

TriStar™ II 3020

Surface Area and Porosity System

 micromeritics
TriStar II
Surface Area and Porosity

 micromeritics®
The Science and Technology of Small Particles™

Analytical Versatility/High Throughput/Small Footprint

TriStar II 3020 Surface Area and Porosity System

Surface area and porosity are important physical properties that influence the quality and utility of many materials and products. Therefore it is critically important that these characteristics be accurately determined and controlled. Likewise, knowledge of surface area and especially porosity often is an important key to understanding the formation, structure, and potential application of many natural materials.

High Sample Throughput/ Analytical Versatility

The TriStar II 3020 is a fully automated, three-station, surface area and porosity analyzer that delivers high-quality data at an affordable price. It is capable of increasing the speed and efficiency of routine quality control analyses, yet has the accuracy, resolution, and data reduction capability to meet most research requirements. The TriStar II also features a new Krypton Option, allowing measurements in the very low surface area range. The instrument also combines versatility in analysis methods and data reduction to allow the user to optimize analyses to specific applications.



A Small Footprint/Packed with Features

- Three analysis ports operate simultaneously and independently of one another. Three BET surface area measurements can be performed in less than 20 minutes. For additional throughput, four TriStars can be operated with one computer.
- Surface areas as low as 0.01 m²/g can be measured with the standard nitrogen system. The TriStar II also accommodates the use of argon, carbon dioxide, and other non-corrosive gases such as butane, methane, or other light hydrocarbons. A Krypton Option can extend surface area measurements to as low as 0.001 m²/g.
- A dedicated P₀ port is standard, allowing the measurement of saturation pressure on a continuous basis. Saturation pressure can be entered manually, measured continuously, or collected over the sample. The TriStar II provides the flexibility to control and fine-tune analysis speed and accuracy.

- Incremental or fixed dosing routines prevent overshooting pressure points while minimizing analysis time.
- Free space can be measured, calculated, or manually entered providing maximum flexibility in accommodating special sample types and emphasizing speed when needed.
- Enhanced product support features include: video clips; Ethernet communication between the computer and TriStar; bar code reader capability; built-in electronic test points and diagnostic software; ability to perform remote diagnostics via the internet; and the ability to read and compare historical TriStar and Gemini data to TriStar II data.
- A 2.75-liter Dewar and extended length sample tubes allow complete adsorption and desorption isotherms to be collected without operator intervention.
- The TriStar II can collect up to 1000 data points. Fine details of the isotherm can be observed and recorded providing high resolution and revealing pore structure details.
- Intuitive and powerful Windows®-based software allows more versatility in data archiving and networking. However, the most powerful features of this software are found in its expanded range of data reduction and reporting. SPC reports, new isotherm and thickness models, isosteric heat of adsorption, and integrated DFT models are all included.
- Optional sample preparation devices are available combining flowing gas and/or vacuum with heat to remove atmospheric contaminants, such as water vapor and adsorbed gas, from the surface and pores of the sample.
- An attractively designed cabinet combines a small footprint with easy accessibility.



Typical TriStar II Applications

Pharmaceuticals – Surface area and porosity play major roles in the purification, processing, blending, tableting, and packaging of pharmaceutical products as well as their useful shelf life, dissolution rate, and bioavailability.

Ceramics – Surface area and porosity affect the curing and bonding of greenware and influence strength, texture, appearance, and density of finished goods. The surface area of glazes and glass frits affects shrinkage, crazing, and crawling.

Adsorbents – Knowledge of surface area, total pore volume, and pore size distribution is important for quality control of industrial adsorbents and in the development of separation processes. Surface area and porosity characteristics affect the selectivity of an adsorbent.

Activated Carbons – Surface area and porosity must be optimized within narrow ranges to accomplish gasoline vapor recovery in automobiles, solvent recovery in painting operations, or pollution controls in wastewater management.

Carbon Black – The wear lifetime, traction, and performance of tires are related to the surface area of carbon blacks used in their production.

Catalyst – The active surface area and pore structure of catalysts influence production rates. Limiting the pore size allows only molecules of desired sizes to enter and exit, creating a selective catalyst that will produce primarily the desired product.

Paints and Coatings – The surface area of a pigment or filler influences the gloss, texture, color, color saturation, brightness, solids content, and film adhesion properties. The porosity of a print media coating is important in offset printing where it affects blistering, ink receptivity, and ink holdout.

Projectile Propellant – The burn rate of propellants is a function of surface area. Too high a rate can be dangerous; too low a rate can cause malfunction and inaccuracy.

Medical Implants – Controlling the porosity of artificial bone allows it to imitate real bone that the body will accept and allow tissue to be grown around it.

Electronics – By selecting high surface area material with carefully designed pore networks, manufacturers of super-capacitors can minimize the use of costly raw materials while providing more exposed surface area for storage of charge.

Cosmetics – Surface area is often used by cosmetic manufacturers as a predictor of particle size when agglomeration tendencies of the fine powders make analysis with a particle-sizing instrument difficult.

Aerospace – Surface area and porosity of heat shields and insulating materials affect weight and function.

Geoscience – Porosity is important in groundwater hydrology and petroleum exploration because it relates to the quantity of fluid that a structure can contain as well as how much effort will be required to extract it.

Nanotubes – Nanotube surface area and microporosity are used to predict the capacity of a material to store hydrogen.

Fuel Cells – Fuel cell electrodes require high surface area with controlled porosity to produce optimum power density.



Superior Data Presentation Capability

Operating Software

The TriStar II 3020 Windows interface provides a familiar environment for the user. It is easy to collect, organize, archive and reduce raw data, and store standardized sample information for later use. The reports may be generated to screen, paper, or data transfer channels. Cut-and-paste graphics, scalable-and-editable graphs, and customized reports are easily generated.

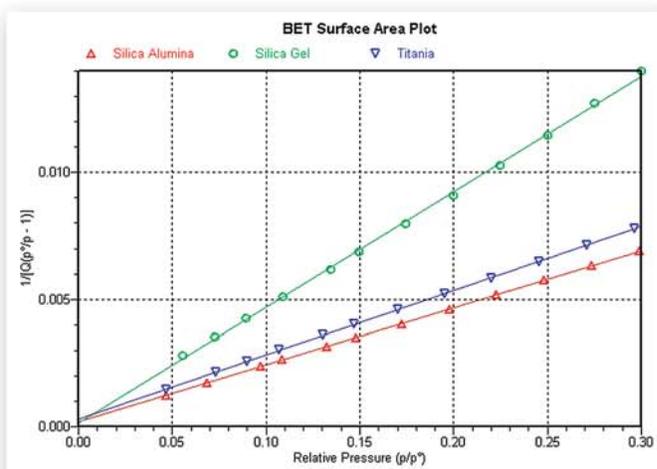
In addition to controlling instrument operation, the Windows software also reduces the raw data collected during analysis. The reduced data can be reviewed or printed in a variety of easy-to-interpret tabular and graphical reports. These include:

- Single- and multipoint BET surface area
- Total pore volume
- Langmuir surface area and isotherm reports
- t-Plot
 - Harkins and Jura Thickness Equation
 - Halsey Thickness Equation
 - Carbon STSA
 - Broekhoff-de Boer
 - Kruk-Jaroniec-Sayari

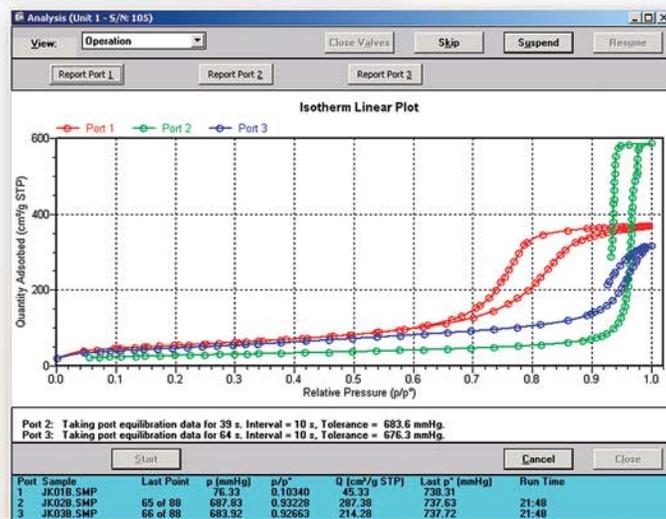
- BJH adsorption and desorption
 - Standard
 - Kruk-Jaroniec-Sayari correction
- Dollimore-Heal adsorption and desorption
- Mesopore and Macropore
 - Volume and area distributions by pore size
- MP-Method
- DFT pore size
- DFT surface energy

- Summary report
- SPC reports
- Validation reports

For applications that fall under the FDA's 21 CFR 11 rule, Micromeritics' optional **confirm™** software option provides the security features and audit trails required by this regulation.

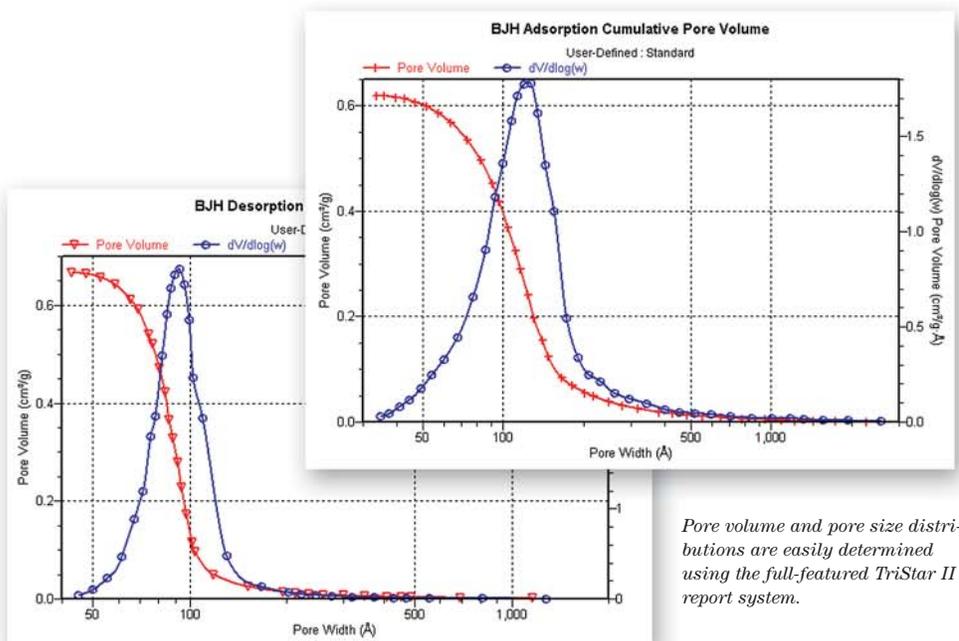


This BET plot of 200 m²/g silica alumina (red), 50 m²/g silica gel (green), and 200 m²/g titania (blue) demonstrates the versatility of the TriStar II. Three different materials may be analyzed simultaneously and then overlaid for comparison.

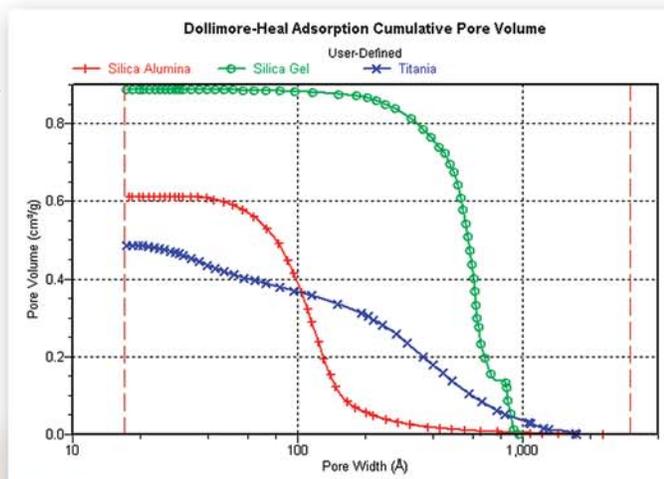


An example of an analysis in progress – the TriStar II features an embedded microprocessor that controls the unit operation and sample analysis. This user-friendly Windows software allows the user to control the instrument from a workstation, monitor the progress of the analysis, and view the results of the experiment.





Pore volume and pore size distributions for several samples may be overlaid to emphasize the difference between various materials.



TriStar II Reference Materials

Silica-alumina

The silica-alumina is a typical porous high surface area reference material. The surface area of silica-alumina typically exceeds 200 m²/g and the pore size is a nominal 100 Å. This material is recommended for users that analyze amorphous materials with surface area ranging from 10 to greater than 300 m²/g and for both non-porous and porous materials in the 40-3000 Å range. Silica-alumina is suitable for use with BET, t-plot, and BJH pore size reports.

Carbon Black

Standard Reference Blacks are available from 20 to greater than 100 m²/g. These are stable and very well characterized materials. They are recommended for all users but may be especially suited for researchers in the carbon, tire, and filler industries. The carbon black reference materials are suitable for use with the BET and STSA reports.

Glass

A 5 m²/g glass reference material is recommended for industries and users that characterize materials in the 1 through 50 m²/g range. Glass reference material is suitable for use with the BET surface area report.

Alumina

Low surface area alpha alumina (less than 1 m²/g) is available for use with krypton. Alpha alumina is recommended for researchers in the pharmaceutical, abrasives, soil, and other areas that require the characterization of materials with surface areas less than 1 m²/g. Alpha alumina reference material is recommended for use with krypton and the BET surface area report.



Innovative Design

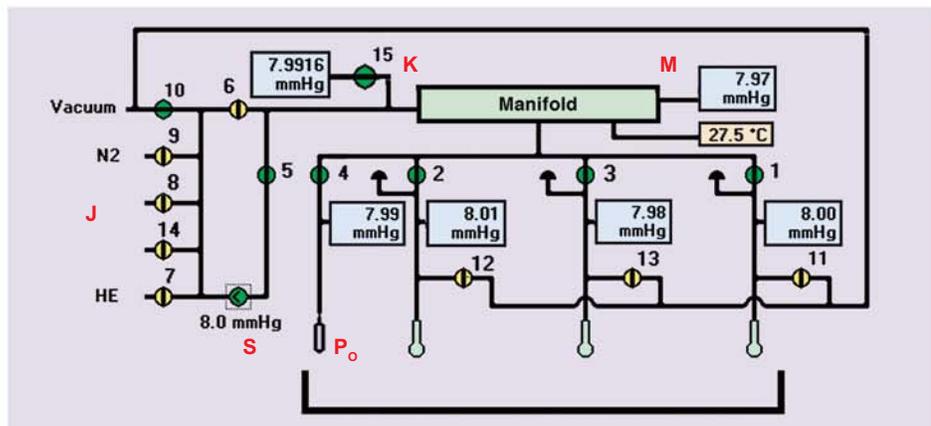
The TriStar II Technique

Micromeritics' TriStar II utilizes the static adsorption technique. The instrument features a thermally stable dosing manifold (M), a three-port sample manifold, a dedicated tube for measuring saturation pressure (P_o), and a rapid response servo valve (S). The main manifold features a 1000-torr transducer and is coupled to the rapid response servo valve. The servo valve is used for rate-controlled evacuations of the sample, and used throughout the analysis to rapidly and accurately dose the manifold. The servo-controlled evacuation rate may be set by the user to minimize the risk of fluidizing powder samples. For pellets, spheres, extrudates, or other formed samples, the evacuation rate may be set very high to rapidly evacuate the sample tube.

During a typical analysis, the manifold, sample tubes, and the P_o tube are evacuated. After a sufficient vacuum has been achieved, the manifold is filled with helium and then sample port 1 valve is opened to determine the warm free space of sample 1. This sequence is repeated for samples 2 and 3 to determine the free space at room temperature. The elevator is raised and the samples are cooled to nearly 77 K. This allows the free space to be determined at the analysis temperature. Once the free space analysis is finished, the saturation pressure of the adsorptive is determined using the P_o tube. Typically nitrogen is dosed into the tube above atmospheric pressure. The nitrogen is allowed to condense and the vapor pressure of the nitrogen is easily monitored by a transducer throughout the analysis.

The adsorption isotherm is rapidly collected by using the servo valve to dose nitrogen into the manifold. The pressure and temperature of the nitrogen are recorded, a sample port is opened, and the nitrogen is allowed to adsorb onto the sample. The quantity of nitrogen removed from the manifold is recorded as the quantity dosed. The sample valve is then closed and the adsorption is allowed to proceed to equilibrium. The quantity adsorbed can be calculated from the quantity dosed minus any residual nitrogen in the sample tube. This process is repeated for all three samples. The analysis is a parallel operation so that while one sample is equilibrating a different sample can be dosed with nitrogen.

The order of sample analysis is based on the material, not sequential operation. Samples that adsorb nitrogen rapidly can be analyzed in parallel with materials that are tortuous and require additional time to adsorb nitrogen. This allows the user to characterize three unique materials simultaneously providing rapid, reliable, and repeatable results.



The TriStar II also features four gas inlets for probe molecules (J). A typical configuration will feature nitrogen as the analysis gas and helium for determining the void volume of the sample tube. However, two additional gases may be connected for additional flexibility. For example, carbon dioxide is often used for difficult-to-analyze microporous materials, and krypton may be preferred for low surface area samples (requires additional hardware for krypton analysis option-K).

- Three Parallel Sample Analyses
- Continuous P_o Monitoring
- Sliding Shield
- Large Capacity Dewar



Accessories

Sample Preparation Devices

Micromeritics' sample preparation devices prepare batches of samples for surface area and pore volume analysis. They combine flowing gas and/or vacuum with heat to remove atmospheric contaminants, such as water vapor and adsorbed gas, from the surface and pores of the sample. The quality of the data produced by surface area and pore volume analyses depends greatly on the cleanliness of the sample surface. All Micromeritics' sample preparation devices accept helium, nitrogen, argon, and other non-corrosive gases.

The **SmartPrep™ 065** is a flowing-gas degassing unit which removes adsorbed contaminants from the surface and pores of a sample in preparation for analysis. It contains six sample ports, each one independently temperature-controlled for greater flexibility. It contains two serial ports, one for connecting to the computer and the other available for connection of an additional SmartPrep. The temperature, ramp rates, and soak times of each sample are individually controlled by the computer. Up to five ramps and soaks are allowed. All degas information is integrated into the sample data file for future reference.

The **FlowPrep™ 060** applies both heat and a stream of inert gas to the sample. The heat causes contaminants to desorb from the surface and the stream of inert gas sweeps them out of the sample tube. It lets you choose the temperature, gas, and flow rate best suited for your sample material and application. Needle valves allow you to introduce the flowing gas slowly to prevent fluidization of samples.



SmartPrep 065

VacPrep 061

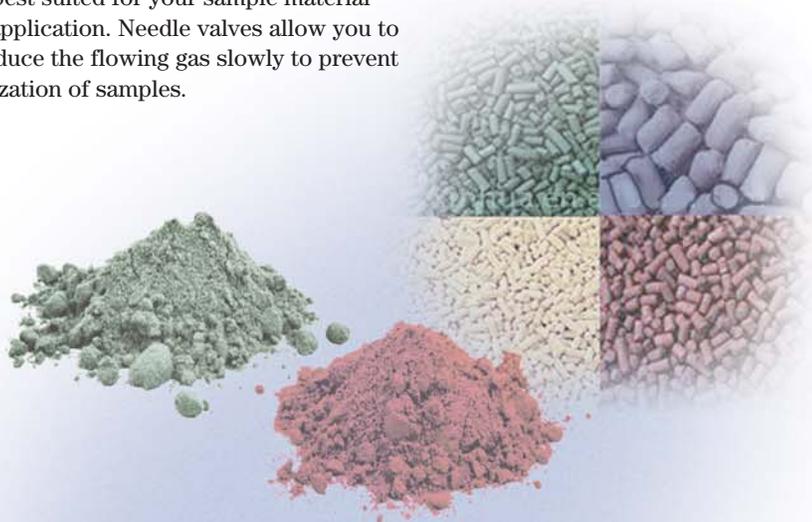
The **VacPrep™ 061** offers two methods for removing contaminants. In addition to flowing gas, it provides vacuum to prepare samples by heating and evacuation. This combination allows you to choose the preparation method that is best suited to your material or application. The VacPrep features six degassing stations, and a choice of vacuum or gas flow preparation on each of the six stations. Needle valves are also provided allowing you to introduce the flowing gas or vacuum slowly to prevent fluidization of samples.

Model 021 LN₂ Transfer System

The Model 021 LN₂ Transfer System allows you to transfer liquid nitrogen or liquid argon from a nonpressurized storage Dewar into smaller containers used in laboratory experiments.

The system was specifically developed for conveniently filling Dewars for gas adsorption instruments but also can be used for other cryogen applications. The Model 021 can discharge cryogens at adjustable rates up to 3 liters/min. The roller base makes it easy to move the 021 System to the location where the cryogen is needed. The spigot and insulated, flexible hose enable convenient filling and refilling of analysis Dewars. The system can hold liquid nitrogen or liquid argon up to 30 days allowing cost-efficient use of your cryogen.

Additional accessories are available for special applications.



To request a quote or additional product information, visit Micromeritics' web site at www.micromeritics.com, contact your local Micromeritics sales representative or our Customer Service Department at (770) 662-3636.



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