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Particulate Systems Adds New Products

In a previous issue of the microReport, Micromeritics announced the formation of Particulate Systems, an umbrella brand created to market an auxiliary selection of laboratory instruments that complement Micromeritics' own line of materials characterization instruments. In 2008, Micromeritics introduced the Particle Insight size and shape analyzer under the Particulate Systems brand. Since that time, Particulate Systems has added new products and announced a strategic collaboration with Surface Measurement Systems Limited.

The **HPVA-100 High-Pressure Volumetric Analyzers** are designed to measure high-pressure adsorption isotherms with hydrogen, methane, carbon dioxide, and other gases using the static volumetric method. Typical applications for this instrument include catalysts, zeolites, activated carbons, carbon nanotubes, and hydrides. Understanding the adsorption characteristics of materials is critical in the research and advancement of hydrogen storage, fuel cells and batteries, stack gas scrubbers, and hydrocarbon traps.



The HPVA product operating pressure ranges from high vacuum to 200 bar. Sample temperature is from cryogenic to 500 °C. Sample analysis data collection is fully automated to assure quality data and high reproducibility. In addition to the single-station model, the HPVA is available in a four-station version that allows up to four sample runs to be completed simultaneously.

The **MA-1040 Magnetic Analyzer** was first designed in 1976 to meet the needs of the abrasive grain industry for ultra-high sensitivity detection of magnetic traces in materials. Since then, the Magnetic Analyzer has undergone numerous improvements including increased sensitivity to 0.00001% magnetic content, increased accuracy, smaller footprint, and many other ease-of-use improvements. The MA-1040 is useful for detecting minute quantities of iron in a wide range of materials including high-purity glass used for fiber optics and plastics used in wiring insulation. The MA-1040 can also be used to detect low magnetic iron levels in food, precious gems, pharmaceuticals, and many other materials. Detecting low levels of iron in raw materials is critical to the outcome of the final product.

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Particulate Systems cont.

In addition, Particulate Systems and Surface Measurement Systems Limited have announced a Global Strategic Collaboration. In the initial phase of this collaboration, Particulate Systems will have the exclusive distribution rights for key Surface Measurement Systems products in selected regions of the United States as well as in South America, Central America, and the Middle East with co-distribution rights in China. These products include the DVS Dynamic Vapor Sorption Systems that utilize dynamic gas flow and the gravimetric technique to produce high-resolution adsorption and desorption isotherms of water and organic vapors on essentially any solid material. Surface Measurement Systems will extend their service and support for customers in the USA via the Particulate Systems extensive aftercare network.



DVS Intrinsic

For more information, visit www.particulatesystems.com

Micromeritics Announces Recent Instrumentation Grant Awards

In 2009 Micromeritics selected two worthy research programs to receive instrument grants as part of Micromeritics' ongoing Instrument Grant Program. An ASAP 2050 Xtended Pressure Sorption Analyzer was awarded to the Physics Department at Southern Illinois University Carbondale and an ASAP 2020 Accelerated Surface Area and Porosimetry System was awarded to the Department of Chemistry at the University of North Texas (UNT), Denton, TX.

Aldo Migone, Principal Investigator, Professor and Chair of the Department of Physics at Southern Illinois University Carbondale stated, "Research activities will span a broad area of material characterization through adsorption/desorption measurements in three departments: Physics, Chemistry, and Mechanical Engineering. The research that this project will encompass includes, but is not limited to, my own work on porous metal-organic frameworks and carbon nanotubes, Prof. Talapatra's work on metal hydrides and carbon nanotubes, Prof. Dave's work on porous sol-gel glasses, and Prof. Mondal's research on active catalysts. The investigations carried out on the ASAP 2050 will provide critical information for developing applications of these nanostructured materials to areas such as gas separation, gas storage, gas sensing and catalysis." Saikat Talapatra (Co-PI, Department of Physics) states, "The availability of this instrument will also give a tremendous boost to the ongoing research efforts on energy that are taking place in and around the SIUC campus."

According to Dr. Mohammad Omary, Principal Investigator and Professor of Chemistry at the University of North

Texas, "Fluorous Metal-Organic Frameworks (FMOFs) have the potential to be used as practical adsorbents for hydrogen fuel cell based technology. The idea is well supported by the fact that the stability, flexibility, and selectivity in gas storage, separation, and catalytic properties of porous materials will be largely improved by substitution of hydrogen atoms with fluorine to make fluororous pores. Indeed, FMOFs have been shown to exhibit superior volumetric capacity for the storage of multiple gas molecules, giving rise to a very promising avenue of hydrogen economy and gas separation research based on this new class of adsorbents. The ASAP 2020 with related components will be an integral and critical piece of characterization equipment within the UNT's Hydrogen Storage and Catalysis Laboratory, which also includes Dr. Chi Yang and several other researchers. I am very grateful to Micromeritics not only for awarding us this grant, but also for helping us generate important data that contributed to the realization of the potential of our materials, as published in leading journals (*J. Am. Chem. Soc.* and *Angew. Chem.*) and independently highlighted by multiple scientific and technology media. With this instrument, we will be readily able to characterize our new materials in hydrogen storage application, as well as in other gas and organic vapor uptake for catalytic and a variety of other applications."

Micromeritics congratulates each of these worthy programs and looks forward to supporting more ground-breaking research in the future. For application details and more information on Micromeritics' Instrument Grant Program, visit our website at <http://www.micromeritics.com/Pressroom/Particle-Characterization-Instruments-Grants.aspx>


www.micRx.com

Micromeritics is pleased to announce the formation of Micromeritics Pharmaceutical Services (MPS), a new contract services organization, dedicated to the drug development and quality control needs of pharmaceutical scientists. A DEA-licensed, FDA-registered, cGMP/GLP-compliant contract analytical laboratory, MPS has the experience necessary to assist today's pharmaceutical scientists with their analytical characterization needs.

Micromeritics Pharmaceutical Services was created to enable Micromeritics to better serve its pharmaceutical customers through contract services and instrument user applications support. This new division enables Micromeritics to expand its analytical service

capabilities and meet the growing demand for contract services from the pharmaceutical industry. MPS also provides a comprehensive service program to provide instrument users with method development and method validation.

New mandates that require additional testing of raw materials have placed material characterization in the spotlight. Navigating all of the available instrument technologies and test methods is particularly overwhelming. MPS can assist in the identification of critical quality attributes (CQAs) and critical process parameters (CPPs) by thorough characterization of raw, in-process, and finished materials. Once the identification is completed, MPS can perform analytical methods devel-

opment and validation studies that enable incorporation of new test methods into appropriate processes as well as providing required information for FDA submissions.

Recently, capabilities added are dynamic vapor sorption, surface energy, and light obscuration for particulate matter testing. Micromeritics Pharmaceutical Services is dedicated to offering the test methods and technology required by today's pharmaceutical scientist and to providing the comprehensive analytical and development services designed to meet the materials characterization needs of the pharmaceutical industry.

Micromeritics Pharmaceutical Services Offers Analytical Methods Development and Validation

Method Development and Method Validation are extremely important to regulatory agencies for submissions and in the development of drug materials. Oftentimes, specific areas of expertise may not be available within the confines of a company or research institution. In many cases, scientists working on investigational new drugs (INDs), new drug applications (NDAs), and abbreviated new drug applications (ANDAs) do not have published compendia or pharmacopeias to reference. Furthermore, many of the analytical technologies currently being used to characterize API's (active pharmaceutical ingredients) and excipients have not been sufficiently developed or validated for these materials and applications.

Micromeritics Pharmaceutical Services is now offering Method Development and Method Validation services. MPS services utilize a variety of analytical tools including BET Surface Area, Particle Size (6 techniques), Particle Shape, Mercury Porosimetry, Vapor Sorption, Surface Energy, and many more. Methods Development and Validation projects that take months and significant expense to develop and complete can be outsourced to MPS for a fraction of the time and cost. MPS resources include an experienced project manager, thoroughly trained analytical chemists, and a fully equipped cGMP/GLP-compliant analytical laboratory that is ready to deliver a developed and validated method in a matter of days.

Whether you are an instrument user seeking assistance with your application or in need of contracting services to support your FDA submission, MPS can provide a highly professional outsourcing solution.

For more information on how we can help with your Method Development and Validation needs, call (770) 662-3393 or visit our website at www.micRx.com.

Electrical Sensing Zone Advantages when Quantifying Particle Contamination in Lubricating Oils

by: Rick Shimkus, Instrumentation Specialist

It is very important to monitor the number and size of contaminants in lubricating oil used in power plant generators, as well as other equipment in many production processes. Contaminated lubricating oils can cause accelerated wear of critical parts, often resulting in premature failure of vital equipment. Reliable particle size and count data allow timely monitoring of lubricating oil cleanliness and scheduling of contaminated oil replacement before it can cause excessive parts wear or breakdown of critical equipment.

Three measuring techniques are generally used to monitor and analyze particle contamination in lubricating oils:

- light microscope
- light blockage (obscuration)
- electrical sensing zone (Elzone)

Microscopic Analysis

The microscopic technique entails examination of particles retained on a filter after a sample of the contaminated oil is passed through the filter. If the particles are unevenly distributed in the oil, this technique often misses one or more classes of particles. Also, microscopic examination only looks at a two-dimensional

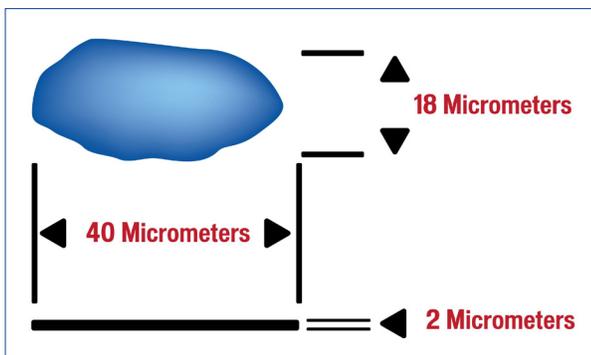


Figure 1. Microscopic examination of this particle could yield cross-sectional sizes from 36 to $\approx 700 \mu\text{m}^2$ depending on particle orientation.

presentation of the particles, essentially length and width (Figure 1). It is very easy to obtain widely varying results depending on the orientation of the particles on the filter media, and microscopic analysis is tedious because many particles must be measured to give reliable statistics.

Light Blockage Analysis

The light blockage (obscuration) technique requires that particles pass, one at a time, between a light source, e.g., a high-intensity white light or a laser, and a detector. Each particle is counted as it passes through the beam and sized by mathematically analyzing its shadow on the detector. This type of analysis does not require capture of particles on a filter; however, some dilution of the sample may be needed to ensure particles pass individually through the beam. Like the microscopic technique, this technique looks only at two dimensions (Figure 1) or the shadow projected onto the detector, and can report particles undersize or oversize depending on their orientation.

The size reported is the diameter of a sphere having the same projected cross-sectional area as the particle. Therefore, an individual flake of metal of sizes depending on its orientation when it transits the beam. Optical properties are also of concern with the light blockage technique. Transparent or nearly transparent particles may not be seen at all.

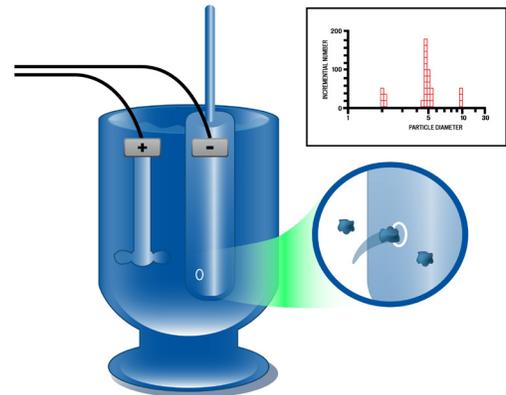


Figure 2. Schematic representation of an electrical sensing zone analyzer.

Electrical Sensing Zone Analysis

The electrical sensing zone technique, used by the Elzone II 5390 Particle Size Analyzer from Micromeritics, counts and measures the size of particles dispersed in an electrolyte or electrically conductive liquid or solution. The electrolyte containing dispersed particles is placed in a special beaker. An orifice tube (a closed-end tube with a small orifice near the closed end) is lowered into the electrolyte. A vacuum applied to the open end of the tube pulls electrolyte containing the dispersed particles from the beaker through the orifice and into the orifice tube. Two electrodes, one inside the orifice tube and one in the beaker, establish an electrical field through the orifice (Figure 2). As individual particles are pulled through the orifice, the electrical resistance between the two electrodes is disturbed as the volume of the particle displaces some of the electrolyte as it passes through the orifice. The change in electrical resistance between the electrodes is proportional to the volume of the particle. Therefore,

each particle passing through the orifice is counted and sized. The data are sent to a computer and presented as a histogram with up to 300 size channels. The sizes of the particles are reported as the diameter of a sphere of equivalent volume.

Optical properties, shape, and density are not problems for the electrical sensing zone technique. The particle's registered volume remains the same when passing through the orifice regardless of the orientation of the particle relative to the orifice. The electrical sensing zone technique also can detect particles in contaminated oil that may be missed by other techniques. Finding contaminated oil early allows prompt corrective action and avoids possible damage of critical, expensive equipment and costly downtime.

The electrical sensing zone technique also can be used to identify unclean containers, a major source

of contaminated oil. Unused containers may look clean but often have unwanted particles inside. This can prevent clean oil from being placed into presumed-clean containers only to become contaminated.

Analysis Methods

The electrical sensing zone technique uses two methods for counting and sizing contaminated particles in oils:

- For light-weight oils such as heating oils, the contaminated oil is mixed with a solvating organic electrolyte and the particles are analyzed.
- For heavier oils such as hydraulic fluids, the contaminated particles are captured by filtration and the filter media with the particles is placed into an electrolyte where the particles are freed using ultrasonic energy. The filter media is removed and



Micromeritics Elzone II

the electrolyte containing the particles is analyzed.

The electrical sensing zone (Elzone II) can be expected to provide more reliable and accurate data than an optical analyzer.

We welcome articles and information concerning particle technology applications performed with Micromeritics instrumentation. See the back page for further information.

Micromeritics New European Technical Center opens in Germany

Micromeritics has opened its new European Technical Center in the Avantis Science Park near Aachen, Germany. "Micromeritics feels it can better serve its customer base across central Europe by locating at the border triangle where Germany, The Netherlands, and Belgium meet," says Ton Hustings, regional manager of Micromeritics Germany and Benelux.

Micromeritics European Technical Center offices are located in a new building with over 500 m² (5,382 sq. ft.) of floor space. The European Materials Analysis Laboratory, offering a wide range of contract materials analysis services, also operates from this new location. Micromeritics joins more than 25 other high technology companies located in this 40.5-hectare (100-acre) office park.

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MAS has recently acquired two new capabilities that complement its current gas sorption capability. The **Dynamic Vapor Sorption (DVS) Analyzer** uses a gravimetric moisture sorption measurement technique to determine sorption isotherms of liquid vapors. The isotherms can be measured from 5 to 60 °C.

Common applications for DVS are:

- Study hygroscopicity of powders, fibers, and solids
- Kinetics of water sorption
- Water- and solvent-induced morphology changes
- Surface energies of powders
- Determination of amorphous content
- Study hydrate/solvate formation
- Heats of sorption
- Effect of moisture on texture
- Diffusion coefficients and activation energies

The **Inverse Gas Chromatograph (IGC)** is basically the inverse of a conventional gas chromatograph. A cylindrical column is uniformly packed with the solid sample material, typically a powder, fiber, or film. A pulse or constant concentration of gas is then injected down the column at a fixed carrier gas flow rate, and the time needed for the pulse or concentration front to elute is measured by a detector. A series of IGC measurements made with different gas phase probe molecules then allows determination of a wide range of physical and chemical properties of the solid sample. IGC provides unique access to the following physical-chemical properties of a wide range of solid materials in a controlled humidity environment:

- Dispersive and polar surface energies
- Heats and entropies of adsorption
- Acid-base interactions
- Phase transitions
- Sorption isotherms
- Permeability, solubility, and diffusion
- Competitive (multi-component) adsorption

Free Water Vapor Sorption Analysis

For a limited time, Micromeritics Analytical Services is offering one free volumetric water vapor sorption analysis (\$750 value) of your sample on the Micromeritics ASAP 2020. For those customers who are new to water vapor sorption, this is an excellent, no-obligation opportunity to determine the water uptake of your sample under a controlled environment. Please go to particletesting.com, and click Sample Submission Form to submit your sample. Complete the form, include an analysis temperature (typically 0 - 40 °C), and simply enter "free water vapor offer" as the type of payment. We'll take care of the rest. Once you receive your results, please feel free to contact us for a complimentary data consultation.

For a limited time, Micromeritics Analytical Services is also offering a 25% discount for gravimetric water vapor sorption on the Surface Measurement Systems DVS. Contact us at (770) 662-3630 or mas@particletesting.com.

Look for MAS at this upcoming event:

Pittcon 2010
 February 28 - March 5, 2010
 Orange County
 Convention Center
 Orlando, FL

Meet the members of MAS



Patricia (Trish) DeSousa is the new Lab Manager for MAS. Trish comes to MAS from Corning, Inc., where she was the lab manager in their Materials Characterization Lab. During her time at Corning, Trish worked with many of the same techniques used by MAS, including pore structure by mercury intrusion, surface area and pore size by gas adsorption, density by gas pycnometry, and particle size by laser light scattering. Trish holds a Master's Degree in Biology from East Stroudsburg University. Her education, management experience, and materials characterization experience are a perfect fit for MAS and we are extremely fortunate to have her as part of our team. Please feel free to contact Trish with any questions; she will be happy to assist in any way possible.

Instrument Training

Micromeritics provides basic start-up training for most instruments during installation. However, when operators wish to maximize their proficiency and broaden their capability with their Micromeritics instrument, more advanced training is needed. To achieve this goal, Micromeritics offers targeted classes for most instruments in which customers may expand their ability and improve their understanding by learning from the experts who designed their instruments. These classes, periodically held at Micromeritics headquarters in suburban Atlanta, Georgia, include:

Theory Overview

Learn about the science upon which each instrument is based and how this science applies to successful sample preparation, analysis, and results interpretation.

Detailed Operations

Effective sample file creation, use of analysis parameters, and manual sample entry are all covered. Increase efficiency and learn to use the full power and flexibility of the operating software.

Automatic Analyses

Develop correct analysis procedures to optimize collection of accurate, reproducible data. Much class time is spent performing hands-on analyses in a controlled, tutorial environment.

System Utilities

Learn instrument software utilities which help manage sample files and directories, protect data, and select system options.

Report Optimization

Learn to configure reports and obtain the most useful information, as well as improve report comprehension.

Troubleshooting

Learn techniques to quickly locate and resolve instrument and software problems.

User Maintenance

Under the guidance of a Micromeritics Maintenance Specialist, practice routine maintenance procedures which improve operation, reduce downtime, and increase data accuracy.

Course Enrollment

Training courses last from two to four days and are designed to provide hands-on, performance-based instrument skills and knowledge. Small classes allow individual instruction and peer interaction. Course materials include a Study Guide, a complimentary copy of *Analytical Methods in Fine Particle Technology*, and a wealth of other educational material. A Certificate of Completion is awarded to each trainee.

Visit www.micromeritics.com, select Service Center, and then Training Center for additional information, to enroll for a specific course, or for a complete course schedule.

Visit our website for the complete 2010 course schedule and registration.
www.micromeritics.com

2010 Training

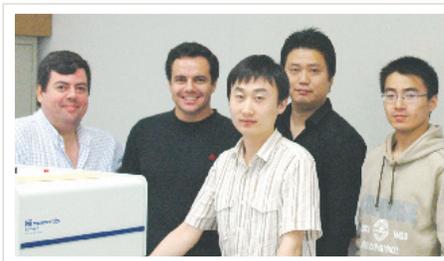
TriStar® II 3020
January 26 - 28

AutoChem™ II 2920
February 9 - 11

Saturn DigiSizer® II
February 23 - 25

Gemini® 2390
March 23 - 24

For additional information or to register for the class of your choice, visit www.micromeritics.com/Service-Center/Training-Class-List.aspx or phone 770.662.3607. Early registration is recommended since class space is limited.



Students attending a recent Elzone course

Events

Pittcon 2010

February 28 - March 5, 2010
Orange County Convention Center
Orlando, FL
Booth #1721

ACS Spring 2010

March 21 - 25, 2010
The Moscone Center
San Francisco, CA
Booth #1116

Analytica 2010

March 23 - 26, 2010
Messe München GmbH
Munich, Germany

Interphex 2010

April 20 - 22, 2010
Jacob K. Javits Convention Center
New York, NY
Booth #426

Visit our website for a complete Event schedule www.micromeritics.com

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Subscriptions are free to particle technology users and can be started simply by writing to The microReport editor.

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Attention Authors

We welcome articles and information concerning particle technology applications performed with Micromeritics instrumentation. Everything from a single plot with operating conditions to an in-depth article on physisorption, chemisorption, etc. with supporting graphs will be considered. If your material is published in The microReport, you will receive a copy of Analytical Methods in Fine Particle Technology by Paul A. Webb and Clyde Orr.

Send your article to:
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Include your title, return address and phone number. Contributions cannot be returned, but each will be acknowledged.

mi micromeritics®

The Science and Technology of Small Particles™

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Micromeritics offers over 50 sales, service, and distribution offices throughout the world. For additional information, a free product demonstration, or the location of the office nearest you, call or write:

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