THE microReport

micromeritics®
The Science and Technology of Small Particles™

MICROMERITICS INSTRUMENT CORPORATION

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In This Issue

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www.micromeritics.com

The New ChemiSorbs Meet a Variety of Needs

Micromeritics is pleased to introduce two new instruments designed to assist in the development, testing, and production of catalysts. The ChemiSorb 2720 and Chemisorb 2750 can be equipped to perform a full suite of experiments including both physical and chemical adsorption.

These instruments employ the dynamic (flowing gas) technique of analysis by which the quantity of gas taken up by the sample or released from the sample surface is monitored by a Thermal Conductivity Detector (TCD). The temperature at which the uptake or release occurs also is recorded. Data obtained by the instruments can be used to calculate key parameters for catalyst characterization: metal dispersion, active surface area, B.E.T. surface area, average crystallite size, surface acidity or basicity, and activation energy via first order kinetic models.

The Basic System: The ChemiSorb 2720

This basic system without the TPx software option makes chemisorption and physisorption analyses affordable to even the most modestly funded laboratories. The instrument is designed to rapidly and accurately perform pulse chemisorption studies and surface area analyses. The ChemiSorb 2720 features one port dedicated to performing the sorption analysis and a second port designed for sample preparation. It also features a built-in cooling fan for the sample port, four carrier gas inlets, one prep gas inlet, and the optional capability to accommodate a mass spectrometer or other external detector attached at the exhaust port. In addition to chemisorption experiments that include determining the percent metal dispersion, active metal area, crystallite size, and quantifying acid and base sites; a range of physisorption experiments including BET surface area, Langmuir surface area, and total pore volume can also be measured. Hands-on calibration and dosing procedures make it an excellent teaching tool for gas-solid surface interaction studies.

The basic instrument (without the ChemiSoft TPx option) provides two ways to collect data: 1) via a front panel meter that may be calibrated to display gas volumes sorbed or desorbed from a sample, and 2) by a chart recorder monitoring the analog output from the thermal conductivity detector.

The Higher-Capability ChemiSorb 2750

The ChemiSorb 2750 (built upon the same design elements as the Chemisorb 2720) has been further enhanced with the addition of an injection loop for pulsing active gases on the catalyst and features an enhanced dual-port design that allows *in-situ* preparation and analysis of two samples. Its dual-function sample ports have the capability to be used as either an analysis port or a degas port, eliminating the need to move the sample. This requires less effort and reduces the chances of contaminating an activated sample from exposure to stray gases.

Performing different types of analyses is also easier. In addition to the four carrier gas inlets and three preparation gas inlets, a dedicated gas inlet for the pulse chemisorption gas has been added (see sidebar). Thus the increased number of ports provides a rapid method for gas change-overs without the need to manually disconnect, reconnect, and purge gas lines; this further minimizes the risk of contamination and improves the ease of operation.

Optional TPx Software

The optional programmable furnace system and accompanying ChemiSoft TPx software further enhance the capabilities of the ChemiSorb 2720 and 2750. The ChemiSoft TPx option provides additional hardware and software capabilities that assist in performing Temperature-Programmed analyses (TPx). The analyses include temperature-programmed reduction (TPR), temperature-programmed oxidation (TPO), and temperature-programmed desorption (TPD). The ChemiSoft TPx software provides a system for the analyst to design and execute experiments with a simple and convenient series of informative messages and prompts for chemisorption, physisorption, and temperature-programmed analyses.

The ChemiSoft TPx software and data acquisition system are a powerful combination for easily recording the detector signal, sample temperature, furnace temperature, and time during the analysis. Advanced peak integration and peak deconvolution via curve fitting are standard features of this integrated peak-editing software. The software can also be configured to run on a workstation without being connected to an instrument; this provides the user with additional flexibility to review data files, modify the calculation parameters, perform all peak editing and peak fitting, and generate reports without the necessity of being connected to the analytical equipment.

The sorption data can then be easily reduced and reported automatically via the full-featured report system in the ChemiSoft software, which is compatible with both models of the ChemiSorb instrument. The standard reports include percent metal dispersion, peak area volume, active (metallic) surface area, active particle (crystallite) size, activation energy (first order kinetics), single- and multipoint B.E.T. surface area, Langmuir surface area, and total pore volume. And in addition to graphical reports of the TCD signal, sample temperature and furnace temperature, you have the capability of overlaying the signals from multiple sample analyses.

When used with the ChemiSorb 2750, additional reports on injection loop calibration, injection loop volume with temperature change, and calibration error are generated.

The ChemiSorb 2720 is a simple, economically priced instrument for users not requiring a high degree of automation or high throughput. The ChemiSorb 2750 provides more capability and throughput for users that need a more advanced instrument. Both models provide the accurate, high-quality measurements that you expect from a Micromeritics Instrument.

Sidebar....

Pulse Chemisorption

The first step in a pulse chemisorption analysis is to select an appropriate gas to react with the active metal being tested. The reactive gas is injected into an inert gas stream that passes through the bed of the catalyst. Downstream from the reactor is a detector which determines the quantity of gas that is removed from each injection. Pulse chemisorption tests are made with the sample at an elevated temperature so that only chemisorption occurs. The active surface of the sample is saturated when the detector indicates that the total quantity of subsequent injections passes through the sample bed without any loss. The sum of the injected quantity minus the sum of the quantity of gas that passed without adsorption equals the quantity adsorbed.

The number of gas molecules required to cover the active surface is determined; this information is used to calculate surface area. Applying the stoichiometry factor for the gas/metal reaction yields the number of accessible atoms of active metal. Using the total quantity of active metal per gram of catalyst material (determined from the manufacturing formula) leads to the determination of the percent dispersion of active metal. Using the information gathered plus the density of the metal, the size of the metal crystallite can be estimated if it is assumed that these particles have uniform geometry of known volume-to-area ratio.

Uninterruptible Power Supplies to Be Used With the ASAP 2020

Many locations where analysis instrumentation is installed may suffer from an occasional power failure. Most such failures last only a short time. However, even a momentary loss of power to an instrument that is running an analysis can result in loss of data. Since some analyses can last several days, there is the potential loss of many days worth of work. Under such circumstances, many users have asked about using an Uninterruptible Power Supply (UPS) to protect their system during brief power failures. Micromeritics does not sell UPS systems, nor do we recommend any particular manufacturer. The user will need to purchase a UPS to suit their needs. The material which follows is provided to assist you in the choice of a UPS if you prefer to use one.

The data collected during an analysis are stored in the embedded processor of the instrument. The computer does not require power-fail protection, although it is usually good practice to do so.

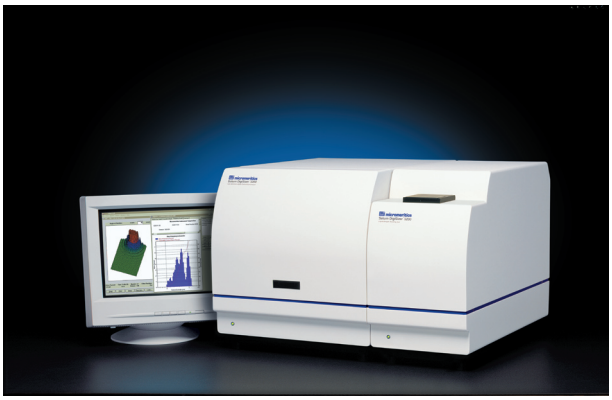
Rating a UPS for the Analyzer

The ASAP 2020 instrument is rated at 800 VA. This rating supports an ASAP 2020 with all options installed, which includes Chemisorption and a SmartVac degassing system with a dry high-vacuum pump system. Many UPS systems are available commercially which will be adequate to support the ASAP 2020 for several minutes or more, depending upon what the instrument is doing, and how many options are installed. For example, a 1000 VA rated UPS will supply full power for about 6 minutes, or half power for 15 to 20 minutes. Similarly, a 2000 VA UPS will provide full power to an ASAP 2020 for approximately 30 minutes.

Vacuum pumps

The instrument will continue to operate using the power from the UPS, at least until an extended evacuation is performed. This is because the foreline vacuum pumps do not receive their power from the instrument; they are plugged into the laboratory power system. Foreline vacuum pumps provided by Micromeritics are either oil-sealed rotary vane pumps or dry diaphragm pumps, depending upon the options installed in the instrument. The dry pump is rated at 115 Watts. The oil-sealed pump is rated at 160 Watts. If the user provides the vacuum pump, then the power requirement may be different. There is a further difficulty with selecting a UPS for the vacuum pumps. The motors of such pumps draw a heavy starting current when first turned on. For example, the oil-sealed pump supplied by Micromeritics may draw a starting current of 5 Amps (at 120 Volts) for up to a second. Therefore the UPS would need to be rated at 600 VA for each pump, unless the UPS manufacturer has allowed for start-up surge currents.

Use of Minimum Signal Fraction Setting in Saturn DigiSizer Sample Information Files



The light scattered by particles is only a portion of the total laser light scattered during analysis with the Saturn DigiSizer. The optical components of the analyzer and the dispersing liquid also scatter light; this is known as *background scattering*. The light scattered by the particles compared to that of the background is called the *scattering signal fraction*. In order to limit the effects of scattering noise, a threshold known as the **Minimum signal fraction** can be specified in the Saturn DigiSizer sample information file. Any collected data that do not exceed the background by this specified intensity percentage are not used in particle size calculations.

The minimum signal fraction default value is 16%. This means that scattering data collected which have intensities equal to or greater than 16% above the background level at that angle are used in the particle size calculations. Data points where the scattering intensity is less than 16% above the background are not used in calculations, although data are collected at all angles where the intensity is 3% above the background.

If you need to increase the signal fraction at particular angles, increase the concentration of the sample. This results in a linear increase in signal fraction and, therefore, data at more angles (generally smaller angles) are used. This also allows detection of larger particles which may have been missed in the original experiment. There is a limit, however, in using higher concentrations. If small particles are present, multiple scattering may result. In this case, what do you do in order to size the larger particles in the presence of the smaller ones in a sample with a broad particle size distribution? You change the minimum signal fraction to allow more data to be used.

In Figure 1 the original default minimum signal fraction of 16% is used to calculate the size of an alumina sample analyzed with an obscuration of 10.9%. The large end of the distribution is artificially truncated because the signal from the larger particles is less than 16% above background. For this sample, the concentration could not be increased to raise the signal for the larger particles since doing so would result in multiple scattering by the smaller particles. This can be seen in Figure 2 when the sample is analyzed with an obscuration of 29.0%. In Figure 3 the minimum signal fraction is reduced to 5% for the original sample. Notice that the full distribution is calculated since the largest size is the same as when the concentration was increased (Figure 2).

So, should you always use a lower signal fraction rather than a higher concentration for samples that contain only large particles? No; use a higher concentration when possible. This improves the statistics, and thus the reliability, of the analysis since a larger number of total particles is analyzed. It also reduces the effects that noise can have on the analysis.

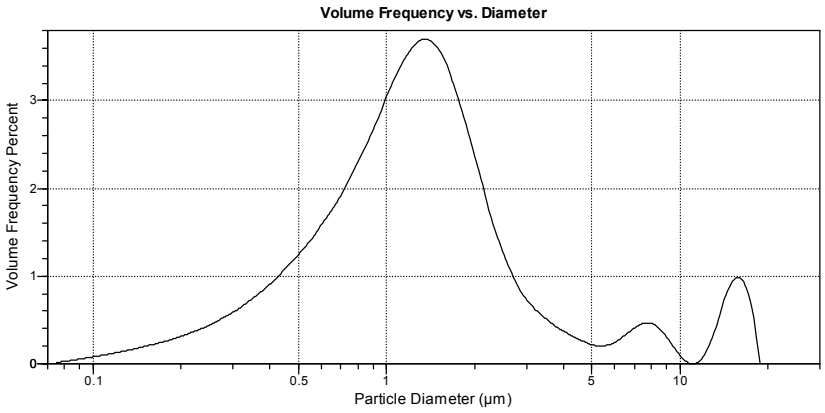


Figure 1. Alumina sample analyzed at 10.9% obscuration and 16% minimum signal fraction.

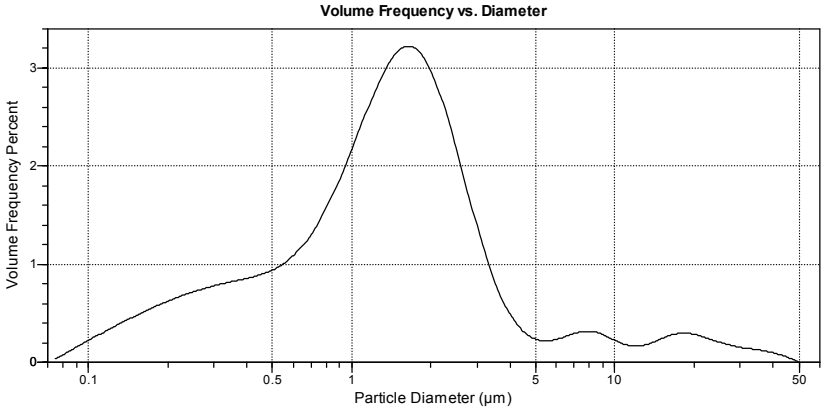


Figure 2. Alumina sample analyzed at 29.0% obscuration and 16% minimum signal fraction.

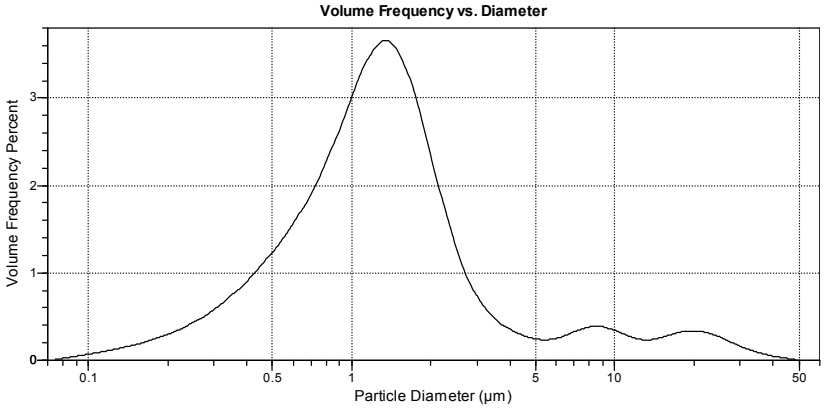


Figure 3. Alumina sample analyzed at 10.9% obscuration and 5% minimum signal fraction.

Micromeritics' AquaPrep™ Helps Eliminate Air Bubbles

The use of water as a dispersing liquid for particle size analysis can often result in misleading or even incorrect data due to the presence of air bubbles. If the air bubbles are not removed, the particle size analyzer may detect them and report them incorrectly in the measured distribution. To solve this problem, Micromeritics has developed the AquaPrep.

The AquaPrep operates by recirculating water through a hydrophobic capsule consisting of many thin-walled capillaries. A vacuum pump in turn provides suction on the outside of the capillaries. This results in a diffusion of dissolved air from the water through the capillary walls and into the vacuum pump. The air removed from the water is exhausted through a small tube at the front of the instrument. The AquaPrep is convenient, small in size, and simple to use. For more information on the web, see www.micromeritics.com/pdf/products/AquaPrep.pdf

Micromeritics' Tony Thornton Receives ASTM 2004 Award of Merit

Tony Thornton, Senior Scientist at Micromeritics, has been awarded 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.

The committee states that, "As Committee B09 Secretary for more than eight years, Tony Thornton's accurate and timely minutes have kept the committee on track through many complex standard proposals and revisions. In addition to his work in B09, his enthusiastic efforts were instrumental in the resurgence and current vitality of Committee E29. He continues to lead and advise that committee as Past Chairman and in other leadership positions. Tony's technical efforts in Committee E29 have brought order to the sometimes chaotic world of powder particle size measurement, particularly in the reporting of measurement results."

He has also served as liaison among 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.

Customer Satisfaction Surveys

Customer satisfaction is the key to success. Getting you, our customers to tell us what's good about our products or services, and where we need improvement helps us to ensure that our business measures up to your expectations.

We are offering on our website a number of customer satisfaction surveys designed to help us gather this important information.

In addition to the Customer Satisfaction surveys, we are offering surveys designed to keep us informed about needs in analytical laboratories. The information is used not only to improve current products, but to provide us with a basis for future product development. We would like to include your opinions and comments in the current studies. Please look for these surveys on our website www.micromeritics.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 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 quickly locate and resolve instrument problems.

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 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 Schedule

Saturn DigiSizer® 5200
June 8 - 10

AutoChem™ II 2920
August 17 - 18

TriStar™ 3000
August 24 - 26

SediGraph™ III 5120
August 31 - September 2

Events

Fundamentals of Adsorption
May 23 - 28
Sponsored by Cleveland State University
Hilton Sedona Resort
Sedona, AR

Lab 04
September 14 - 16,
Lillestrom, Norway

International Coatings Expo
October 27 - 29
McCormack Place
Chicago, IL

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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