. The

products of oxidation, CO and CO_2 , need not be trapped. The specially chosen filaments used in the Autosorb iQ TCD detector are resistant to oxidation and operate normally in the suggested gas mixtures.

Different forms of carbon such as amorphous, nanotube, filament and graphitic, oxidize at different temperatures due to varying availability of reactive carbon-carbon bonds. In this way, fullerenes, soots, cokes on catalysts, etc., can be quickly characterized and differentiated. Oxidation catalysts, e.g. those incorporating chromium, cobalt, copper and manganese, and redox supports like ceria and titania can also be characterized by TPO.

- Carbons, Fuel Cells, Electrodes, etc.

Pulse Titration: Quantitative Analysis



This technique is used to determine a wide variety of reactive material properties, such as strong chemisorption uptake, active metal area, metal dispersion, and average nanocluster (crystallite) size.

After suitable in-situ preparation, which may be combined with TPR/TPO, the sample is automatically titrated* with small, known volumes (pulses) of reactive gas. The detector senses the excess gas which does not react with the sample. The total volume of gas which does react with the sample is automatically determined by simple back calculation using TPRWin[™] software.

Supported Metals (Reforming, Partial Oxidation, Hydrogenation, Automotive Exhaust, etc.)

* Requires loop injection option.

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ASiQWin Software

The Windows[®]-based comprehensive control, acquisition, calculation and reporting software communicates with the analyzer via Ethernet, directly or via a LAN.

A typical physisorption analysis starts with degassing of the sample. The desired protocol of heating rates, hold times and automatic readiness testing are loaded, and then the iQ takes over. After completing the timed protocol, or sooner if the residual pressure rise passes the user-defined test limit, the iQ shuts down the degasser, backfilling the sample cell or leaving it under vacuum if preferred.

Target Temp.	Rate (deg/min)	Soak Time (mir	n) Test?	🔁 Add
140.00 300.00	20.00 10.00	30 180	yes	🗖 Delete
Pressure C	Controlled Heating, L	imit: 10	torr	Edit
Completion Stat Backfill C Vacuum	te	ment 2 📩	50.000 mtorr/min a	at every: 15 min
Pressure (torr)	roccoulory 38.00		Backfills 775.00	

Analysis parameters can be recalled from a saved set, or freshly created, and can import the degassing details. You always have a choice of analyzing using the classical helium or, alternatively, He-free void volume methods.

Request data at targeted relative pressure points, automatically supplemented during the run by additional "maximum delta volume sorbed" points (in regions of the isotherm that show steep up-take or desorption) or let them be acquired according to a fixed volume dosing algorithm called Vector Dose. When sorption takes place in large amounts and quickly, the popular Maxidose feature minimizes total analysis time. Hysteresis in physisorption isotherms can be "scanned" - that is, multiple ad/ desorption cycles over desired P/P₀ ranges can be programmed. Comprehensive physisorption calculations include specific surface area (single and multi-point BET, Langmuir, STSA, t-plot, alpha-s, DR), total pore volume and pore size distribution (BJH, DH, DA, MP, HK, SF, Monte-Carlo, NLDFT, QSDFT) with their corresponding surface area values, Kr pore size for thin films, heats of adsorption, plus NK and FHH fractals.

The DFT library is the most inclusive available commercially, providing no less than twenty three mathematical models describing different pore shapes, chemical characteristics of the surfaces, and specific adsorbate and temperature pairs.



Choose chemisorption parameters that include insitu pretreatment, and a second (reversible) isotherm measurement, and combine multiple pretreatments and isotherm analysis conditions (different gases, different temperatures) into an extensive batch mode.

Chemisorption results include metal area, dispersion, nanocluster (crystallite) size, and monolayer capacity by a number of methods: extrapolation, Langmuir, Freundlich and Temkin. Heats of chemisorption are also available using manometric (static isotherm) data. Flow methods of analysis (with the TCD option) expand the range of measurements to include activation energy, quantitative TPD, TPO and TPR, with the ability to deconvolute overlapping TPX peaks, and/or to identify effluent gases using an optional built-in mass spectrometer.



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Flexibility & Modularity: Available Options & Accessories

The intelligent modular approach of the iQ extends to its wide range of available options and accessories. These options can be added at the time of ordering, or many can be added in the field after purchase. This provides the ultimate in modular flexibility to accommodate your future characterization needs.

Multiple Physisorption Stations:

Second and third stations are available in various pressure ranges. Each station is equipped with its own complete set of pressure transducers (no sharing).

- AG = 1000 torr
- MP = 1000, 10, 1 torr
- XR = 1000, 10, 0.1 torr

Thermal Conductivity Detector (TCD):

When equipped with a built-in TCD the Autosorb iQ Chemisorption models can perform fully automatic flow-based experiments, which include temperature programmed desorption, oxidation and reduction (TPD, TPO and TPR) and optionally pulse titration (automatic loop injection).

Mass Spectrometer:

The iQ's turbo-pumped chemisorption models can also be equipped with a close-coupled mass spectrometer (no separate vacuum required) for detailed catalyst characterization involving identification of gaseous species. The instrument's software controls data acquisition by the mass spectrometer, so only one PC is required.

Vapor Sorption Option:

The vapor generator is housed within the manifold chamber, where it is heated and thermostatically controlled. A solenoid valve opens the pump ballast for all-important venting of condensible vapors. This option automatically switches between vapor and gas feeds, includes automated soak/hold for chemisorption/TPX runs, and can be added at a later date.

Heated Manifold

Insulated and thermostatted, a heated manifold provides stable pressure response in a poorly controlled lab climate. It is included with the Vapor option.

CryoCooler:

Powerful and precise thermostat for physisorption isotherms at any temperature between 20K and 320K without liquefied gases. Fits all models and requires no liquid nitrogen or other cryogen.

CryoSync:

Cryo-cooling accessory capable of enabling IUPACrecommended argon gas analyses at 87K, and experiments between 82K and 115K, using liquid nitrogen instead of liquid argon or more involved alternatives.

Mass Flow Controller:

A popular upgrade for the C model. Programmed from the ASiQWin software it accurately controls the flow rate of any attached gas during chemi-pretreatment or TCD -based analysis.

Calorimeter Interface:

Measure heats of adsorption directly at the sample cell using this attachment to interface with a commercially available third party calorimeter.

Rolling Cart:

Sturdy and durable optional cart used when bench space is limited or when the instrument needs to be moved around in the lab. It can accommodate the CryoCooler's compressor on the bottom shelf, saving even more bench space.

21 CFR 11 Compatible Software

The Autosorb iQ software (ASiQWin) is available in a version that can support your compliance with 21CFR 11 requirements. Software features include multiple user/access levels, password expiration, incorrect password lockout, audit trail, and more.

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Specifications

Standard Features	Benefits	
Small cold zone technology	Enhanced sensitivity of sorption amounts	
Standard Cryogen Dewar (90+ hours), 3 liter	Extended operation without operator attention	
Vacuum pump(s) Included	Space-saving noise-reduction	
Built-in Degasser stations*	Space saving – no external devices needed	
Degas cold trap*	Enhanced vacuum level and efficiency	
Degas test function using Pirani gauge	Shortens degassing time without sacrificing quality	
Multiple adsorbate gas input ports (automatic switching with purge/evacuation routines)	Ease of use and gas purity: eliminates swapping gas connections in order to use a different adsorbate.	
Dedicated helium input port	Eliminates contamination by other gases	
Dedicated backfill gas input port	Ensures consistent sample weights	
Furnace with auto lid close/open (C models only)	Eliminates manual intervention and provides maximum furnace cooling rate.	
Fan-assisted furnace cooling (C models only)	No utility/service air required	
Built-in auto isolation valve for flow-cell (C models only)	Fully automatic transition between flow mode and static (manometric/volumetric) mode. Eliminates manual reconfiguring valve between chemisorption and vapor/physisorption	
Intelligent sample evacuation using multiple valves	Eliminates powder elutriation out of the sample cell	
*Not iQ-C-AG-ND		

Performance	Specification
0.1/1 torr transducer accuracy (MP/XR)	<0.15% of reading
Ultimate vacuum, analysis (MP/XR)	5x10 ⁻¹⁰ mbar
Sensitivity (XR)	< 2 x 10 ⁻⁹ moles adsorbed/desorbed gas
P/Po range (XR)	<1 x 10 ⁻⁸ – 0.999
Pore Size range	0.35 to 500 nm (3.5 to 5000 Å)
Ultimate vacuum, degas (MP/XR)	5x10 ⁻¹⁰ mbar
Chemisorption furnace, max temperature (C models only)	1100°C
Degassing temperature*	350°C (450°C with quartz glassware)
*Not iQ-C-AG-ND	



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 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.

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