Quantachrome
INSTRUMENTS
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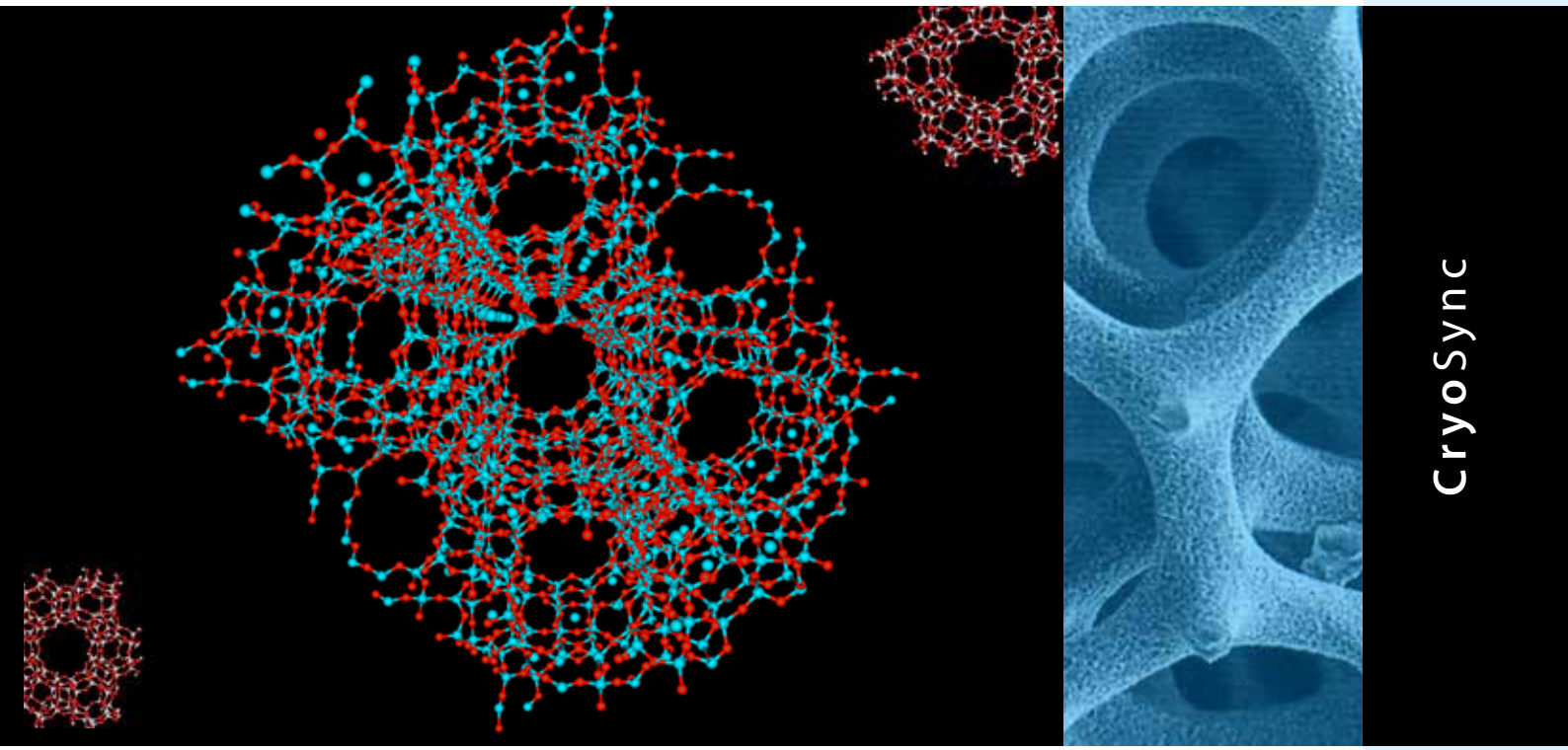
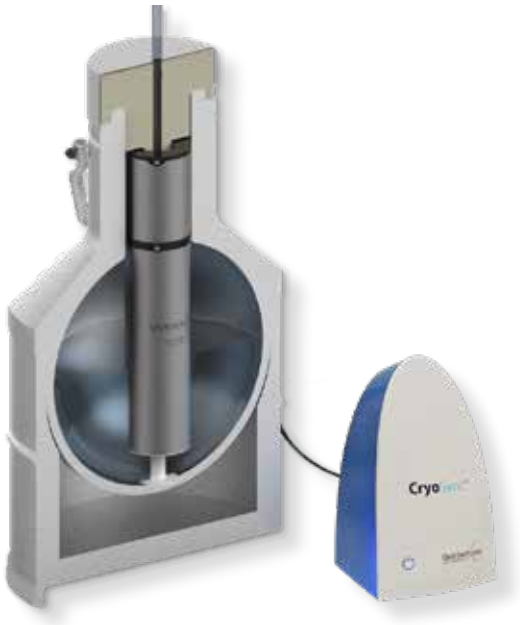
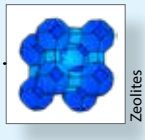
CryoSync®

Cryostat Accessory for Autosorb-iQ



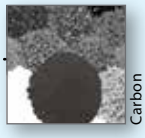


Zeolites



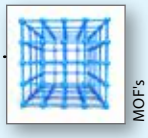
Catalysts

Carbon



Polymers

MOF's



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CryoSync®
Cryostat Accessory for Autosorb-iQ

CryoSync® Accessory to Cryostat Accessory for Autosorb-iQ

Enabling Cost-Effective IUPAC-Recommended Argon Sorption Analyses at 87.3 K

The fundamental and practical advantages of using argon at 87.3 K to characterize porous materials are now well established [1]. For example, compared to nitrogen, argon has no quadrupole moment. Therefore, argon molecules do not tend to orient or pack themselves in special ways in response to interactions with polar surface sites (ions, functional groups, etc.). This means that argon isotherms are insensitive to surface chemistry and sensitive only to surface physical and pore structural features.

Argon sorption isotherms could be collected at 77.4 K (liquid nitrogen temperature). However, at 77.4 K argon falls slightly below its bulk triple point temperature, and its bulk reference state (and hence its saturation pressure, P_O) is less well defined. In contrast, at 87.3 K (liquid argon temperature), experimental conditions are ideal for obtaining full adsorption/desorption isotherms that can be more directly and unambiguously correlated with surface and pore structural features. Argon adsorption at 87.3 K is therefore considered to be more reliable and is now recommended by IUPAC for gas sorption analysis, most particularly for materials containing micropores [1].

Physisorption measurements at 87.3 K can be performed by using either liquid argon (instead of liquid nitrogen) or a cryocooler (or cryostat). However, liquid argon tends to be significantly more expensive than liquid nitrogen, and its continuous use involves additional fixed (tank rental, storage) and variable (cryogen refill) costs relative to those of using liquid nitrogen alone.

Figure 1.
Autosorb iQ model with **CryoSync®** Accessory.

On the other hand, compressor-based cryocoolers tend to be expensive by design. Consequently, there has been a need in the art for a cost-effective means to enable the generation of argon isotherms at 87.3 K as recommended most recently by IUPAC. Quantachrome Instruments is proud to introduce such a means in the form of a **CryoSync®** Accessory for the Autosorb iQ. **(Figure 1)**.

CryoSync® Features at a Glance

Introducing the CryoSync® Accessory to Gas Sorption Analyzers

The **CryoSync**® is a deceptively simple device entirely developed by Quantachrome Instruments (patent pending). This amazingly powerful and affordable cryocooling accessory allows users of **Autosorb iQ** analyzers and other suitable gas sorption instruments to smoothly synchronize:

(i) the generation of highly precise sorption analyses at 87.3 K using liquid nitrogen instead of more expensive cryostats or liquid argon sources, with (ii) the latest IUPAC recommendations for obtaining the most reliable gas sorption analyses [1].

The **CryoSync**® Accessory (**Figure 2**) was initially developed in response to the rising demand for argon analyses at liquid argon temperature (87.3 K). As such, its ability to reproduce the results of standard ASiQ analyses (performed with liquid argon, continuous P_O measurement and a coolant level sensor) is excellent – see **Figure 3**. However, this product is much more than a low-cost alternative to conventional cryostats. In fact, **CryoSync**® users can perform long-duration (>50 hours at 87.3 K) physisorption analyses at virtually constant temperatures (±0.005 K) in the range 82-115 K.

Figure 2.
Quantachrome **CryoSync**® Accessory.

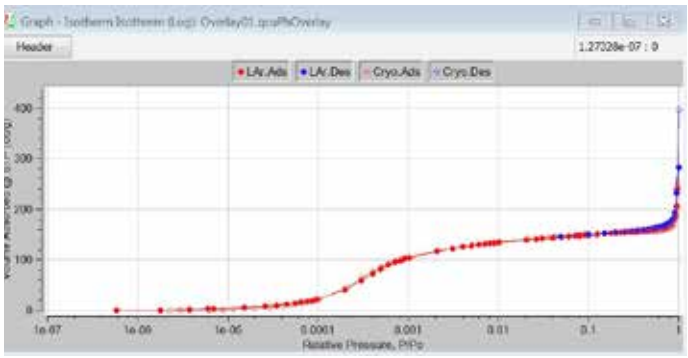

Figure 3.
Overlay of BCR-704 Reference Zeolite Analysis by Argon at 87.3 K Using Standard ASiQ with:
(a) Liquid Argon; and
(b) **CryoSync**™ Accessory.

CryoSync® Feature at Glance (Continued)

CryoSync® Capabilities

Thanks to this outstanding ability, **CryoSync®** users no longer need to perform continuous P_O measurements or dedicate instrument ports or pressure transducers solely to compensate for unavoidable changes in cryogen-dependent P_O values with environmental factors (cryogen purity, ambient pressure, etc.). The exceptional long-term stability of measured temperature readings and calculated P_O values attained using the **CryoSync®** during an extended microporous sample analysis at a user-selected target temperature of 87.3 K are illustrated in **Figure 4.**

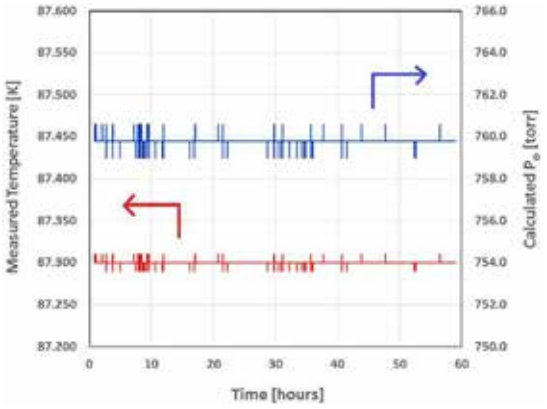

Figure 4.
Temperature and P_O Stability Measured During an Extended (~60 hour without optional refill) **CryoSync®** Analysis at a Target Temperature Set at the boiling point of liquid argon (87.3 K) for illustration.

The ability of the **CryoSync®** to enable the acquisition of high resolution isotherms within the range 82-115 K with remarkable precision is illustrated in **Figure 5**. Isotherms collected at multiple temperatures allow users to perform heats-of-adsorption (HoA) calculations via the standard Clausius-Clapeyron method, in order to obtain additional information about the surface energetic heterogeneity of their samples [2].

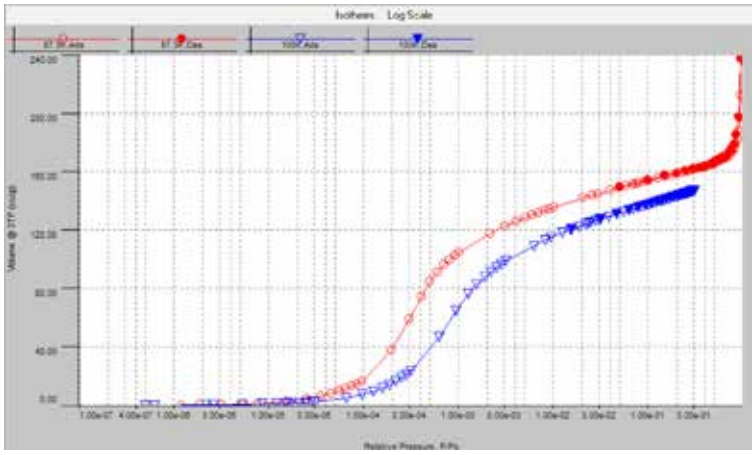

Figure 5.
Illustration of **CryoSync®** Sensitivity to Small Target Temperature Changes Through Overlay of BCR-704 Reference Zeolite Analysis by Argon Adsorption at 87.3 and 115.0 K.

Visit www.quantachrome.com for more detailed instrument specifications and downloadable brochures.

CryoSync® Operation and Software

Principle of Operation

The **CryoSync**® operates by precisely controlling the temperature of a highly stable insulated metal thermostatic block, inside of which the sample to be analyzed is placed. The temperature of the thermostatic block is regulated by warming a heat sink immersed in liquid nitrogen using an external temperature controller. The temperature of the thermostatic block is measured and fed to an advanced software algorithm that controls the power delivery to the heater in the heat sink. The **CryoSync**® design is capable of maintaining temperature stability in the thermostatic block with a precision as remarkable as ±0.005 K (Standard Deviation) for extended duration analyses typically required by microporous materials – for example, as noted above, a minimum of 50 hours when controlling at liquid argon temperature (87.3 K).

Although the **CryoSync**® can function without a PC, it is supplied with software that allows users to monitor and record temperature readings in real time. The **CryoSyncWin**® Controller software program (**Figure 6**) enables the collection, storage and display of up to seven consecutive days of data comprising temperature and power output signals at user-selectable sampling rates and temperature units (ºC or K).

The software is required when users need to perform measurements at temperatures other than the default 87.3 K temperature (i.e., that of liquid argon). The software is also useful to monitor and confirm temperature and power output stability throughout analyses, and to calibrate the analysis temperatures. It is important to note that small changes in temperature can induce large changes in P_O values. For example, the vapor pressure of argon is extremely sensitive to temperature variations – it can vary by around 10 torr for every 0.1 K change at around 87 K. This is one of the main reasons why precise temperature control is so important, and one of the main benefits of the **CryoSync**®, since it provides constant temperature (and therefore, constant P_O) readings throughout extended analyses, thereby eliminating variations related to P_O drifts and costs associated with dedicating ports and transducers to measuring P_O continuously.

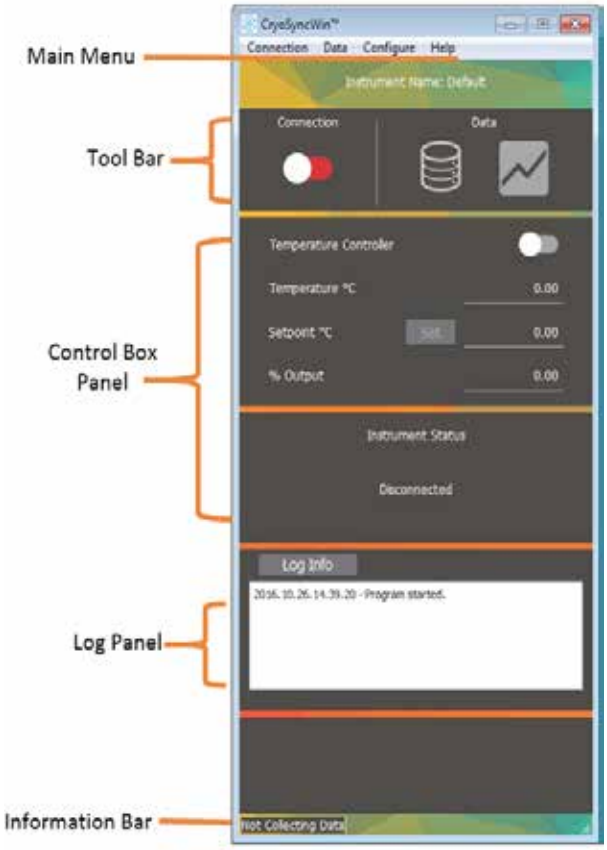

Figure 6.
CryoSyncWin®
Temperature Monitoring Software Screen.

References.

1. M. Thommes, K. Kaneko, A.V. Neimark, J.P. Olivier, F. Rodriguez-Reinoso, J. Rouquerol and K.S.W. Sing, "*Physisorption of Gases, with Special Reference to the Evaluation of Surface Area and Pore Size Distribution (IUPAC Technical Report),*" Pure Appl. Chem. **87**(9-10), 1051-1069 (2015).

2. S. Lowell, J.E. Shields, M.A. Thomas and M. Thommes, "*Characterization of Porous Solids and Powders: Surface Area, Pore Size and Density,*" Springer, Dordrecht (2006), p. 248.

Quantachrome INSTRUMENTS
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CryoSync®

Cryostat Accessory for Autosorb-iQ

CryoSync® Features

Features

- Performs analyses at liquid argon temperature (87.3 K) using liquid nitrogen (77.4 K).
- Temperature stability as precise as ± 0.005 K (std. dev.).
- Analysis duration greater than 50 hours without optional refill.
- Wide temperature range (82-115 K).
- Menu-driven, easy-to-use software allows the user to collect, display, analyze, and archive data.
- Multiple instruments can be controlled by a single computer.
- USB interface between instrument and PC allows remote access to all functions and data.
- Universal accessory, easily adapts to any commercial gas sorption analyzer able to accept an external temperature control option.
- Ideally suited to synchronize the need for liquid-argon-free analyses with **IUPAC-Recommended** high resolution micropore analyses using argon gas at liquid argon temperature (87.3 K).
- Entirely developed and exclusively supplied by Quantachrome (patent pending).

Figure 7.
CryoSync® Assembly Detail.

Visit www.quantachrome.com for more detailed instrument specifications and downloadable brochures.

CryoSync® Benefits

Benefits

- Significant cost savings (no liquid argon or cryostats needed) for **IUPAC-Recommended** Ar/87 K analyses.
- Highly precise isothermal analyses without the need to compensate for P_o changes or for dedicated P_o measurements.
- Suitable for **extended micropore analyses**.
- Enables heats of adsorption measurements.
- Allows performance monitoring in real time to ensure high data quality.
- Saves PC and bench space, while providing easy control from a central location.
- Grants flexible access to all data and software functions.
- Can be adapted for use with gas sorption analyzers other than Quantachrome's instruments.
- Follows the latest **IUPAC-Recommendations** to avoid issues with N_2 quadrupole interactions and provide faster and more reliable Ar/ 87 K analyses.
- Combines experimental advantages (**CryoSync®**) with advanced fundamental models (e.g., **QSDFT** kernels enabling hybrid combinations of pore geometries and independent analyses of metastable fluid-corrected adsorption branch vs. equilibrium desorption branch to ensure the most meaningful evaluation of pore structure network/percolation effects[a]).

[a] http://www.quantachrome.com/technical/dft

Figure 8.
CryoSync® Operation Mode.

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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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Serving Porous Materials and Powder Characterization Needs Since 1968