THE microReport
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The Science and Technology of Small Particles™
MICROMERITICS INSTRUMENT CORPORATION
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In This Issue

CERTIFIED ISO9001 MANUFACTURER NORCROSS, GA

www.micromeritics.com

New Gemini V Series

Micromeritics is pleased to introduce the new Gemini V Series surface area and porosity analyzers. The new Gemini offers an extended range of capabilities, while retaining elements of the successful design of the previous series of Gemini products.

The Micromeritics Gemini V series rapidly and reliably produces accurate and repeatable surface area and porosity determinations to assure that you have reliable information about these important characteristics of your sample material. Its simplicity of use, reliability, and ruggedness has earned the Gemini its place in laboratories worldwide as an essential tool in both research and quality control environments.

The new Gemini V analyzers retain the immensely successful, patented twin-tube design of the previous series of Gemini products, which assures fast and accurate dosing of the adsorptive gas without pressure overshoot. This is paired with the extended capabilities of the embedded software and a powerful new Windows® control, data archiving, and reporting software option.

Design Features

The secret of Gemini's unique capabilities lies in the sample and balance tube design. The sample and balance tubes are physically identical, as is the thermal environment in which they reside since the sample and balance tubes share the same cryogenic bath. Conditions within each tube exactly reproduce the conditions within the other, the only difference being associated with the presence of the sample in the sample tube. This common mode technique means that any changes observed in the otherwise-balanced system are due entirely to the sample itself. Free-space errors introduced by thermal gradient variations are canceled because the balance tube essentially has the same free-space variation as the sample tube.

Accelerated dosing of the analysis gas

In a traditional static volumetric analysis system, the analysis gas is dosed onto the sample in predetermined quantities. In the unique Gemini design, the sample uptake rate controls the rate at which the gas is delivered, therefore the adsorptive is delivered as fast as the sample can adsorb it. In this manner of dosing, there is no under-dosing in which the sample waits for more adsorptive, nor over-dosing in which case the target pressure is exceeded. The result is a surface area analyzer that is extremely fast and retains accuracy and reproducibility.

Two categories of software control

Embedded software: The Gemini V series continues to be a stand-alone physical adsorption analyzer. The Gemini V series offers two versions of embedded software that allow the unit to be operated without need for an external PC. The Gemini V embedded software provides a wider range of analysis choices and data reduction capabilities than in previous Gemini models.

Intuitive and powerful Windows-based software: The optional Windows-based software allows the Gemini V series to be controlled from a PC, thus providing more versatility in data archiving, networking, and printer options. However, the most powerful features of this software are found in its expanded range of data reduction and reporting.

Gemini Models

There are two models of The Gemini available so you get the analyzer that best suits your specific needs. The Gemini 2365 determines single-point and multipoint BET and Langmuir surface areas, total pore volume, and micropore analysis by the t-method. The Gemini 2380 has all the abilities of the Gemini 2365 and additionally can provide BJH pore volume distributions. Up to one thousand isotherm data points may be collected.

Operating Software

Gemini analyzers can be operated from a keypad or, optionally, from a computer. Using computer control, you can operate up to four Geminis simultaneously from a single computer.

The Gemini V series provides the following reports:

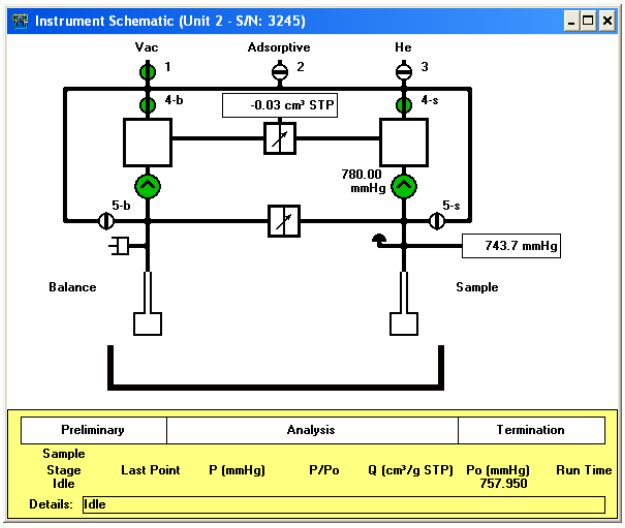
- Adsorption isotherm
- Automatically generated and user-defined pressure table
- Single and multipoint BET surface area

2380 Model

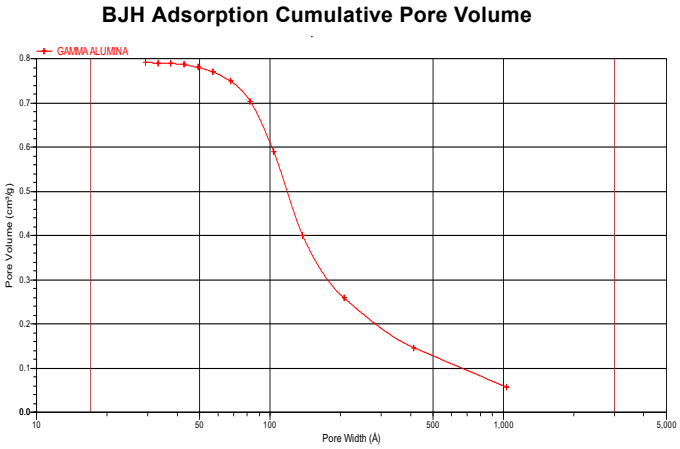
- Desorption isotherm
- Langmuir surface area
- Total pore volume
- t-plot method
- Thickness curves
  - Halsey
  - Harkins-Jura
  - Carbon Black STSA
  - Broekhoff-de Boer
  - User defined isotherm
- BJH pore size distribution using adsorption or desorption isotherm
- Analysis log

Windows Option

Gemini software utilizes a Windows interface to help plan, launch, and control the analysis. You can collect, organize, archive and reduce raw data, and store standardized sample information and analysis conditions for easy access during later applications. Finished reports may be generated to screen, paper, or data transfer channels. Features include cut-and-paste graphics and tables,



With the Windows option the schematic screen shows in detail the current status of the instrument and the analysis in progress.



Example of pore volume distribution derived from adsorpton isotherm

scalable-and-editable graphs, customizable report formats, and the ability to convert StarDriver files.

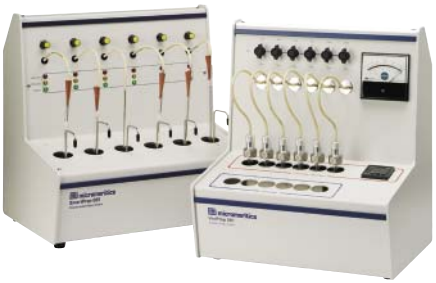
21 CFR Part 11 Option

Also available is ***confirm***™ software, which addresses the many requirements specified by 21 CFR Part 11 validation, security, audit trails, reporting, and more. For more information, visit our website at www.micromeritics.com.

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 He, N_2, Ar, and other non-corrosive gases.

The **SmartPrep™ 065** is a flowing-gas degassing unit which removes adsorbed contaminants from the surface and pores of your 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 easy reference in the future.



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 a 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.

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. You can also choose the StepPrep method. This method, which is best for removing water vapor from certain samples, allows repeated evacuate-backfill-evacuate cycles. 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.

Ensuring Quality in Particle Size Distribution Analysis

By Anthony Thornton, Senior Product Scientist, Micromeritics Instrument Corp., Norcross, Ga.
First Published in Ceramic Industry, April 2003

Quality departments and laboratories use a variety of particle size analyzers and analytical techniques to determine millions of particle size distributions per year. This information is a key indicator of a material's quality—knowing the particle size distribution enables a material user to predict how that material will behave in the manufacturing process. However, for this information to be considered reliable, the material user must have confidence in the quality of the analysis results.

Defining Accuracy

How is the quality of an analysis technique measured? First, we need to define some terms relating to quality. Everyone strives for "accurate" analyses. However, no specific definition exists for the accuracy of many particle sizing techniques due to the lack of a well-defined, universally accepted standard.

Many techniques measure a size-related physical property or phenomenon related to the particle behavior under defined conditions, but these measured properties or phenomena are often influenced by non-size-related particle characteristics, such as shape or porosity. To achieve reliable analyses, we must speak in terms of "agreement," or relative accuracy, rather than absolute accuracy. This leads to the need for standard reference materials (SRM), where a national standard group has certified the particle size distribution of a particular lot of material determined under specified analysis conditions using a given technique. Such materials form the basis for relative accuracy. If a selected particle size analyzer can produce the specified result for a given SRM, we would expect the particle size distribution produced for a test sample using the same analyzer to be accurate, assuming that the sample is properly prepared and that the analyzer is set up and operated properly. In short, the instrument does not know which sample is being analyzed; however, if it is working properly for the SRM, it should work for the test sample.

Unfortunately, the number of certified standard reference materials available is quite small, the quantity of each is limited, and they are typically expensive. However, we can develop secondary standards that are traceable to these primary national standards through controlled inter-laboratory testing using instruments that have been certified using a national SRM. As an example, Table 1 contains the specifications for a garnet traceable secondary reference material qualified using laser particle size distribution analysis.

Ensuring Precision

SRMs and secondary reference standards can be used to ensure the relative accuracy of

Table 1. Specifications for a traceable garnet reference material.

Statistic	Central Value	Tolerances
Mean Diameter	5.30 µm	± 0.12 µm
Median Diameter (50th Percentile)	4.85 µm	± 0.10 µm
90th Percentile	9.94 µm	± 0.30 µm
10th Percentile	1.210 µm	± 0.82 µm
Cumulative Volume Finer than 10.0 µm	90.2%	± 1.0%
Cumulative Volume Finer than 8.0 µm	79.5%	± 1.0%
Cumulative Volume Finer than 6.7 µm	69.1%	± 1.0%
Cumulative Volume Finer than 5.7 µm	59.3%	± 1.0%
Cumulative Volume Finer than 4.8 µm	49.4%	± 1.0%
Cumulative Volume Finer than 3.9 µm	39.2%	± 1.0%
Cumulative Volume Finer than 3.0 µm	9.4%	± 1.0%
Cumulative Volume Finer than 2.1 µm	9.9%	± 1.0%
Cumulative Volume Finer than 1.2 µm	9.8%	± 1.0%

our analyses, but is accuracy all that is needed? Accuracy is a measure of control of systematic, predictable errors. On average, we could have a high level of accuracy, meaning we are not suffering from systematic errors that are out of control. But random errors can and do occur with every particle sizing technique, providing the potential for a high degree of spread in individual measurements. The level and effect of random errors can be defined with the term "precision."

Reproducibility and repeatability are both components of precision, but each is significantly different in definition and function. Repeatability, when applied to particle size distribution analysis, is a measure of agreement between multiple analyses performed by a single operator using a single instrument analyzing a single sample. Reproducibility, on the other hand, is a measure of agreement between multiple analyses performed by multiple operators or using multiple instruments, potentially in multiple laboratories, on different days, for multiple samples, from different lots, and frequently different locations, plants and processes.

In terms of quality, both are important measures of precision—the effect of random analysis errors. But to determine whether we have a good level of repeatability and reproducibility, we have to perform a series of tests under a controlled set of conditions with a well-characterized sample. This is often called an inter-laboratory or round-robin study.

Table 2. Repeatability statistics for a Saturn DigiSizer 5200 instrument

Statistic	Average	Standard Deviation	Coefficient of Variance
Mean Diameter	5.341 µm	0.004 µm	0.08%
Median Diameter (50th Percentile)	4.856 µm	0.004 µm	0.08%
90th Percentile	10.159 µm	0.006 µm	0.06%
10th Percentile	1.277 µm	0.003 µm	0.24%
Cumulative Volume Finer than 10.0 µm	89.39%	0.04%	0.04%
Cumulative Volume Finer than 8.0 µm	78.83%	0.05%	0.06%
Cumulative Volume Finer than 6.7 µm	68.84%	0.05%	0.08%
Cumulative Volume Finer than 5.7 µm	59.24%	0.07%	0.13%
Cumulative Volume Finer than 4.8 µm	49.56%	0.05%	0.10%
Cumulative Volume Finer than 3.9 µm	39.25%	0.05%	0.14%
Cumulative Volume Finer than 3.0 µm	29.75%	0.05%	0.18%
Cumulative Volume Finer than 2.1 µm	19.90%	0.05%	0.27%
Cumulative Volume Finer than 1.2 µm	9.03%	0.05%	0.51%

The Materials Analysis Laboratories at Micromeritics Instrument Corp. have participated in a number of such studies. In some cases, the studies have been used to determine how well certain operators are doing, while others were used to test how well certain instruments work. In still other cases, the labs have participated in studies undertaken by material and product manufacturers to test their various locations, instruments, and operators. These labs have also participated in studies to determine the precision and bias of standards, such as those developed by ASTM International, and stud-

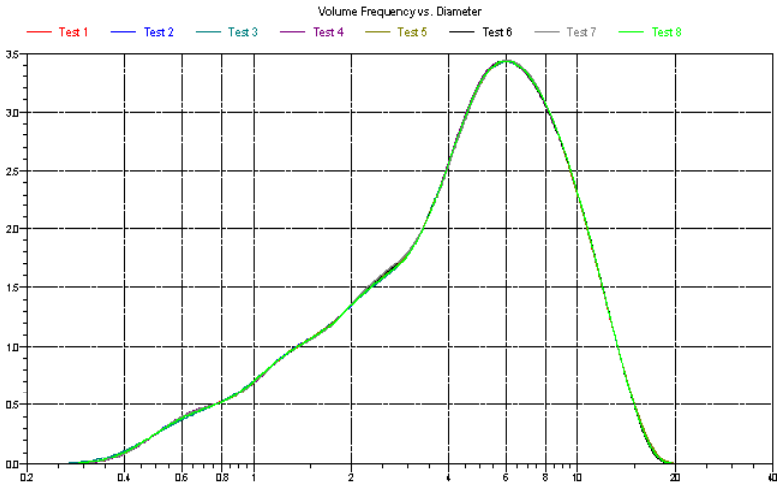


Figure 1. An overlay of eight analyses of one sample using the Saturn DigiSizer 5200

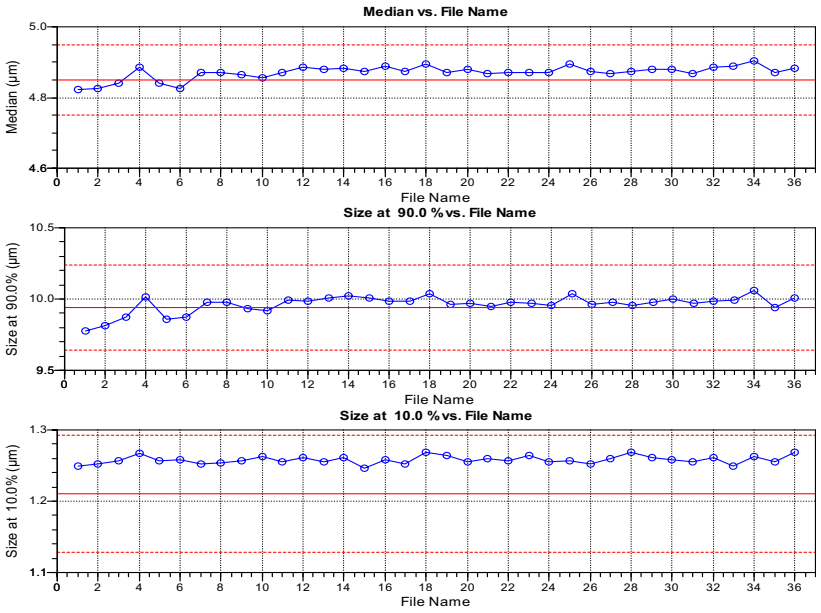


Figure 2. SPC report for 36 samples analyzed with one Saturn DigiSizer 5200

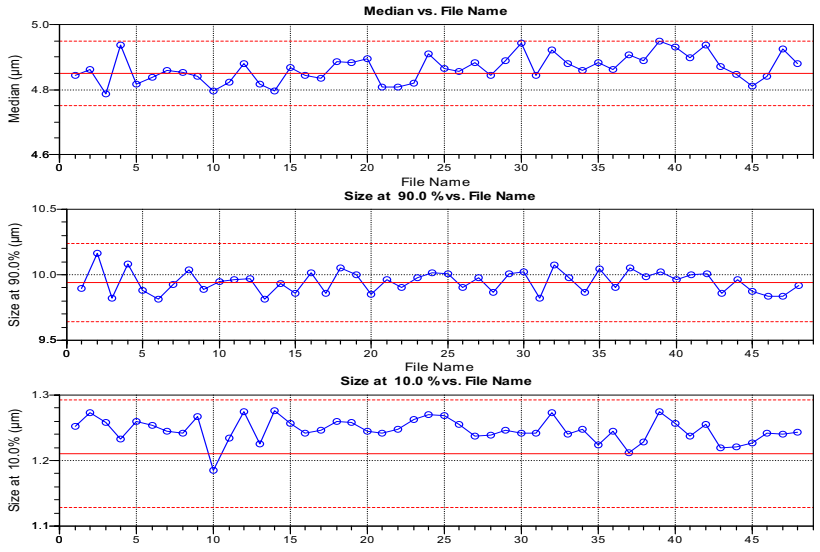


Figure 3. SPC report for one sample analyzed on each of 48 Saturn DigiSizer 5200s

ies that led to the certification of national standard reference materials.

Some of the data taken from the labs' internal studies to determine precision related to both the laboratory analysts and the laser particle size analyzers produced by Micromeritics. In both studies, the garnet-traceable secondary reference material was analyzed using a number of Saturn DigiSizer 5200 high-definition laser particle size analyzers. The sample preparation and analysis parameters were well defined in the booklets that accompanied each sample, and these were used throughout the tests. Following is an overview of some of the test results.

Repeatability Results

The repeatability of an instrument is how well it produces the same answer for the same sample analyzed a number of times. To determine repeatability, we can look at the standard deviation and coefficient of variance (CV) of several test statistics over a group of repeat analyses. Table 2 contains the average, standard deviation, and CV of mean diameter, three median percentiles (10th, 50th and 90th), and the cumulative volume percentiles finer than 9 specific diameters, which were calculated from statistics from eight tests performed on a single garnet sample using one instrument. Figure 1 shows an overlay of the frequency distribution for the eight tests. From this table and figure, it can be seen that not only do the results from each of the eight tests fit within the specifications for the traceable reference material, but also that there is essentially no variability between the tests. Such instrument repeatability is needed to ensure that any variation in results is caused by differences in the sample presented to the instrument and not in the instrument itself.

Reproducibility Results

In evaluating reproducibility, we need to consider both sample-to-sample reproducibility, where a single instrument is used to analyze a number of different samples, and instrument-to-instrument reproducibility, where a different instrument is used to analyze

the same sample. The SPC report capabilities built into the Saturn DigiSizer 5200 help to illustrate sample-to-sample reproducibility in Figure 2 for three monitored statistics: median diameter, 90th percentile and 10th percentile. It is evident that the different samples analyzed vary only slightly, and that the use of the protocols for sample preparation and analysis produces results well within the window of expected results. If the results varied greatly, it could indicate that the protocols might be inadequate to yield proper particle size analysis results for this sample. The SPC control chart shown in Figure 3 provides a plot of the same statistics from single analyses performed using 48 different instruments. As can be seen in the figure, the level of reproducibility between instruments is only about twice that seen within one instrument for multiple sample analyses. Such data indicate that results from different instruments can be compared and contrasted to determine whether samples analyzed in different locations with different instruments have similar or different particle size distributions. Companies with multiple locations and multiple processing facilities need such instrument-to-instrument reproducibility to ensure that the same product can be delivered to their customers from any of the facilities producing that product.

Table 3. Reproducibility statistics for four Saturn DigiSizer 5200 operators.

Statistic	Average	Standard Deviation	Coefficient of Variance
Mean Diameter – Operator 1	5.310 µm	0.041 µm	0.77%
Median Diameter – Operator 1	4.846 µm	0.049 µm	1.01%
90th Percentile – Operator 1	9.962 µm	0.067 µm	0.67%
10th Percentile – Operator 1	1.264 µm	0.011 µm	0.85%
Mean Diameter – Operator	2 5.324 µm	0.031 µm	0.58%
Median Diameter – Operator 2	4.879 µm	0.041 µm	0.84%
90th Percentile – Operator 2	9.978 µm	0.051 µm	0.51%
10th Percentile – Operator 2	1.257 µm	0.008 µm	0.64%
Mean Diameter – Operator 3	5.348 µm	0.042 µm	0.78%
Median Diameter – Operator 3	4.916 µm	0.061 µm	1.24%
90th Percentile – Operator 3	10.016 µm	0.066 µm	0.66%
10th Percentile – Operator 3	1.266 µm	0.013 µm	1.03%
Mean Diameter – Operator 4	5.342 µm	0.043 µm	0.80%
Median Diameter – Operator 4	4.903 µm	0.054 µm	1.10%
90th Percentile – Operator 4	10.037 µm	0.101 µm	1.01%
10th Percentile – Operator 4	1.255 µm	0.027 µm	2.15%

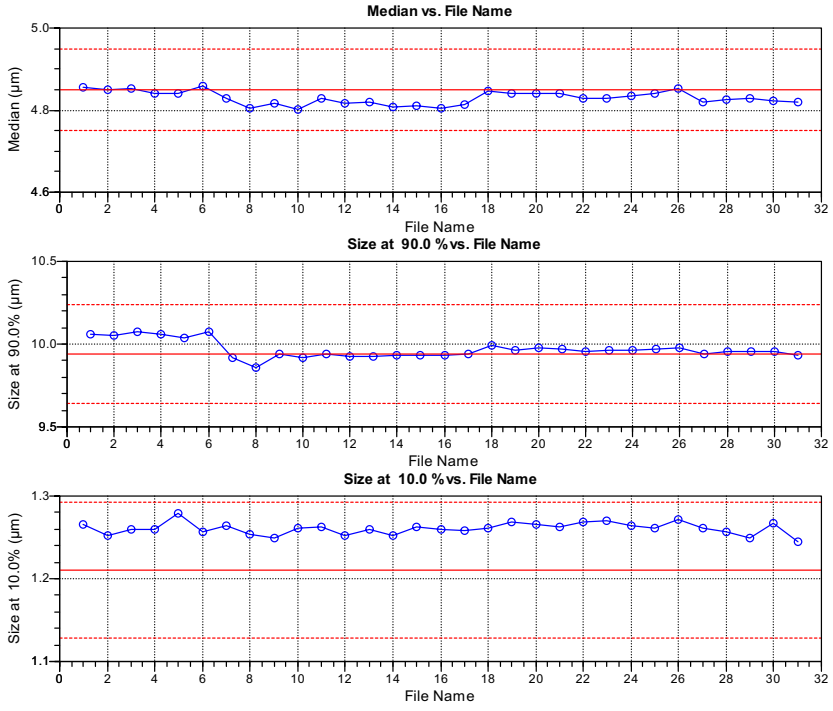


Figure 4. SPC report for one analyst using one Saturn DigiSizer 5200

Analyst Reproducibility

Figure 4 shows the reproducibility for a single analyst. In this case, the analyst prepared and analyzed a number of samples. The SPC chart shows that this analyst is able to carry out multiple analyses of the same material without variation in results. Thus, we can have confidence in results obtained by this analyst and know that if a test sample yields out-of-specification results, these results are due to a bad sample and not bad practices of the analyst. (It should be noted that in all cases, random bad analyses are possible with any analyst and total faith should not be based upon a single test. Micromeritics' laboratories generally perform three replicate particle size determinations for each sample analyzed.) Table 3 compares a portion of the results obtained by four analysts. These results demonstrate that each of the analysts tested is capable of producing quality results, and that any of these analysts should be able to carry out analyses of test samples in a quality laboratory. However, the quality manager might wish to take a close look at the techniques used by Operator 4 to see if an obvious explanation exists for the slightly higher coefficients of variance calculated from the results obtained by this analyst.

Understanding Quality

The foregoing text has provided some examples of how to quantify the two components of precision—repeatability and reproducibility—in terms of the instrument operators and the instrument used. Similar comparisons can be carried out between multiple locations within an organization, from one day to the next, and between organizations, depending on how the tests are defined and how the resulting data are compared. By understanding the standards used to measure quality in particle size distribution analysis and ensuring that the instruments they use meet those standards, ceramic manufacturers can be assured that their analysis results are accurate—and that their materials will perform as desired throughout the manufacturing process.

Take Our Customer Satisfaction Survey

We are providing customer satisfaction surveys on our website. If you are currently operating a Micromeritics instrument and would like to provide feedback on that product and/or the service you have received, please visit this page on our website: www.micromeritics.com/customer_service/Customer_surveys.aspx. You will need a product serial number to submit the survey.

You can also register your Micromeritics product online. The information you provide will be used exclusively by Micromertics Instrument Corp. for the purpose of tracking products and (optionally) to update users on issues concerning the registered product. Register your instrument at: www.micromeritics.com/customer_service/product_registration.aspx

New Application Notes and Product Information Now Available

Several new and revised application notes have recently been posted on our website. We update the site on a regular basis, adding more application and product information. Browse our website at

www.micromeritics.com

Platinum Catalyst Research Aided by New Subambient Temperature CryoCooler II

Subambient temperature reduction is critical in understanding the activity of platinum catalysts. Platinum-based catalysts are by far the most significant class of materials used in petrochemical and chemical reactors. Platinum supported on graphite and promoted by ruthenium, for example, is currently the focus of much research for use as the anode in fuel cell applications. Platinum undergoes a two-step reduction from Pt^{4+} to Pt^{2+} at subambient temperature and then to Pt^{0}. This Pt^{4+}-to-Pt^{2+} transition is a strong indication of catalytic activity. Researchers who work with platinum, ruthenium, and nickel will find the CryoCooler II accessory an invaluable aid in their efforts.

Micromeritics' AutoChem fitted with the CryoCooler II accessory creates a powerful combination for characterizing platinum-based catalysts. The CryoCooler II now incorporates a new centrifugal system for pumping liquid nitrogen and new programming which result in sample and furnace cool-down to -100 °C, smooth upward and downward temperature ramps, stable subambient temperature holding, and seamless transition into above-ambient temperature

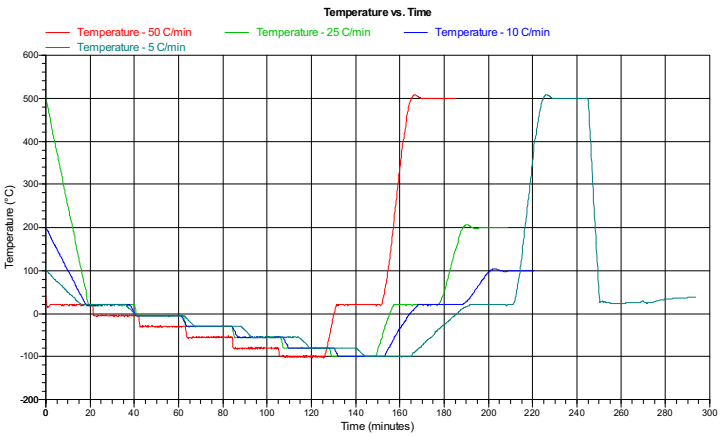


Figure 1. Examples of Furnace Temperature Control at Both Subambient and Elevated Temperatures

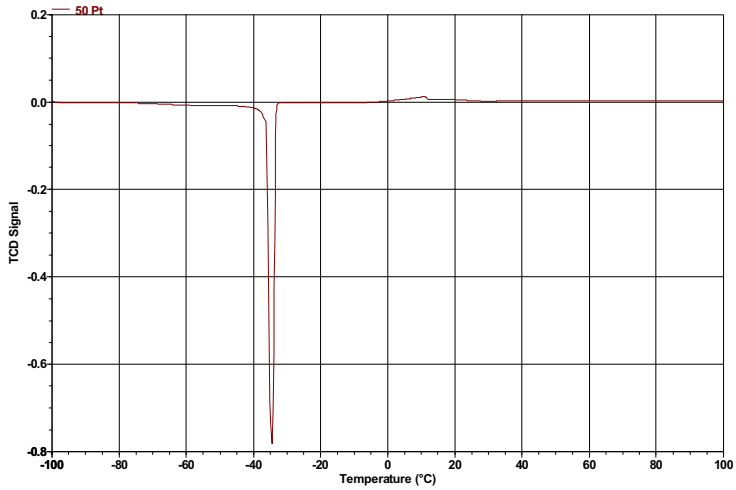


Figure 2. Temperature-Programmed Reduction of Platinum on Carbon Catalyst

regions. Figure 1 is a typical plot of furnace temperatures with time starting at and above ambient, ramping down and up in temperature at rates from 5 to 50 °C /min, holding subambient temperature, and merging into elevated furnace temperatures. Figure 2 shows the temperare-programmed reduction of platinum supported on graphite. There are two significant features on this plot. The first is the very strong reduction peak at -36 °C which is characteristic of the transition from Pt^{4+} to Pt^{2+}, and the second is a desorption or slight release of surface hydrogen near 10 °C. By adding ruthenium to platinum, as shown in Figure 3, there is created a completely different reduction profile. The primary reduction peak has shifted to 18 °C with several reduction peaks indicating oxidation states for the catalyst. There is, however, a similar release of hydrogen after the reduction. The addition of ruthenium to the platinum has created a new alloy that has very different properties from platinum alone. It is very easy to distinguish these effects using the AutoChem and CryoCooler II by overlaying these plots as in Figure 4.

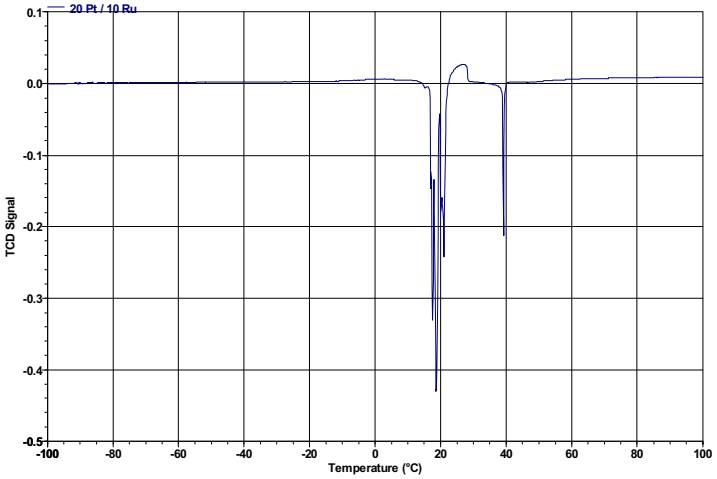


Figure 3. Temperature-Programmed Reduction of Platinum/ Ruthenium on Carbon Catalyst

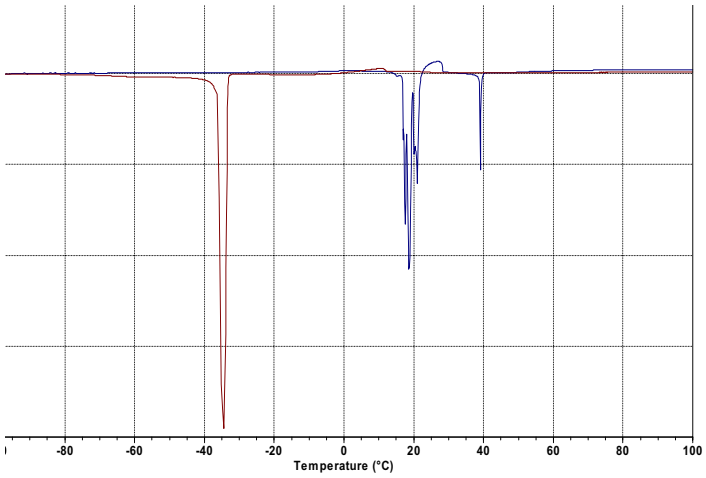


Figure 4. Overlay of Data from Figures 2 and 3

Visit our website **www.micromeritics.com** for additional application information.

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 quickly become proficient 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 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 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.

Troubleshooting
Learn techniques that enable you to locate and resolve instrument problems quickly.

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.

Training

AutoPore IV
October 7-9

SediGraph 5100
November 4-6

ASAP 2020 Chemisorption
November 18-20

ASAP 2020 Physisorption
November 11-13

Events

AAPS (American Association of Pharmaceutical Scientists)
October 26 - 30, 2003
Salt Palace
Salt Lake City, UT

ICE (International Coatings Expo)
November 12 - 14, 2003
Pennsylvania Convention Center
Philadelphia, PA

Chem Show
November 18 - 20, 2003
Jacob K. Javits Convention Center
New York, NY

Visit our website at www.micromeritics.com.

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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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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