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New Elzone Combines Modern Technology with Proven Method

Micromeritics is preparing to release our new Elzone® II 5390 particle sizing and counting instrument. The Elzone series is based on the “electrical sensing zone” technique, also known as the “Coulter Principle.” The first instrument of this type was invented in the 1950’s by Wallace H. Coulter, founder of the Coulter Corporation, and it quickly became the preferred method for counting blood cells.



Today, this technique is widely accepted and recognized as a highly effective method of sizing and counting particles composed of a wide variety of organic and inorganic materials. Unlike other particle measurement techniques such as laser light scattering, sedimentation, or image analysis, the electrical sensing zone method can analyze samples of varying optical properties, densities, colors, shapes, and orientations. Micromeritics’ Elzone particle size analyzer uses this powerful particle characterization technique to determine quickly and accurately the size, number, concentration, and mass of a wide variety of finely divided materials. It determines particle diameters from approximately 0.4 μm extending to nearly three orders of magnitude larger – a dynamic size range suitable for a wide variety of industrial, biological, and geological specimens.

An Elzone analysis begins by taking a sample (a dry powder, paste, or emulsion) and dispersing it at low concentration in an electrolyte solution. This dispersion is placed in

a sample cup that contains an electrode. The sealed end of a glass tube is immersed in the dispersion. In the side of this tube and below the liquid surface is a precision hole (orifice) through which the electrolyte is drawn by maintaining a specific pressure differential across the orifice. Within the glass tube is another electrode of opposite polarity from the exterior electrode. A conductive path is formed between the electrodes with current flowing by way of the electrolyte through the orifice. As the electrolyte is pulled through the orifice, so are suspended particles. Each particle while in the orifice displaces a volume of electrolyte equal to its own volume. Displacement of the electrolyte impedes the current flow through the orifice, that is, it increases circuit resistance, decreasing conductivity. This produces an electrical pulse, the amplitude of which is proportional to the particle volume. The Elzone counts the pulses and sorts them by amplitude (volume) and pulse width (related to transit time). This provides data from which are calculated the particle population in a variety of relative and absolute dimensions. A precision metering pump also is available allowing concentration to be determined.

The small diameter of the orifice and its short length mean that it has a small internal volume. If the number concentration of the suspension is considerably less than one particle per volume, then in

the majority of cases, particles pass through the orifice one at a time. Thus, coincidence error is largely eliminated from measurements by the low particle concentration that is required to perform an analysis. However, there remains some finite probability that two or more particles will be within the orifice volume simultaneously. To compensate for these events, coincidence correction algorithms are applied.

The advantage of this particle sizing technique is its high precision and very high resolution, defined as the minimum width of the size increment (class) that can be discriminated. The Elzone produces fidelity of sizes of 95% or better when a narrow span standard material is measured and a comparison is made between the ratio of true width to measured width of the particles. The Elzone is easily calibrated to produce precision and reproducibility of results to within $\pm 2\%$.

The instrument reports data as volume percent, population, or surface area distribution in cumulative or differential form on linear or log scales following the guidelines of International Standards ISO 13319. Plots can be overlaid for comparisons and data from a series of analyses can be reported in Statistical Process Control charts or as 'cause and effect' plots using linear regression to determine what degree of correlation exists between a dimension measured by the instrument

and some external parameter that is entered by the user, for example, grinding time.

A novel feature available with the new Elzone® II 5390 takes advantage of the pulse width data that are recorded with each pulse height data point. The amplitude of the pulse is related to volume, but for a given flow rate and pulse amplitude there may be various pulse widths. One way this may be accounted for is particle shape. Micromeritics' new Elzone presents these data as a three-dimensional plot of particle size, pulse width, and quantity of particles.

Examples of Applications That Benefit from Sizing by Elzone Analyzers

There are a number of materials for which this type of particle size analysis is uniquely suited. For example, today's toner manufacturers have a need to produce microfine toners that have very narrow size distributions, both by volume and population. The Elzone data are crucial in determining to what extent this objective has been met by sizing and counting particles essentially one at a time with the accuracy of electronically based volume displacement technology. In this application, the combination of high resolution and the ability to count makes electrozone sensing a natural choice.

The Elzone has also found a place in the determination of particle-size distributions

of packing materials used for high performance liquid chromatography (HPLC). Both particle size distribution and average particle diameter are required measurements since packing material performance from different lots can vary greatly even though the lots appear to have the same average particle size. Lots are evaluated based only on one of these parameters. For this application, particle size distributions are enhanced by the high-resolution capability of the electrical sensing zone technique allowing subtle shifts in the distribution to be observed.

Geological materials such as soil, silt, and sediments are composed of a variety of components, both organic and inorganic. Accurate sizing by sedimentation requires that the sample be composed of materials of the same density. Light scattering techniques require that all particles have essentially the same optical properties. Both techniques are sensitive to particle shapes that vary greatly from spherical. Sizing by electrical sensing zone is not affected by these characteristics of a sample.

Although the electrical sensing zone technique was introduced to count blood cells, it now provides excellent measurements on a wide array of materials.

Micromeritics' Tony Thornton Appointed Chairman of ISO Sub- Committee on Particle Sizing

Will Assist in the Establishment of U.S. and International Particle Sizing Standards

Micromeritics' Senior Product Scientist Tony Thornton has recently been appointed Chairman of the ISO Technical Committee 24 (Sieves, Sieving and Other Sizing Methods) sub-committee 4 (Sizing by Methods Other Than Sieving). His years of technical contributions and involvement with both ASTM and ISO have distinguished him as a respected expert in the field of particle characterization, and his work has been instrumental in the establishment of standard methods in this field.

Last Spring, Tony received the ASTM Award of Merit for outstanding technical contributions to and masterful leadership of Committees B09 on Metal Powders and Metal Powder Products, and E29 on Particle and Spray Characterization. He has actively and productively participated in several ASTM committees for more than 16 years.

He has also served as liaison among ASTM Committees B09, E29, C21, and C28 all dealing in some capacity with analysis of powder materials, his area of expertise. He has ensured that the standards written by all these committees are technically similar, though dealing with different classes of materials. Tony has conducted and



spoken at numerous technical seminars on powder characterization techniques, imparting information that serves as the basis for ASTM standard test methods and revisions. This information extends to the international arena, serving also as input to ISO standards based on the ASTM versions.

Tony is currently Micromeritics' Senior Product Scientist and Training Manager. He also assists with methods development, applications support, and analyses interpretation.

MIC is proud of Tony's contributions to the field and is fortunate to have his expertise supporting our line of particle sizing products.

Micromeritics Showcases Instruments At the Pittsburgh Conference

Pittcon (February 27 - March 4 in Orlando, Florida) is one of the world's premier annual conferences devoted to laboratory science and instrumentation. Micromeritics, located in booth 1876, will be showcasing several products at this year's exhibition.

Micromeritics' new generation SediGraph™ III 5120 particle size analyzer combines the proven SediGraph analytical technique with advanced instrumentation features to provide superior repeatability, accuracy, and reproducibility. The SediGraph III measures mass by X-ray absorption and particle size by sedimentation - no modeling is required. Complete particle accountability assures that all of the introduced sample is accounted for, even that fraction of the sample that is outside of the measurement range.

New confirm™ Software for the 21 CFR Part 11 environment is now offered with a Micromeritics suite of products that includes: the Saturn DigiSizer® 5200 High Resolution Laser Particle Size Analyzer, ASAP™ 2020 Accelerated Surface Area and Porosimetry System, TriStar™ 3000 Surface Area

and Porosimetry Analyzer, the Gemini™ V Series Surface Area Analyzers, and the Elzone® II 5390 particle size analyzers.

Combined with Micromeritics' IQ and OQ services, the user can be assured that each system is validated for accuracy, reliability, consistent with intended performance, and provides safeguards to protect the integrity of analysis records.

The Gemini V Series is ideal for measuring the surface area and porosity of a wide variety of materials. The Gemini 2365 determines single-point and multipoint BET and Langmuir surface areas, total pore volume, and micropore analysis by the t-method. The capability to determine statistical thickness surface area (STSA) of carbon blacks is also included. The Gemini 2380 can additionally provide BJH pore volume distributions.

The new Elzone® II 5390 utilizes the electrical sensing zone method to analyze samples without regard to optical properties, densities, colors, and shapes. The Elzone quickly and accurately determines the size, number, concentra-

tion, and mass of a wide variety of organic and inorganic materials. Automated features include: start-up, run, and shut-down routines; blockage detection and clearing; flushing/rinsing; and calibration. A high level of accuracy and resolution, speed, ease-of-use, and compact size make the Elzone equally suitable for industry, quality control, and research and development laboratories.

Perform Longer Surface Area Analysis with the Gemini V 2385C

The Micromeritics Gemini V Series of surface area analyzers rapidly and reliably produces accurate and repeatable surface area and porosity results. Their simplicity of use, reliability, and ruggedness have earned the Gemini a place in laboratories worldwide as an essential tool in research and quality control environments. Some of our customers have expressed an interest in an analytical instrument that permits longer analysis times with more thermal stability; to meet their needs, we have developed a new model of Gemini.

The new Gemini V 2385C has the same unique capabilities as other Gemini models. It employs the same innovative use of twin sample and balance tubes. These tubes are identical in every aspect, including their thermal environment, since both tubes share the same cryogenic bath during analysis. Pressures in both tubes follow the same pressure table as well. Therefore, conditions within one tube are exactly reproduced within the other. Each tube is connected by a valve to a precision reservoir that supplies pressurized analysis gas as needed. The sample and reference

reservoirs, like their associated tubes, are physically identical and are initially charged to the same pressure. During an analysis, differential pressure between reservoirs is monitored. This common mode technique assures that any differential pressure is solely due to adsorption by the sample and not to factors that lead to variations in free space during an analysis.

This new model of Gemini has a larger cabinet to facilitate longer sample tubes and a larger Dewar. This allows for a much longer analysis time than a standard Gemini V, and assures a more stable thermal environment in which to measure the isotherm. This improves the repeatability from one sample to the next.

In addition to the enlarged cryogen system, the new Gemini has the capability to monitor continuously the saturation vapor (P_0) of the adsorptive using a dedicated P_0 tube and transducer. Continuously monitoring the P_0 allows the instrument software to detect and accommodate at each data point any minute change in P_0 that may occur during the course of analysis. This achieves a more accurate determination of relative pressure.



The Gemini V 2385C is ideal for any industry that requires longer analysis times or more precise data on their sample analyses. Carbon black manufacturers will find this Gemini model extremely useful.

Gemini V 2385C Features

- Automatically generated pressure tables with user-selected end points
- User-defined pressure table
- Free-space determination and correction options:
 - * Measure
 - * Calculate
 - * Use previous value
 - * No correction

Micromeritics Partners with MVA Scientific Consultants for Microscopy Services



Micromeritics Analytical Services and MVA Scientific Consultants are pleased to announce that they have joined forces to offer their clients additional analytical choices while improving customer service.

“Micromeritics Analytical Services’ goal is to improve continually and expand our product offering to meet our customers’ changing needs,” says Greg Thiele, Business Manager for Micromeritics Analytical Services. “We are a lab-service industry leader because we provide exceptional service, highest quality results, useful data interpretation, and quick turn-around times at a fair price. Our strategic alliance with MVA Scientific Consultants further demonstrates our commitment to serving our customers’ analytical needs.”

MVA Scientific Consultants will provide electron and light microscopy services, including SEM, TEM, Scanning White Light Microscopy, Polarized Light Microscopy, and FTIR. MVA’s staff of world-class specialists offers over 100 years of combined experience in particle identification and source determination, materials characterization, and

surface metrology. Clients from across the world have sought out the firm to help address unique challenges involving QA/QC, litigation, industrial hygiene, environmental forensics and a host of other issues.

“At MVA Scientific Consultants, our mission is to meet and exceed our clients’ expectations through detailed innovation, integrity, and value-added interpretative services,” states Rich Brown, Executive Principal. “Working with Micromeritics Analytical Services is in keeping with that mission and will be a benefit to our clients.”

For additional information on MVA Scientific Consultants, please visit: www.mvainc.com.



OTHER NEWS AT MAS

DEA License

We are pleased to announce that Micromeritics Analytical Services is licensed by the U.S. Drug Enforcement Agency to provide contract analytical services for controlled substances, schedules 2, 3, 4, and 5.

Successful Audit Completed

To ensure cGMP/GLP compliance, our lab was audited by an independent expert witness. Our lab was found to be compliant to all relevant sections of 21 CFR Part 11, current Good Manufacturing Practices, and Good Laboratory Practices. Copies of this audit are available upon request.

New Services Offered

Micromeritics Analytical Services is also pleased to offer calibration services for mercury porosimeter penetrometers and Tristar 3000 sample tubes.

For more information or if you would like to submit samples to our lab, please contact us: email: mas@micromeritics.com
Phone: 770-662-3630, or www.particletesting.com

Micromeritics Instrument Training Courses

Training is provided for most Micromeritics instrumentation at the time of installation. This training presents all the information required for a new operator to become quickly proficient in operating the instrument. In cases where personnel changes occur or more advanced training is required, Micromeritics conducts a variety of classes for many of our instruments. These courses are held at our headquarters in suburban Atlanta, Georgia. The courses include:

Detailed Operational Procedures

Items covered are effective sample file creation, use of analysis parameters, and manual sample entry. You'll learn how to utilize the full power and flexibility of the operating software.

Automatic Analysis

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

Systems Utilities

Discover all of the instrument software utilities which help you manage sample information files and directories, protect data, and select system options.

Troubleshooting

Learn techniques that enable you quickly to locate and resolve difficulties.

Report Generation and Comprehension

Learn to configure reports and obtain more useful information, as well as improve comprehension of the reports produced.

User Maintenance

Practice routine maintenance procedures which improve operation, reduce downtime, and increase data accuracy.

Theory Overview

Learn about the scientific theory upon which each instrument is based and how it applies to the critical factors relevant to successful sample preparation and analysis performance.

Enrollment

Training courses last from 2 to 3 days and are designed to provide hands-on, performance-based instrument knowledge. Small classes guarantee close individual attention. Included in the course materials are a Study Guide, an instrument Operator's Manual, and other handout materials. Certificates of Completion are also awarded to all trainees.

Training 2005

Saturn DigiSizer® 5200
8/16 - 8/18

SediGraph 5120
8/23 - 8/25

AutoPore IV
8/30 - 9/1

TriStar 3000
3/15 - 3/17, 10/11 - 10/13

Gemini V
3/22 - 3/23, 10/4 - 10/5

ASAP 2020 Physi
4/5 - 4/7, 11/8 - 11/10

ASAP 2020 Chemi
4/12 - 4/14, 11/15 - 11/17

AutoChem II
5/3 - 5/5, 12/6 - 12/8

Elzone II 5390
6/7 - 6/9, 12/13 - 12/15

For additional information or to register for the class of your choice, contact the Micromeritics Training Department at 770.662.3607. Early registration is recommended since class space is limited.

Events

Pittcon 2005

February 27 - March 4
Orange County Convention Center
Orlando, FL

Laborama

March 8
Tennis club du Parc
Louvain-La Neuve, Belgium

ACS Spring Exposition

March 13 - 17
San Diego Convention Center
San Diego, CA

107th Annual Exposition of American Ceramic Society

April 11 - 12
Baltimore Marriott Waterfront
Baltimore, MD

Interphex 2005

April 26 - 28
Javits Convention Center
New York, NY

19th NAM

May 23 - 26
Wyndham Franklin Plaza Hotel
Philadelphia, PA

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 plots 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.

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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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