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## **Introducing the New ASAP 2020 Accelerated Surface Area and Porosimetry System**

Accurate and reproducible surface area and porosimetry measurements are essential to the determination of the effectiveness, performance, and quality of a wide variety of materials. The new ASAP 2020 Accelerated Surface Area and Porosimetry system provides extended capabilities for producing high-quality surface area and porosimetry information for development, production monitoring, and quality control of materials such as pharmaceuticals, ceramics, carbons, catalysts, paint & coatings, propellants, electronics, cosmetics, and aerospace materials.

### **ASAP 2020 Advancements**

The ASAP 2020 utilizes two independent vacuum systems, one for the analytical system and one for the sample preparation system. This proven design allows sample preparation and analysis to proceed concurrently and eliminates the possibility of cross-contamination between the sample analysis and preparation.

The ASAP 2020 retains the stainless-steel monolithic analysis manifold system of its ASAP 2010 predecessor and features an additional manifold that automatically controls up to six analysis gases. The Chemisorption option includes a high temperature oven, flow control, and six additional gas inlets for a total of twelve gas inlet ports. Also as with the original ASAP 2010, there are independent ports included for free-space gas. Further, the ASAP 2020 includes an independent inlet port specifically designed for vapor



adsorptives as well as a port to extract analysis gas for examination by an optional mass spectrometer. An optional dry (oil-free) vacuum system is available for optimum MS performance.

The optimized manifold design assures efficient evacuation, highest possible stability, and precise dosing while maximizing protection against manifold contamination. Together, these features combine to assure extremely accurate measurements of adsorbed gas volumes.

Long-duration dewars (typically greater than 72 hours) combine with Micromeritics' patented Isothermal Jackets to maintain a constant thermal profile along the sample and the saturation pressure ( $P_0$ ) tubes throughout extended analyses. (Virtually unlimited analysis time is possible by refilling the dewar during analysis.)

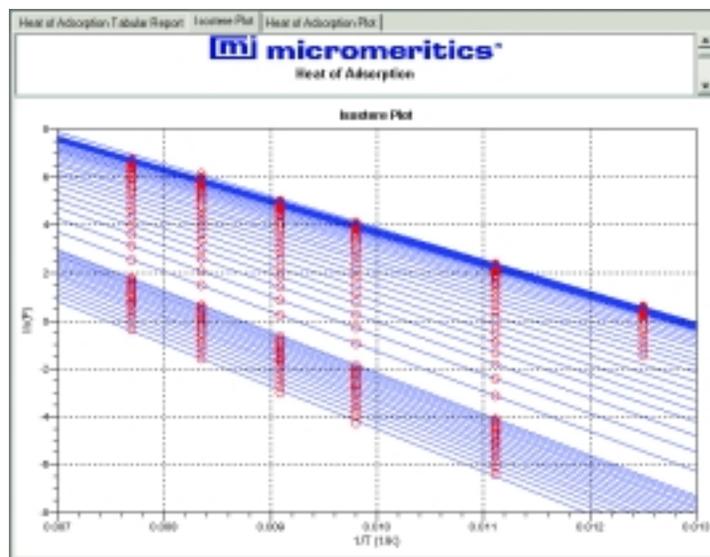
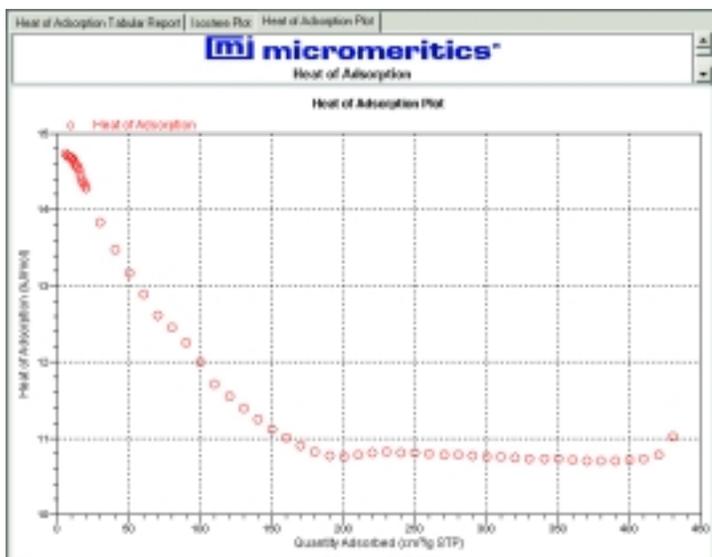
(ASAP 2020 Cont.)

## Available Options

Some of the new options available with the ASAP 2020 include an Enhanced Chemical Resistance Option for use with aggressive gases or vapors as the adsorptive, a water vapor reservoir accessory for use with water vapor experiments, a dry vacuum system for use with a mass spectrometer; and a stand-alone sample preparation system.

## Enhanced Software Features

- The ASAP 2020 uses a Windows® interface that includes wizards and applications to help plan, launch, and control the analysis.
- You can collect, organize, and reduce raw data, and store standardized sample information and analysis conditions for easy access during later applications.
- Advanced capabilities include cut-and-paste graphics, scalable-and-editable graphs, and customizable reports. The graphics module can display up to nine overlaid graphs.
- Degas temperature profiles and treatment time data are integrated with the sample file for future reference and verification of SOP compliance.
- Exportable data tables provide for merging and comparing data from other sources in a unified single spreadsheet file.
- The user can enter any reference isotherm into the system so it can be used in place of the pre-programmed thickness curves when calculating thickness for t-Plots,  $\alpha_S$  (Alpha-S) Plots, and BJH pore size distribution.
- Additional isotherm models are included, including Freundlich, Temkin, and Langmuir.
- Harkins-Jura, Halsey, and Broekhoff-de Boer thickness models are also available.
- Isostatic heat of adsorption is automatically calculated.
- Crystallite size may be determined with the Chemisorption option.
- ISO and IUPAC standard units are user-selectable.

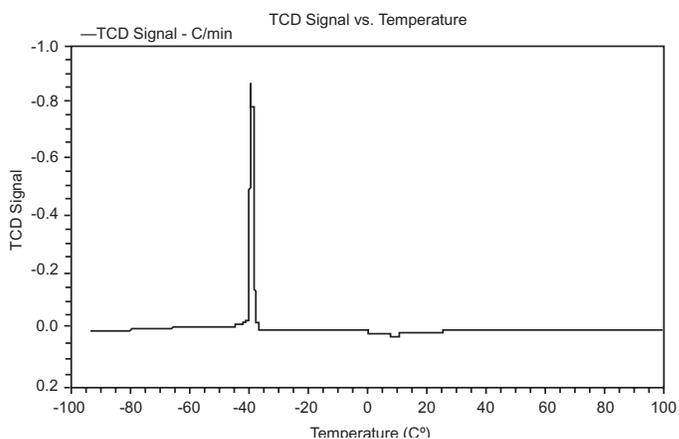


The isosteric heat of adsorption report uses classic thermodynamics to determine the heterogeneity of a surface. This full-featured report displays the adsorption isotherms and the heat of adsorption as a function of surface coverage.

## Newly Designed Micromeritics CryoCooler™ II Accessory

Micromeritics' all new CryoCooler II accessory for the AutoChem models 2910 and 2920 provides excellent temperature control for fully automated temperature programmed reduction, desorption, and reaction analysis. Subambient analyses are essential for characterizing the reduction of noble metals. These techniques extend the wide range of applications that can be developed using the AutoChem Series. The development of fuel cell technology that require platinum and ruthenium catalysts makes subambient analysis an essential part of the characterization program. Both Pt and Ru are reduced in the subambient to ambient temperature range. The Cryocooler II combined with the AutoChem Series is an integrated environment for automatically characterizing the systems. The subambient temperature option can be programmed to control temperatures from  $-120^{\circ}\text{C}$  to  $1100^{\circ}\text{C}$ .

Control of the CryoCooler II is integrated into the instrument control software. Both negative and positive temperature ramps in the sub-ambient range is selectable in  $1^{\circ}\text{C}/\text{min}$  increments over a range of  $5^{\circ}\text{C}/\text{min}$  to  $50^{\circ}\text{C}/\text{min}$ .



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# Interlaboratory Comparison of Analytical Results a Measure of Quality Control

By Eng<sup>a</sup> Conceição Fonseca, Dra. Rosário Amaral and Rui Lucas of CTCV – Technological Centre of Ceramic and Glass – Coimbra - Portugal

An **Interlaboratory Comparison** is an external way of assuring quality control among laboratories. It allows the participants to detect unsuspected errors and deficiencies in their methodology. Recently, the Technological Centre of Ceramic and Glass (CTCV) in Portugal conducted an interlaboratory comparison among eight laboratories that employ the granulometric analysis technique. Each laboratory was asked to submit results of analysis of silica flour performed with the Micromeritics SediGraph™ 5100. The results were calculated by CTCV according to ISO 5725 – Part 2 standard, and the z-score to determine the testing performance of each participant laboratory.

**Granulometric analysis** is a technique for characterizing pulverized materials and is applied to the determination of the particle size distribution of a wide range of materials. Granulometric analysis by X-ray sedimentation is a technique based on the measurement of the sedimentation velocity of particles dispersed in a fluid that allows, by Stokes' law, the calculation of the diameter of the sphere with the same sedimentation speed of the particle. What is obtained is not a geometrical diameter, rather a hydrodynamical or equivalent spherical diameter.

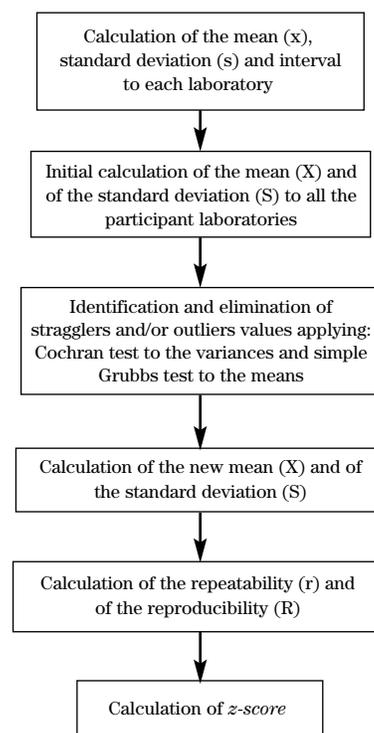
This is known as Stokes diameter, which is the diameter of a sphere whose characteristic property has the same value as that of the particle being tested. [1]

The equipment used in this interlaboratory comparison was Micromeritics' SediGraph 5100. The sedimentation technique using the SediGraph is an incremental technique and is based in the study of the concentration change with time in a zone of measurement described by its depth in the sedimentation cell. At time zero, the suspension is homogeneous and the concentration is 100%. At any time later, the relative concentration (when compared with the concentration at time zero) is equal to the cumulative mass fraction smaller than the Stokes diameter calculated for the smallest particle that could have settled below the measurement zone.

The measure of the mass distribution of particle size in the cell containing the suspension is determined using a source of X-Ray of low energy and a suitable detector. The X-Ray source and the detector remain stationary while the cell moves vertically between them subjecting different measuring zones to the X-Ray.

## Methodology of Calculation

The calculation method used to measure the results of the participant laboratories is indicated in the following flowchart (to each parameter analyzed):



The z-score measures the deviation of the result of each laboratory from the “true” value, by comparison with a reference standard deviation and is given by the formula:

$$Z = \frac{x - X}{S} \quad (1)$$

where:

$x$  = mean value of the results of the participant laboratory

$X$  = value assumed to be true

(it employed the mean of the results of all participants, after excluding the outlier values.)

$s$  = standard deviation of the mean of the results, after exclusion of the stragglers and/or outliers values

The interpretation for the z-score is the following:

$ Z  \leq 2$	Gratifying results
$2 <  Z  \leq 3$	Questionable results
$ Z  \geq 3$	Degratifying results

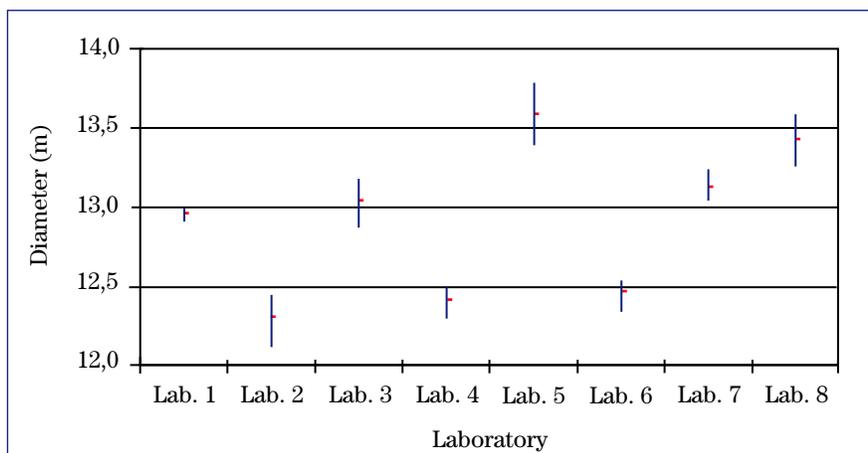


Figure 1 – Interval of variation for the MEDIAN parameter

## Presentation of Results

The parameters analyzed were median (diameter to 50%), d 95%, d 90%, d 75%, d 25%, d 10% and mode.

In the presentation of statistical analyses of test results that follow, all parameters, except the calculation of the z-score, are presented. Z-score is calculated only for the test medians.

## Elimination of Outlier Values

The elimination of outlier values (values appear anomalous compared to other observations in the set) was done by application of the Cochran test to the variances and by application of the simple Grubbs test to the means. The application of the Cochran test leads to the elimination of the outlier values presented in Table 2.

Table 1 – MEDIAN Parameter ( $\mu\text{m}$ )

Laboratory	$X_1$	$X_2$	$X_3$	$X_4$	$X_5$	Mean (x)	Standard Deviation(s)	Interval
1	12,92	12,94	12,99	12,97	12,97	12,96	0,03	12,92 12,99
2	12,45	12,38	12,42	12,15	12,13	12,31	0,15	12,13 12,45
3	12,88	13,16	12,91	13,07	13,18	13,04	0,14	12,88 13,18
4	12,31	12,46	12,49	12,39	12,47	12,42	0,07	12,31 12,49
5	13,40	13,79	13,62	13,58	13,55	13,59	0,14	13,40 13,79
6	12,54	12,54	12,34	12,45	12,46	12,47	0,08	12,34 12,54
7	13,07	13,08	13,24	13,19	13,05	13,13	0,08	13,05 13,24
8	13,57	13,35	13,26	13,59	13,36	13,43	0,15	13,26 13,59

Mean (X): 12,92

Standard Deviation (S) : 0,48

The application of the simple Grubbs test did not lead to the elimination of outliers. The outlier values were retained in the following calculations.

### Calculation of Repeatability and Reproducibility

The values of repeatability (r) and reproducibility (R) comprise Table 3.

**Table 2 – Stragglers and Outliers Values – Cochran test applied to variances**

Parameter measured	Straggler value	NIL – N° ID Lab.	Outlier value	NIL – N° ID Lab.
MEDIANAd 50 (µm)	--	--	--	--
MODE	--	--	--	--
d 95 (µm)	--	--	1**	Lab. 3
d 90 (µm)	1*	Lab. 3	--	--
d 75 (µm)	--	--	--	--
d 25 (µm)	1* + 1*	Lab. 2 + Lab. 8	--	--
d 10 (µm)	--	--	--	--

Legend: 1\* - Straggler value    1\*\* - Outlier value

**Table 3 – Repeatability and Reproducibility**

Parameter measured	s <sup>2</sup> r	s <sup>2</sup> L	s <sup>2</sup> R	r	R
MEDIAN (µm)	0,013	0,224	0,237	0,32	1,36
MODE (µm)	1,373	0,312	1,685	3,28	3,63
d 95 (µm)	0,283	0,728	1,011	1,49	2,82
d 90 (µm)	0,087	0,521	0,609	0,83	2,18
d 75 (µm)	0,026	0,308	0,334	0,45	1,62
d 25 (µm)	0,003	0,221	0,225	0,16	1,33
d 10 (µm)	0,007	0,332	0,339	0,23	1,63

Where:

- s<sup>2</sup>r: Repeatability Variance
- s<sup>2</sup>L: Between – Laboratory Variance
- s<sup>2</sup>R: Reproducibility Variance
- r: Repeatability
- R: Reproducibility

The values of repeatability and reproducibility presented were calculated using a factor of 2, 8, for n=2.

## Calculation of Z – score

The values of z-score to the Median ( $\mu\text{m}$ ) parameter make up Table 4 and are presented in Figure 2.

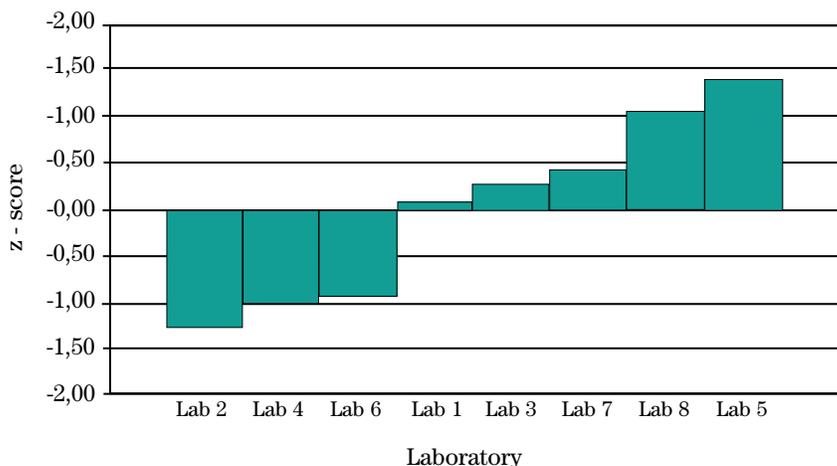
**Table 4 – Z-score - MEDIAN Parameter ( $\mu\text{m}$ )**

NIL – N° ID Laboratory	Mean (x)	Standard Deviation (s)	$n_i$	Z - score
Laboratory 1	12,96	0,03	5	0,08
Laboratory 2	12,31	0,15	5	- 1,28
Laboratory 3	13,04	0,14	5	0,25
Laboratory 4	12,42	0,07	5	- 1,05
Laboratory 5	13,59	0,14	5	1,40
Laboratory 6	12,47	0,08	5	- 0,94
Laboratory 7	13,13	0,08	5	0,44
Laboratory 8	13,43	0,15	5	1,07

Mean (X): 12,92

Standard Deviation (S): 0,48

**Figure 2 – Z-score – MEDIAN ( $\mu\text{m}$ )**



## Final Considerations

- The ‘true’ value, as employed in the calculations, was calculated using the mean of the results of all the participants, after excluding outlier values. The value adopted as ‘true’ refers to results obtained with the SediGraph 5100 applying the test procedures to the sample analyzed.
- The values of repeatability and reproducibility represent the repeatability and reproducibility of the test method, the test conditions, and the sample employed.
- The absolute difference between two results determined in repeatability conditions must not exceed the value of r (repeatability).
- The absolute difference between two results determined in reproducibility conditions must not exceed the value of R (reproducibility).

CTCV wishes to express recognition and to thank the contribution given by IPN – Labgran, and Laboratories of Cerisol, Comital, Lusoceram, Onya Mineral Portuguesa, Sanitana e Somincor in this interlaboratory comparison of analytical results.

## REFERENCES

- [1] F. M. Barreiros, P. J. Ferreira, M. G. Rasteiro, and M. M. Figueiredo “Granulometric Analysis,” Chemical Engineering Department of Coimbra University
- [2] International Standard Organization: Accuracy (trueness and precision) of measurement methods and results, ISO 5725: Part 2: Basic methods for the determination of repeatability and reproducibility of a standard measurement method: 1994

# Micromeritics Launches New Website

Micromeritics has recently redesigned its website! The new site includes a number of features to make it easy for you to get the information you are seeking.

Dynamic pull-out menus allow you to navigate the site easily and quickly and get the information you are looking for. You can find detailed information about our products, our company, and the services we provide. You can submit questions to our service staff and order accessory parts for your Micromeritics instrument. New application notes and technical articles have been added, and others will be continually added on a regular basis.

Software updates are available for download. The latest Material Data Safety Sheets (MSDS) are accessible, as well as information about service agreements and training courses.

You can sign up to get the electronic version of the MicroReport, as well as updates from Micromeritics.

The website is a resource that provides information about our company, our products, and the services we offer. Please visit [www.micromeritics.com](http://www.micromeritics.com) or [www.smallparticles.com](http://www.smallparticles.com) to explore the new site.



## Micromeritics' Materials Analysis Laboratory: Helping to Increase Your Productivity

Micromeritics Materials Analysis Laboratory provides testing that includes measurements of particle size, surface area, porosimetry, chemisorption, density, and zeta potential. We can help you to build competitive superiority, reduce process expenditures, and solve problems for a wide range of industries, as well as assist you in developing new products. Micromeritics-manufactured testing instruments operated by our expert laboratory scientists combine to assure you of the highest data quality possible.

Our staff includes Ph.D. and Masters-level research associates who have extensive experience in the areas of physical adsorption, chemical adsorption, particle sizing, and materials sciences, to help you find the best possible analytical solution for your specific application. If your lab is overworked, or if you need answers that your existing instrumentation just can't provide, give us a call and let our Materials Analysis Lab provide you with fast, cost-effective solutions for your particle characterization requirements.

For more information about services offered by the Materials Analysis Laboratory, contact Ron Holt at 770-662-3634.

## Micromeritics Announces New Vice President of Marketing

Norcross, GA – January 29, 2003—Micromeritics is pleased to announce that Dr. Timothy J. Alavosus has assumed the responsibilities of Vice President of Marketing at our corporate headquarters in Norcross (Atlanta), Georgia. Dr. Alavosus has over 14 years combined experience in marketing and product management in the life science and laboratory instrumentation industries, and will direct Micromeritics' sales, marketing, service, and applications efforts worldwide.

Dr. Alavosus will lead Micromeritics' continued expansion as a highly market-driven company in order to better the needs of customers worldwide, while at the same time pursuing advanced, new instrumentation for existing and growth markets. "I am very excited about the opportunity presented to me by Micromeritics," said Dr. Alavosus. "The Company has been a leader in the particle characterization business for over 40 years, and with our new market-driven corporate culture, we will be developing new products, services, and strategic business alliances that will help us to continue pioneering the next generation of instrumentation for science and industry."

Since joining Micromeritics in 2002, Dr. Alavosus has led a marketing renaissance of the Company that includes all-new corporate logos, instrument colors and designs, and a completely reworked corporate image. This transformation will be in the spotlight at the upcoming Pittsburgh Conference and Exhibition beginning March 10 in Orlando, Florida.

*During peak workloads or when you are in need of a particular piece of equipment, call on Micromeritics*

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- *Chemisorption*
- *Pore Structure*
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- *Micropore Structure and Volume Distribution*
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For more information, call us at  
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# VISIT US AT PITTCON 2003

*March 10-13, Orlando, Florida*

Micromeritics will be introducing several new products at The Pittsburgh Conference and Exhibition. Pittcon is the world's premier annual conference devoted to laboratory science and instrumentation. The conference attracts 25,000-35,000 conferees and exhibitors from more than 120 countries.

Come learn about these great new products:

## The New ASAP™ 2020 Accelerated Surface Area and Porosimetry System

This new instrument is an essential tool for providing surface area and porosity measurements for a broad range of applications such as pharmaceuticals, ceramics, carbons, catalysts, paint & coatings, propellants, electronics, cosmetics, and aerospace materials.



## The Saturn DigiSizer® 5200 Particle Size Analyzer New Low-Volume Liquid Sample Handling Unit

Micromeritics now offers the new, patented Low-Volume Liquid Sample Handling Unit for use with the Saturn DigiSizer 5200. This new low-volume system requires only 1/5 or less the liquid volume of a "standard" system to carry the sample (approximately 100 mL).



## The New AquaPrep™

The AquaPrep helps to eliminate air bubbles during aqueous particle size analysis. Bubbles in the dispersing liquid can often result in misleading or incorrect data when performing particle size analysis.



## The New CryoCooler™ II

The newly designed CryoCooler accessory for the AutoChem models 2910 and 2920 provides excellent control of subambient analysis temperature in the -100 to 20 °C range. This type of accurate, variable temperature analysis can be of considerable value in the analysis of many materials, including catalysts.

## IMPORTANT SOFTWARE ANNOUNCEMENT!

Micromeritics will be making an important new software announcement at PittCon. Be sure and stop by the booth to learn more!

## ASAP 2020 Paper at Pittcon

Dr. Jeff Kenvin, Senior Scientist at Micromeritics will be giving a paper on the new ASAP 2020. Session 12 - Forum 12 - Sensor Technology, Sunday, March 09, 2003, 01:20 PM.

Visit us at booth 3568 for more information about these and other products.

## 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 begin quickly proficient operation. However in some 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 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.

### Report Generation and Comprehension

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

### User Maintenance

Practice routine maintenance procedures which improve operation, reduce down-time, and increase data accuracy.

### Troubleshooting

Learn techniques that enable you to quickly locate and resolve instrument problems. Again, emphasis is placed on practicing these techniques on instruments in the Micromeritics Training Laboratory.

### 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.

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

### Cocoa Beach Conference and Exposition (Ceramics)

January 26, 2003 - January 31, 2003  
Hilton Cocoa Beach  
Cocoa Beach, FL

### Western Coatings Society Symposium

February 17, 2003 - February 19, 2003  
Nugget Resort Hotel  
Sparks, NV

### Pittcon 2003

March 10, 2003 - March 13, 2003  
Orange County Convention Center  
Orlando, FL

### American Chemical Society 2003

March 24, 2003 - March 26, 2003  
Morial Convention Center  
New Orleans, LA

### Interphex

March 31, 2003 - April 2, 2003  
Jacob K. Javits Convention Center  
New York, NY

### AAPS Workshop on Particle Size Analysis

April 30, 2003 - May 2, 2003  
Crystal Gateway Marriott  
Arlington, VA

## Training

### AutoChem II 2920

April 22-24, September 9-11

### Gemini

March 18-19, September 16-17

### SediGraph 5100

April 1-3, November 4-6

### AutoPore IV

April 8-10, October 7-9

### ASAP 2020 Chemisorption

June 10-12, November 11-13

### ASAP 2020 Physisorption

June 3-5, November 11-13

### TriStar 3000

March 25-27, August 26-28

## 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.

All text should be typed and limited to 1500 words. Plots, graphs, and diagrams should be clear and legible.

Laurel Whitmire, Editor

The microReport

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

## How To Reach Us

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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## THE microReport

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