



Laboratory Directory: Application & Measurement Services

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Quantachrome understands that not everyone can purchase an instrument for their porous materials characterization, and that they would rather turn to an experienced laboratory to perform the measurements for them. Welcome to Quantachrome's Application Laboratory whose staff not only has the experience but also the expertise you need for meaningful powder and porous materials characterization. An initial consultation, always free-of-charge, ensures the best test method will be used so that you will receive results that both make scientific sense and that have practical use. When the results are ready, they are sent electronically or faxed, with a hardcopy mailed out if you prefer - again at no extra charge. And, being a customer-oriented company, we will help you interpret the data. Most of the individual measurements that we offer are listed at right, and described briefly below. If you do not see what you need, or need more information, including a competitively priced, up-to-date fee schedule, please do not hesitate to contact the Application Laboratory staff.

Surface Area Measurements

Samples are outgassed under vacuum or inert gas purge, usually at some elevated temperature. If your samples are heat or light sensitive, do not worry, just let us know and we will take appropriate care. The surface area is measured by nitrogen adsorption or, in the case of very low surface areas (say less than 1m² total available area), by krypton adsorption, both at liquid nitrogen temperature.

Pore Size, Pore Volume Measurements

Micropores (i.e. those of molecular size) are analyzed by gas adsorption at very low pressures on state-of-the-art high vacuum systems. Resulting data are appropriately processed by the largest commercially available range of advanced computation methods. Large macropores (i.e. over 0.5 microns, and up to ~900 um diameter) are analyzed by the forced intrusion of liquid mercury. And we take care of mercury disposal concerns. Pores of

intermediate size (mesopores) may be characterized by either gas sorption or mercury porosimetry. We will gladly discuss your application and make suitable suggestions as to which method is more appropriate.

Density Determinations



True (skeletal) density is determined by automatic gas-expansion pycnometer so there are no solubility issues as might be encountered with liquid pycnometers. A variety of gases are available, including (but not limited to) helium, nitrogen, argon, carbon dioxide, sulfur hexafluoride. The correct selection is made according to

sample type (mineral, vegetable, etc). Gas pycnometry is also used for open/closed cell determination of polymer foams. Tapped density, a space-filling property of bulk powders, is reported not as one single value, but as the trend according to number of taps. Hausner Ratio and Carr's Index (both indicators of flow and compressibility properties) are reported at no additional charge. Dry powder pycnometry is used to determine geometric (envelope) volume/density of irregular pellets, granules etc.

Water (And Other Vapor) Sorption

Complete moisture adsorption isotherms can be performed on your samples anywhere from 12°C to 85°C. Two or more isotherms (at different temperatures) can be used to yield adsorption enthalpy. Please note, even hydrophobic samples can adsorb moisture from the vapor phase due to capillary condensation forces! Ideal for investigating coatings, deliquescence, etc. Organic vapors can be used to study differences between polar/non-polar surfaces. ■

For more information about measurement capabilities and how to submit a sample, contact Quantachrome Instruments by phone: (561) 731-4999, fax: (561) 732-9888, email: QC.lab.pharma@quantachrome.com or visit www.quantachrome.com.

SURFACE AREA | USP <846>

- ▶ BET, 1-point, N₂
- ▶ BET, multi-point, N₂
- ▶ BET, multi-point, Kr
- ▶ NSA, see BET
- ▶ STSA, multi-point, N₂
- ▶ Micropore, t-plot method

PORE SIZE | USP <267>

- ▶ Micropore, (gas sorption)
- ▶ Mesopore, (gas sorption)
- ▶ Mesopore, (mercury intrusion)
- ▶ Macropore, (mercury intrusion)

PORE VOLUME

- ▶ Gas adsorption, 0.3nm to 500nm pore diameter
- ▶ Mercury displacement/intrusion, 3.5nm to 900µm



DENSITY | USP <616> | USP <699>

- ▶ True/skeletal, gas pycnometer
- ▶ Open/closed, gas pycnometer
- ▶ Geometric/envelope, powder pycnometer
- ▶ Tapped density
- ▶ Hausner ratio
- ▶ Carr's index



VAPOR SORPTION

- ▶ Water
- ▶ Alcohols
- ▶ Aromatics
- ▶ Other hydrocarbons

For More Information: Please avail yourself of the easy access to our experts if you are at all unsure as to exactly what to request: eg. Pore size analysis is the least trivial of all of the cited techniques.

Contact us at:
QC.lab.pharma@quantachrome.com
or visit us on our website at:
www.quantachrome.com.



Powder & Porous Materials Characterization

Quantachrome
INSTRUMENTS

Instrument Directory: Porous Materials & Powder Characterization

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Selecting the correct instrument to characterize your powders and porous solids can be a daunting task. Use this directory and selection guide for easier and more streamlined decision making. The focus of this article is on those physical properties whose measurement is often overlooked, but that are no less important than e.g. particle size! Sometimes different techniques can do (at least at first glance) much the same thing. See Pore Size Analyzers (below) for example; in this case you will be guided primarily by the range of pore sizes of interest. The listing given at right is not exhaustive, but combine it with the descriptions below and you will be off to a confident start to secure that new addition to your lab inventory.

SURFACE AREA ANALYZERS | USP <846>

If you want to minimize footprint and conserve bench space, it should have on-board degassing (preparation) station(s). Helium-free operation simplifies setup and reduces operating costs. It is usually possible to analyze with N₂ as low as 1m² available area... less than that might require krypton capability. If it does, demand an oil-free vacuum system (and for degassing too).

DENSITY ANALYZERS | USP <616> | USP <699>

True (solid) density is measured by gas pycnometry. Multiple station pycnometers are available, complete with balance interface, printer port and PC data archiving, plus optional temperature control. You'll need a different (mechanical) device for tapped powder bulk density; easily automated, even on two samples at one time. The same tap density analyzers can even measure geometric (envelope) density, at remarkably low cost.

PORE SIZE ANALYZERS

Micropores require gas sorption technology: look for analyzers with advanced techniques like CO₂ and argon adsorption combined with state-of-the-art DFT (Density Functional Theory) calculation models. Most mesoporous materials can be analyzed by either gas sorption or mercury porosimetry. The latter offers much more rapid analysis however. The most up-to-date porosimeters have automatic purging of the hydraulic system, and vapor traps. Macropore sizes extend well above the upper limit of gas sorption and so must be measured by mercury intrusion. On the right instrument it is possible to measure, as a standard feature, up to 900 μm pore diameter.

WATER SORPTION

Speed, automation and robustness are offered in manometric (volumetric) systems. It is now possible to measure complete moisture uptake isotherms at temperatures from 12°C to 85°C.

SUMMARY

Help in matching state-of-the-art instruments to lab throughput and budgetary needs is available... you are already started down the right track. If you need more assistance in finding just the right instrument to meet your R&D needs or to help solve your particle performance problems, do not hesitate to contact the specialists below. ■

For more information about related measurement instruments, contact Quantachrome Instruments by phone: (561) 731.4999, fax: (561) 732.9888, email: QC.pharma@quantachrome.com or visit www.quantachrome.com.

SURFACE AREA ANALYZERS

HIGH THROUGHPUT

- ▶ Novae 4000 (shown)
- ▶ Quadrasorb-SI
- ▶ Autosorb-3B
- ▶ Autosorb-6B



VERY LOW AREA

- ▶ Autosorb-1-Kr/MP
- ▶ Autosorb-6B-Kr
- ▶ Autosorb-3B-Kr
- ▶ Quadrasorb-SI-Kr

ECONOMICAL

- ▶ Novae 2000
- ▶ Monosorb

PORE SIZE ANALYZERS

MACROPORE

- ▶ PoreMaster Macro

MESOPORE/MACROPORE

- ▶ PoreMaster 33
- ▶ PoreMaster 60
- ▶ PoreMaster GT33
- ▶ PoreMaster GT60 (shown)



MESOPORE

- ▶ Autosorb Series
- ▶ Novae Series
- ▶ Quadrasorb-SI Series

MESOPORE/MICROPORE

- ▶ Autosorb-1-MP
- ▶ Autosorb-3B-MP
- ▶ Autosorb-6B-MP

WATER SORPTION ANALYZERS

MANOMETRIC

- ▶ Hydrosorb 1000
- ▶ Hydrosorb HT

DENSITY ANALYZERS

AUTOMATIC GAS PYCNOMETERS

- ▶ Pentapycnometer
- ▶ Ultrapycnometer
- ▶ Ultrafoam Pycnometer (shown)




ECONOMICAL

- ▶ Multipycnometer
- ▶ Stereopycnometer

TAPPED POWDER DENSITY/ GEOMETRIC DENSITY

- ▶ Dual Autotap
- ▶ Autotap

For More Information: Contact us at QC.pharma@quantachrome.com or visit us on our website at www.quantachrome.com.



Particle Size Analysis?

Is the entire size distribution represented in the small scoopful you just put into your analyzer? Probably not, unless you split the original sample on a Rotary Riffler. Reduce variability in your particle size results... make your next sample a representative one.

Ask about Rotary Rifflers from Quantachrome at QC.pharma@quantachrome.com.

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