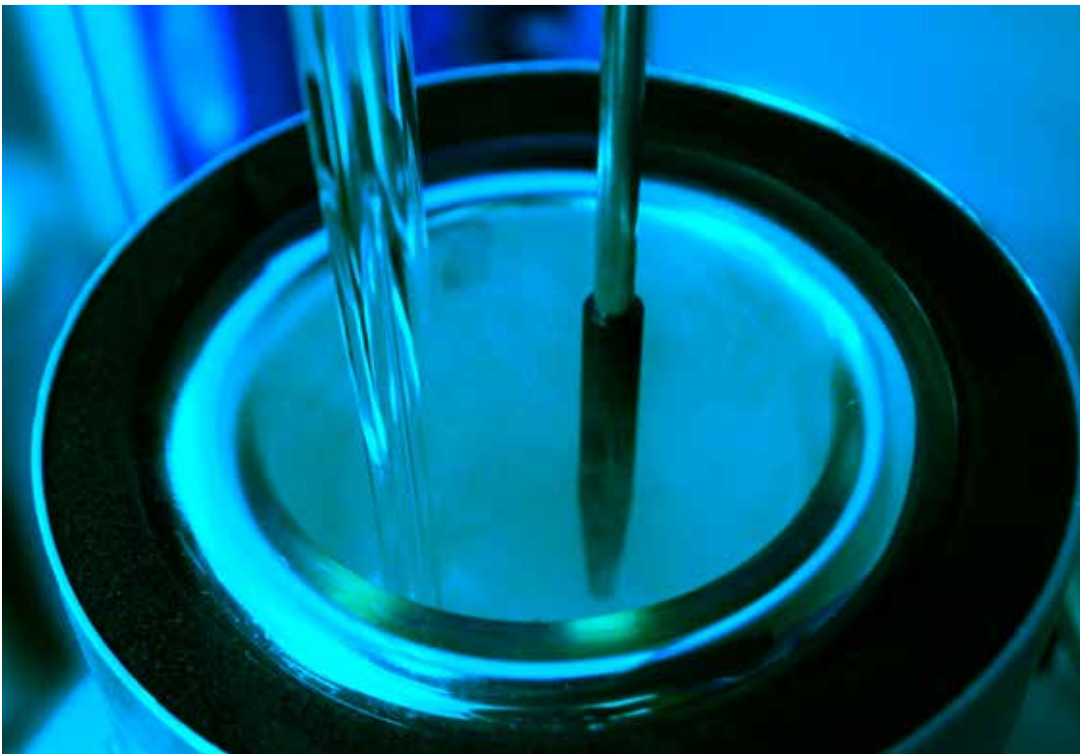




# autosorb<sup>®</sup> iQ Series

automated gas sorption analyzer



GAS SORPTION

surface area, pore size, chemisorption



Catalysts



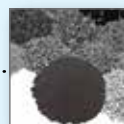
Ceramics



Energy



Carbon



Pharma



Instrument appearance and colors subject to change.

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# autosorb<sup>®</sup> iQ

automated gas sorption analyzer  
surface area, pore size, chemisorption

## Superior Performance For Today, Combined With Future Flexibility

The Autosorb-iQ has been designed to be the most flexible, versatile, and modular surface area and pore size 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<sup>®</sup>-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.

### 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 Vacuum System\*

High vacuum with no oil contamination or back streaming

\* Micropore and MP-chemisorption models



Oil free diaphragm pump    Turbo Molecular

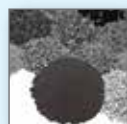
### Technical Excellence

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 micropore and 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 RTD coolant level sensing system. Interruptions to the analysis are minimized by virtue of a dedicated  $P_0$  (saturation pressure) transducer on the  $P_0$  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 ( $N_2$  / 77K).

### Modular Expansion for Higher Throughput

By offering optional 2- and 3-station capabilities, the Autosorb-iQ offers 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 bottlenecks. 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 model can be equipped with up to three physisorption stations.

GAS SORPTION



## Intelligent By Design

- Temperature-monitored dosing manifold chamber houses precision pressure transducers
- Dedicated  $P_0$  transducer
- 0.1 torr transducer(s) (XR models)
- Sample and  $P_0$  stations
- Optional 2nd/3rd physisorption stations
- Cold trap (TPX model)
- Coolant level controller
- \*Physisorption Dewar and elevator (\*90 + hrs with  $LN_2$ )
- High temperature (1100 °C) furnace (chemisorption models)
- Forced air furnace cooling (C- models)



- Optional built-in and thermostatted vapor generator
- Multiple gas inputs for adsorbates, helium (void volume), and backfill
- 24 bit A/D signal processing
- Up to 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.

## Highly Precise Sample Preparation

Quality data come from properly prepared samples. Two 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 model is 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. Also, other techniques are made available through the TPX option, such as transient kinetics, reaction isotherm generation, conversion studies, etc.



## 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.



Instrument appearance and colors subject to change.

### Autosorb® iQ-AG

Ideal for surface area and mesopore analysis, the iQ-AG is available in up to three station versions, and provides an easy upgrade path to high vacuum, low pressure, and chemisorption capabilities, should your needs change. Usable with a host of inert and reactive gases (such as nitrogen, argon, carbon dioxide, ammonia, etc.), this instrument has instant appeal for a wide range of applications. The ability to perform long analyses (90+ hours if needed) with a LN<sub>2</sub> Dewar is standard. The AG units include up to four built-in vacuum degassing ports.

#### Applications

Perfect for mesoporous materials like aluminas, silicas, oxides such as minerals, ceramics and novel M41S solids (e.g., MCM-41), PMOs, KITs, SBAs, tailored carbons, pharmaceutical excipients, etc. Highly versatile and didactic, it easily adapts to academic research and industrial applications alike.

### Autosorb® iQ-AG-C / iQ-AG-CX

Chemisorption and physisorption capable instrument providing versatility and flexibility of analysis. Available in single station version only. The iQ-AG-C variant expands standard iQ-AG features to include 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, condensable vapors. By removing on-board degassing capabilities (which can be performed using external degassers such as the XeriPrep™), the iQ-AG-CX 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.



## Tailored Performance For Your Application

### Autosorb® iQ-MP / iQ-MP-XR

The gold standard in micropore analyzers. Patented, dry high-vacuum system, with ultra-stable transducers covering multiple pressure ranges that seamlessly overlap. Available with up to three stations (2 or 3 MP/XR) 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-MP-XR model expands these capabilities further by adding 0.1 torr transducers to MP stations for ultra-low pressure ( $10^{-8}$  P/P<sub>0</sub>) physisorption measurements.

### 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.

### Autosorb® iQ-MP-C / iQ-MP-C-XR / iQ-MP-C-TPX

Ideal for breakthrough and pioneering research, where maximum analytical capability is essential, and bench space is at a premium. The iQ-MP-C is the only combined chemisorption-physisorption manometric analyzer that features four built-in degassing stations and up to three physisorption stations (2 or 3 MP/XR) for enhanced analysis throughput. Besides providing the standard MP and optional XR capabilities of the micropore units, the highly versatile and user-friendly built-in flow options in the iQ-MP-C-TPX give 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.

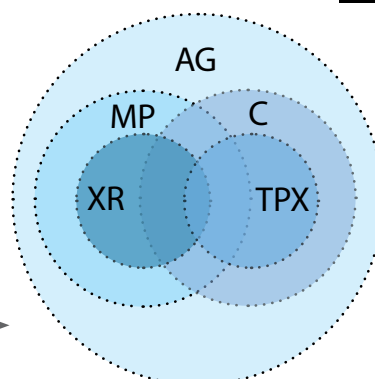
### Applications

Ideally suited for the most comprehensive physicochemical 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 petro-chemicals, 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.

### AG Series:

Within the AG series, you can increase your analytical power by adding a variety of enhancements. If you need detailed micropore analysis or chemisorption capabilities, the MP and C variants can meet your requirements. If in addition you need enhanced features to perform the most advanced analyses and pioneering research, the XR and TPX variants are sure to fulfill your most exacting surface and textural characterization requirements.

Venn diagram illustrating how the ASiQ product variants are interconnected.



## Specifications

GAS SORPTION

Standard Features	iQ-AG	iQ-AG-C	iQ-AG-CX	iQ-XR	iQ-XR-C
Sample Analysis Stations (Base Model)	1	1	1	1	1
Optional Second /Third AG Port (1000 torr)	✓	✓	✓	✓	✓
Optional Second /Third KR Port (1000, 10 torr)				✓	✓
Optional Second/Third MP Port ( 1000, 10, 1 torr)				✓	✓
Po Station w/ Dedicated Transducer	✓	✓	✓	✓	✓
BET/Mesopore Capable (P/Po >1 x 10 <sup>-3</sup> )	✓	✓	✓	✓	✓
Low Pressure Kr Capable				✓	✓
Micropore Capable (P/Po < 1 x 10 <sup>-4</sup> )				✓	✓
Chemisorption Capable		✓	✓		✓
1000 torr Transducers (Base Model)	3	3	3	3	3
10 torr Transducers (Base Model)				1	1
0.1 torr Transducers (Base Model)				1	1
Coolant Level Control Method	RTD	RTD	RTD	RTD	RTD
Standard Cryogen Dewar (90+ hours)	3 liter	3 liter	3 liter	3 liter	3 liter
Vacuum Pump Included	✓	✓	✓	✓	✓
Turbo Pump				✓	✓
Built-in Degasser Stations	4	4		4	4
Degas Cold Trap	✓	✓		✓	✓
Adsorbate Gas Input Ports	5	5	5	5	5
Dedicated Helium Input Port	✓	✓	✓	✓	✓
Dedicated Backfill Gas Input Port	✓	✓	✓	✓	✓
Total Number of Gas Input Ports (Base Model)	7	7	7	7	7
1100°C Furnace with Auto Lid Closing		✓	✓		✓
Fan-Assisted Furnace Cooling		✓	✓		✓

Field Upgrade Capabilities	iQ-AG	iQ-AG-C	iQ-AG-CX	iQ-XR	iQ-XR-C
Upgradeable to XR model	✓	✓			
Upgradeable to MP model	✓	✓			
Upgradeable to KR model	✓	✓			
Upgradeable to C model	✓			✓	
Integrated TCD & Cold Trap Option		✓	✓		✓
Integrated Mass Spectrometer (Requires factory installed option at time of purchase)					✓
Sub-Ambient Capability * (Requires factory installed option at time of purchase)		✓	✓		✓
2nd / 3rd Physisorption Analysis Station(s)	✓	✓		✓	✓
Vapor Sorption Option	✓	✓		✓	✓
Mass Flow Controller		✓	✓		✓

## Specifications

Hardware Features	Analysis Capabilities	iQ-AG	iQ-AG-C	iQ-AG-CX	iQ-XR	iQ-XR-C
Standard Base Model	Surface Area (BET, DR, etc.)	✓	✓		✓	✓
	Mesopore Size Distribution	✓	✓		✓	✓
	Total Pore Volume	✓	✓		✓	✓
	Average Pore Size	✓	✓		✓	✓
	t-plot, alpha-s plot, STSA	✓	✓		✓	✓
	Isotherm Hysteresis Scanning	✓	✓		✓	✓
	Heats of Adsorption	✓	✓		✓	✓
Add Static Chemisorption Capability	Manometric Chemisorption Isotherms		✓	✓		✓
	Strong Chemisorption (Monolayer) Uptake		✓	✓		✓
	Weak Chemisorption Uptake		✓	✓		✓
	Metal Dispersion		✓	✓		✓
	Metal Area		✓	✓		✓
	Metal Crystallite (Nanocluster) Size		✓	✓		✓
	Heats of Adsorption		✓	✓		✓
	In-situ Sample Preparation, incl. Flow		✓	✓		✓
Add Micropore Capability	Low Pressure Micropore Isotherms				✓	✓
	Micropore Size Distributions				✓	✓
	Krypton Capability for Very Low Surface Areas				✓	✓
	Low Pressure Chemisorption Isotherms					✓
Optional Features	Capability Benefits	iQ-AG	iQ-AG-C	iQ-AG-CX	iQ-XR	iQ-XR-C
2nd/3rd Physisorption Port	Doubles/Triples Throughput	✓	✓		✓	✓
2nd Vacuum System	Dedicated Vacuum Pump for Degassing	✓	✓		✓	✓
Mass Flow Controller	Mass Flow Adjustment for Different Gases		✓			✓
TCD With Cold Trap ( <b>TRX</b> )	Flow Chemisorption, Temperature Programmed Methods (TPD/TPO/TPR), Activation Energy		✓	✓		✓
Auto Titration Loop (requires TPX Option)	Automated Pulse Titration and Calibration		✓	✓		✓
Built In Mass Spectrometer	Flow Chemisorption, Ability to Characterize Components of Evolved Gases					✓
Vapor Sorption Option	Evaluate Vapor Solid Interactions (includes heated manifold option)	✓	✓		✓	✓
Additional 7 Gas Input Ports	Increases # of unique analysis gas inputs to 13	✓	✓	✓	✓	✓
CryoCooler	Allows for Analysis to be Performed at Wide Range of Temperatures	✓	✓		✓	✓
CryoSync™	Enables IUPAC-Recommended Argon Gas Analyses at Liquid Argon Temperature (87 K) and Above Using Liquid N <sub>2</sub> as the Cryogen.	✓	✓		✓	✓
Various Elastomer Seals	Allows for Chemical Compatibility with Analysis Gases and Vapors	✓	✓	✓	✓	✓
Heated Manifold (Incl with vapor option)	Thermostatted Operating Temperature in Uncontrolled Ambient Conditions	✓	✓	✓	✓	✓

\* INCL=Included as standard in that model. † Available in XR model; replaces 1 torr transducer(s).

The software program is provided with a site-wide license and can be freely installed on multiple PC's, not just the one connected to the analyzer. It is also available in a version that supports 21 CFR 11 compliance.

GAS SORPTION

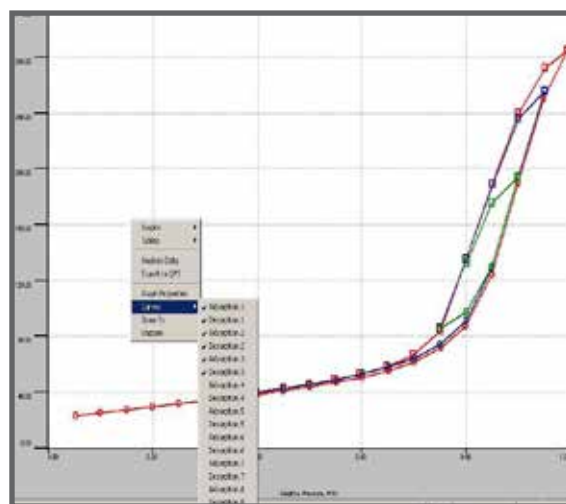
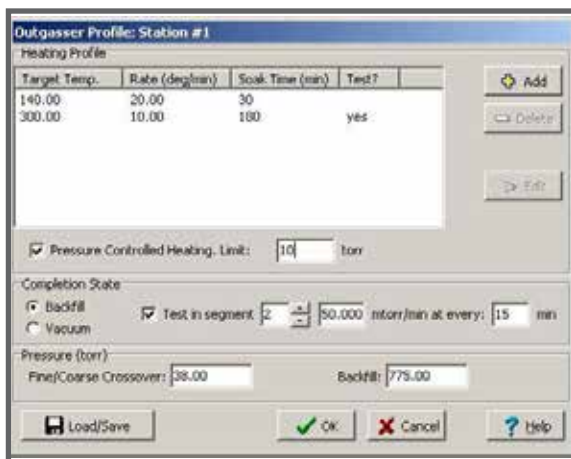


## ASiQWin Software

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

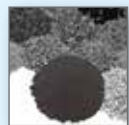
A typical physisorption analysis starts with degassing of the sample. The desired protocol of heating rates, hold times and automatic readiness testing are loaded, and then the iQ takes over. After completing the timed protocol, or sooner if the residual pressure rise passes the user-defined test limit,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

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.



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 uptake 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/P<sub>0</sub> ranges can be programmed.

Choose chemisorption parameters that include in-situ 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.





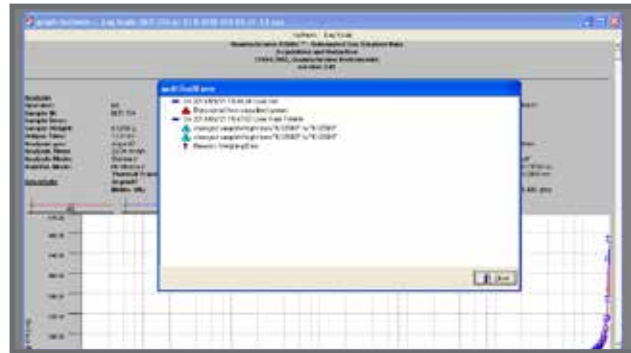
## 21 CFR Part 11 Software

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

### ASiQWin-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 Autosorb® / ASiQWin system.
- Data reduction parameters (meta-data) 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 Autosorb® instrument are flagged as such in the audit trail.



►CFRb Software- Audit Trail.

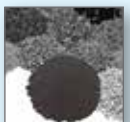
### 21 CFR Part 11

The Autosorb® iQ, when configured for security and used with its 21 CFR Part 11 version of ASiQWin 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 Autosorb® iQ 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.

### 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, and 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 ASiQWin.





**autosorb<sup>®</sup> iQ**  
 automated gas sorption analyzer  
 surface area, pore size, chemisorption

GAS SORPTION

## 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.

### TCD: **TPX**

The Autosorb-iQ TPX, a state-of-the-art manometric/volumetric gas sorption analyzer is also provided with flow chemisorption capabilities. It is equipped with Quantachrome's exclusive built-in TCD capability (TPX option), enabling the user to 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 chemisorption model 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. Can be supplied as the hardware interface alone for use with your own quadrupole mass spectrometer.

### 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 condensable vapors. This option can be added at a later date.

### 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).

### Calorimeter Interface:

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

### 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.



Universal cryo-cooling accessory to gas sorption analyzers, capable of enabling IUPAC-recommended argon gas analyses at 87K, and experiments between 82K and 112K, using liquid nitrogen instead of liquid argon or more involved alternatives.

### Mass Flow Controller:

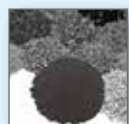
A popular upgrade for the C model. Programmed from the PC it accurately controls the flow rate of any attached gas during chemi-pretreatment or TCD - based analysis.

### 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.



Extended Life Dewar . . .



## iQ = Intelligence With Enhanced Sensitivity

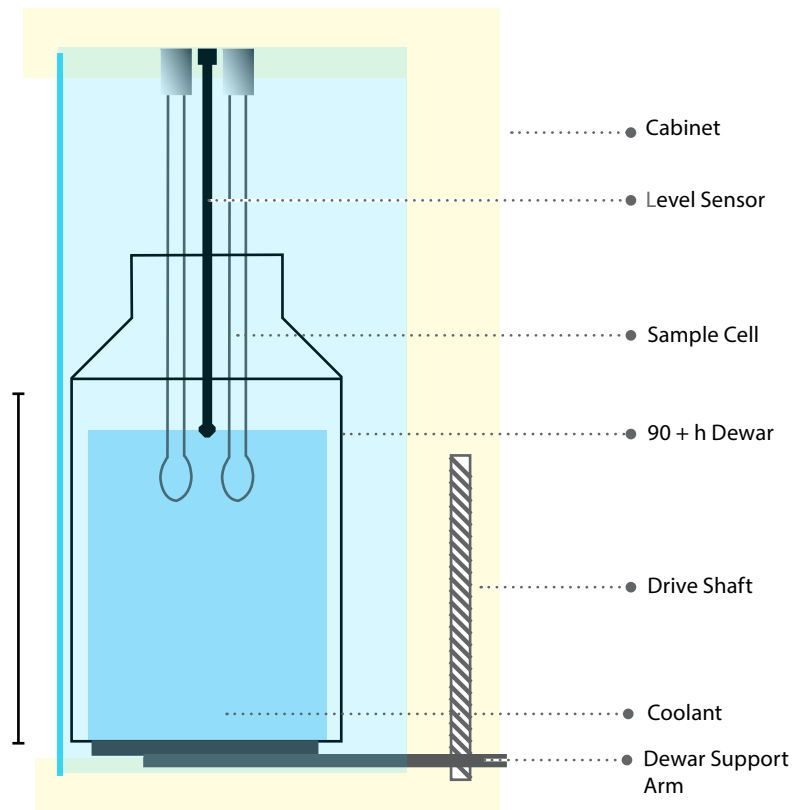
The transparent blue door encloses the physisorption station for additional safety and improved temperature control.

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 zone.

### Longer- Life Dewar

The iQ features a new 3 liter Dewar as standard.

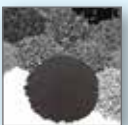
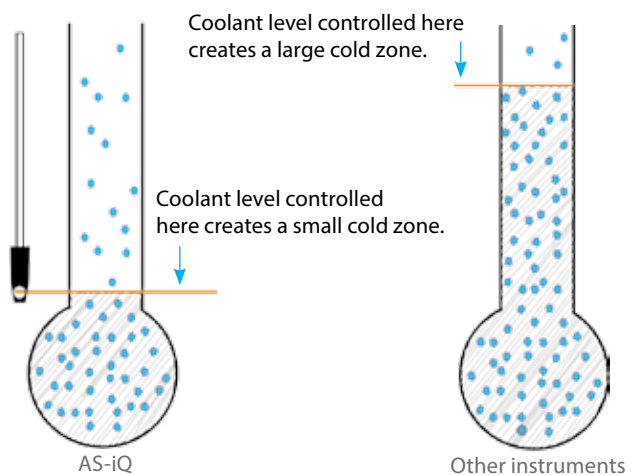
The LN<sub>2</sub> life of the Dewar is 90+ hours.



## 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.

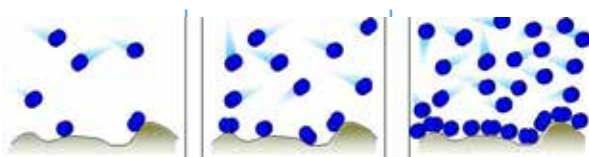
Measurements at lower absolute pressures can also be very effective, and this is why krypton (whose saturation pressure at liquid nitrogen temperature is approximately 1/300<sup>th</sup> that of nitrogen at the same temperature) is often employed when measuring extremely low surface areas (less than 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 cm<sup>3</sup> at liquid nitrogen temperature (77.4K) contains almost four times as many unadsorbed molecules as every cm<sup>3</sup> 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.



## Measurement Capabilities & Applications

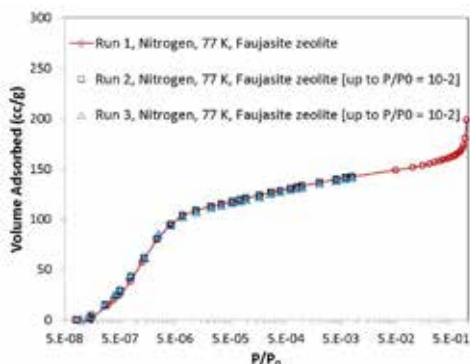
### Gas Sorption

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 m<sup>2</sup>/g and upwards can be accurately determined using nitrogen (at liquid nitrogen temperature) or argon (at liquid argon temperature). Using krypton at liquid nitrogen temperature, surface areas down to 0.0005 m<sup>2</sup>/g can be accurately measured due to the added sensitivity. The ASiQwin software automatically displays the BET plot and computes the BET "C" constant, y-intercept, 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.

## TRX- For Advanced Catalyst Characterization

### TPR: Temperature Programmed Reduction



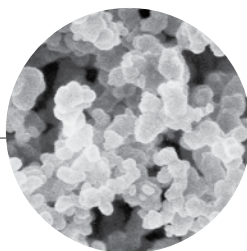
Many heterogeneous catalysts are used as the zero-valent metal, but start life as the oxide. An important factor in catalyst design and use is the ease of reduction of the metal oxide and TPR is a direct measure of that.

A reducing gas mixture, say 2%-5%  $H_2$  in  $N_2$ , 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)

### TPO: Temperature Programmed Oxidation

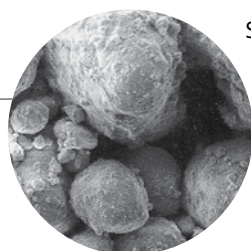


Carbons and carbides are amenable to evaluation by careful oxidation while being heated. A stream of diluted oxygen (e.g. 2-10%  $O_2$  in He) directed over the sample during a linear heating ramp generates a signal due to the loss of  $O_2$  from the gas stream. The products of oxidation, CO and  $CO_2$ , 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.**

### 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)

### 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.





**autosorb®** **iQ**  
 automated gas sorption analyzer  
 surface area, pore size, chemisorption

GAS SORPTION

## Sample Preparation: Degassers

Consistent and reliable surface area and pore size distribution results depend upon proper sample preparation procedures. In terms of BET analysis, the limitation in throughput is often sample preparation. The complete degassing of samples can often require several hours, while surface area measurements may require as little as a few minutes.

Quantachrome manufactures several models of degassers to match and fulfill your sample preparation needs. These degassers provide a virtually continuous supply of properly prepared samples for the AUTOSORB® iQ Surface Area and Pore Size Analyzer to supplement the four standard built-in\* iQ degas ports for high throughput labs.

\* Except in the Autosorb iQ-AG-CX model.

### XeriPrep™ Degasser



The gold standard in sample preparation for physisorption analyzers. The XeriPrep™ provides both manual and automated PC control of up to six heating zones with individual heating ramps and hold times. The built-in cold trap enables efficient removal of condensables at low degassing temperatures and provides for a clean vacuum. A vacuum pump is included, and a Turbo-pumped version is available for rapid high-vacuum degassing.

Vacuum pump is included.

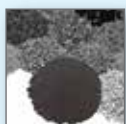
### MasterPrep™ Degasser



Fully featured sample preparation unit. Up to six individually heated stations with programmable heating profiles for R&D, commercial labs and busy multi-user facilities in general. The MasterPrep™ offers dual modes (vacuum or flow) for further flexibility and optimization. Temperature control and logging via PC software are included. An integral cooling station provides additional convenience.

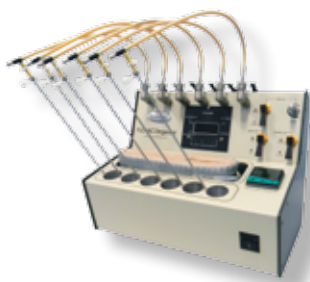
Vacuum pump is not included.

Contact your local representative or call 1.800.989.2476 (in USA/Canada) to request pricing information.



## Flexible Options In Sample Preparation

### 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. Includes individual cooling stations.

Vacuum pump is not included.

### DEGASSERS

Features	FloVac™	MasterPrep™	XeriPrep™
# of Sample Ports ▶	6	6	6
Heating Zones ▶	1	6 (ovens)	6 (mantles)
Maximum Temperature ▶	400°C	425°C	350°C (450°C with optional high temperature mantles for quartz cells)
Temperature Control Via PC ▶	Yes	Yes	Yes
Flow Degas Mode ▶	Yes	Yes	No
Vacuum Degas Model ▶	Yes	Yes	Yes
Digital Vacuum Display ▶	Yes	Yes	Yes
Cold Trap ▶	No	No	Yes
Vacuum Pump ▶	Sold Separately	Sold Separately	Included (turbo pump optional)
Valve Type ▶	Mechanical	Mechanical	Electric solenoid





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.

Renowned innovator for today's porous materials community.

## Quality Function Dependability

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, LabQMC ([www.labqmc.quantachrome.com](http://www.labqmc.quantachrome.com), [qc.lab@quantachrome.com](mailto:qc.lab@quantachrome.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 Dr. Matthias Thommes, 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.

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