THE microReport

MICROMERITICS INSTRUMENT CORPORATION VOLUME 18 NO. 3

In This Issue

Micromeritics Introduces Two New Products

Micromeritics is pleased to introduce the new ASAP 2050 Xtended Pressure Sorption Analyzer as the latest addition to Micromeritics' popular line of Accelerated Surface Area and Porosimetry (ASAP) instruments. For customers who need to perform physisorption tests at elevated pressure (up to 10 atmospheres), the new ASAP 2050 offers an extended range of capabilities while retaining the superior design elements of the well-established ASAP 2020. The ASAP 2050 is particularly well suited for research into fuel cell, hydrogen storage, and pressure swing adsorption applications.

A key feature of the ASAP 2050 utilizes two independent vacuum systems, one for the sample preparation and one for the analytical system. This proven two-vacuum system design allows sample preparation and analysis to proceed concurrently without interruption and eliminates the possibility of cross-contamination between sample preparation and analysis.

The ASAP 2050 also offers a variety of new design features. Long-duration 3-liter Dewars (typically up to 50 hours of operation) combine with Micromeritics' patented Isothermal Jackets to maintain a constant thermal profile along the sample and the saturation pressure (P_o) tubes throughout extended analyses. (Virtually unlimited analysis time is possible by refilling the Dewar during the analysis.) Stainless-steel sample tubes are capable of safe operation up

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to 150 psia. Special heating mantles can be used to prepare samples in situ on the analysis port prior to analysis. These new heating mantles have longer cords so that they can be placed on the sample tube without removing the Dewar or other hardware from the instrument.

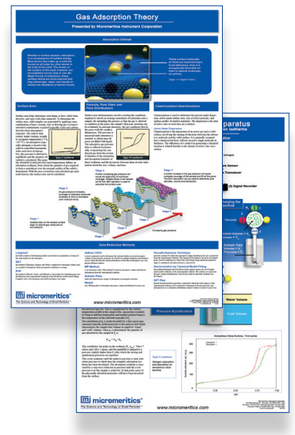
Isotherm cycling software is standard, allowing collection of non-monotonic isotherms more rapidly. An easy-to-use Windows® interface makes it simple to collect, organize, archive and reduce raw data, and store standardized sample information and analysis conditions for quick access. Finished reports may be generated to screen, paper, or data transfer channels. Features include cut-and-paste graphics, scalable-and-editable graphs, and customizable reports.

Dynamic Void Volume Analyzer 4000

The DVVA (Dynamic Void Volume Analyzer) 4000 is a state-of-the art dynamic analyzer capable of measuring the compressed void volume and density of carbon black and other materials with precision and accuracy. This unique instrument can potentially replace the current oil absorption test used by the carbon industry as a predictor of optimal polymer/carbon black ratios in reinforced elastomer materials. The DVVA 4000 is fully automated with an easy-to-use Windows interface. The instrument requires no manual preparation and is self-cleaning. The primary purpose of the DVVA 4000 is to measure the void volume vs. pressure and provides accurate and reproducible data. It is believed to meet all requirements of ASTM D 6086 test method for carbon black void volume. The extrapolation model to estimate COAN remains under development. As the work of ASTM D-24 reaches a consensus for all classes of carbon blacks, the preliminary COAN model now included will be updated.

DVVA 4000 Dynamic Void Volume Analyzer

Gas Adsorption Theory and Gas Adsorption Appartus posters are now available.



Micromeritics Instrument Corporation is releasing a series of posters illustrating the theories and the technologies of our broad line of automated, particle characterization analytical laboratory instruments.

In this series of posters, the various theories used to determine particle characteristics such as surface area, particle size, pore volume, pore size, absolute density, envelope density, bulk density, catalytic activity, and active surface area are presented. These posters are now available through our online catalog, or by calling customer support or your sales representative.

Micromeritics Introduces Comprehensive Product Support Plans

In an effort to serve its customers better, Micromeritics introduces support options to the U.S. market that will lengthen instrument life, minimize cost of ownership, provide software and hardware updates, control maintenance costs, and eliminate the catastrophic costs of downtime. These proactive maintenance programs are intended to eliminate service issues before they arise.

Product support programs with a choice of five customized levels of product support and two levels of response time have recently been introduced for the Elzone Particle Size analyzer and the TriStar Surface Area analyzer. In the near future, Micromeritics plans to cover the company's entire instrument line and to expand this program globally.

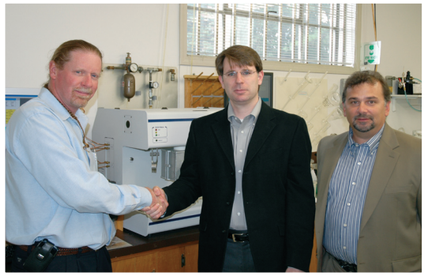
Plan options include 24/7 phone support, tuition-free classroom training, software and hardware updates, free application support, free sample testing services in the event of instrument downtime, discounts on accessories, 48-hour response time, and special website access to premium content. Choose the plan with a combination of services that fits your needs. These hassle-free maintenance plans cover all instrument routine maintenance and repairs, and remove the guesswork from your operations budget with one yearly invoice. You can also reduce your administrative burden (and costs) even further by choosing a discounted multiple-year, multiple-site, or multiple-unit support plan.

Instrument Grant Award Winner

After careful consideration by a special Grant Selection Committee appointed by the president of the company, Micromeritics' grant award winner for the second quarter of 2007 was selected.

Micromeritics recently awarded an AutoChem II 2920 Catalyst Characterization System to the School of Chemical and Biomolecular Engineering at the Georgia Institute of Technology, Atlanta, Georgia.

According to Christopher Jones, principal investigator, "The AutoChem II system will directly enable new research approaches in 21 projects covering four different research groups. The instrument will play a central role in my catalysis and adsorptive separation research program." Dr. Jones is a primary member of the Strategic Energy Institute at Georgia Tech, where he directs a research effort in catalysis, adsorption, and materials synthesis. According to Preston Hendrix, Micromeritics' president, "This program is designed to promote and advance the acquisition and use of particle characterization instrumentation not generally available through other means to non-profit universities and institutions. We are very proud and excited to present this award in an ongoing grant program to support important research."



Preston Hendrix, President Micromeritics; Dr. Christopher Jones, Georgia Tech; Dr. Jeff Kenvin, Senior Scientist Micromeritics

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Nitrogen Adsorption on Lithium Exchanged X Zeolite (Li-X) at Multiple Temperatures Using the ASAP 2050

Reid Davis

Introduction

When gas physically adsorbs onto a sample, temperature plays a very significant role in the amount of gas that the sample is able to adsorb. Performing analyses on a sample at multiple temperatures allows the correlation between temperature and adsorbance to be seen. Zeolite Li-X (lithium exchanged X Zeolite) is a very good sample for the purpose of temperature-controlled analyses as it is highly dependent on temperature. Useful information can be extracted from the adsorption data at multiple temperatures; for example, finding a temperature which maximizes efficiency of adsorption on a large scale. Heat of adsorption data can also be extracted. The heat of adsorption is the amount of energy that is released when the adsorbate adsorbs onto the sample. The ASAP 2050 software includes a heat of adsorption report to allow easy calculation of the data. Heat of adsorption reports require at least two separate isotherms at different temperatures over the same pressure range to report accurate and repeatable results. The gas compressibility factor can be changed based upon the temperature and the thermochemical properties of the gas. The compressibility factor relates to the behavior of the gas at certain temperatures when compared to an ideal gas. Changes in the compressibility factor over a small temperature range are usually small, but have a noticeable impact on the adsorption data; therefore they cannot be ignored.

Preparation

Approximately 1.2 grams of Li-X was manually degassed on the analysis port of the ASAP 2050 at 350 °C for at least two hours in a steel sample tube. The sample was degassed on the analysis port to prevent atmospheric exposure after degas and, thereby, reduce potential causes for discrepancies.

ASAP 2050 Xtended Pressure Sorption Analyzer

Analysis

After degassing, the sample was analyzed at six different temperatures (-5 °C, 0 °C, 10 °C, 20 °C, 30 °C, and 40 °C) to obtain a range of temperatures for computation of a heat of adsorption isotherm.

The analysis was conducted over a pressure range of 0 mmHg to 7500 mmHg with both adsorption and desorption data being taken. To keep the sample free of helium during the analysis process, free space was measured after each analysis as a separate analysis file. Isothermal jackets and a Dewar were not used for the analysis since temperature was controlled through a thermoelectric cooling device. The device uses a thermoelectric cooler, also known as a Peltier, to cool the block in which the sample tube fits. The Peltier holds the temperature at a stable, precise temperature during the analysis.

Results

The Li-X uptake of nitrogen is highly dependent upon temperature as is shown in Figure 1. As temperature increases, nitrogen adsorption decreases; it can be seen in the isotherms that a small variation in temperature causes a noticeable variation in the amount of nitrogen adsorbed. Because of the temperature's effects on the isotherms, the thermoelectric cooling device was used to maintain a stable temperature.

From the multiple isotherms at different temperatures, the isosteric heat of adsorption isotherm can be calculated. Figure 2 shows the isosteric heat of adsorption calculated from the nitrogen adsorption data given in Figure 1. The data show the energy associated with the N_2 adsorption onto the Li-X sample. The negative sloping sections are where energy is released as the N_2 adsorbs. The upward sloping sections are where work has to be performed by the gas to adsorb onto the surface. Nitrogen adsorbs readily onto the sample; the only time that work is performed is when high quantities of nitrogen are adsorbed. The beginning and end of the heat of adsorption graph are not as stable as the middle section because the higher quantity adsorbed section is more dependent on lower temperatures and the lower quantity adsorbed section is more dependent on higher temperatures. Overall, the most noticeable attribute of this heat of adsorption plot is that Li-X has an unusually high amount of energy released for a sample undergoing physical adsorption of nitrogen.

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

Figure 1:

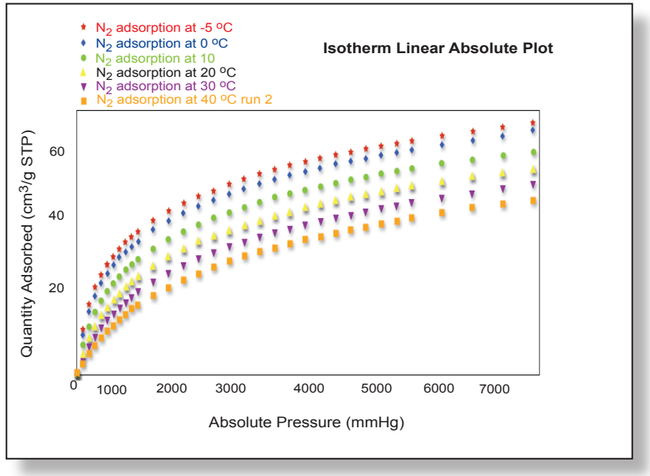
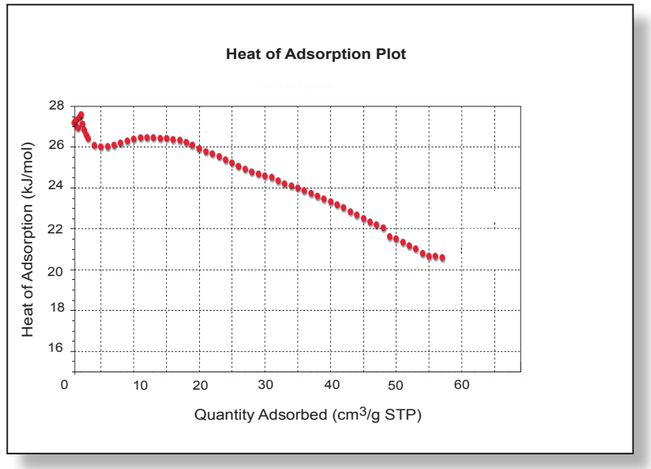


Figure 2:



What's New at MAS

The mission of Micromeritics Analytical Services (MAS) is to provide all our customers with the best in contract analytical services. We will continually strive to improve and expand the services provided to meet our customers' needs.

MAS is pleased to offer particle size and particle shape analysis using dynamic image analysis. Using an automated camera-based system we can determine the particle size of a two-dimensional projected image. The system can also generate and report many additional shape parameters such as sphericity, roundness, aspect ratio, and Feret diameters. In total, there are 24 different shape parameters which can be calculated for each sample. This technique is perfect for materials where particle morphology may be more important than an equivalent spherical diameter particle size.

Coming soon:

Micromeritics Analytical Services is leading a research project which is studying the effect that particle shape has on the reported particle size distribution using five different particle size analysis techniques. We will demonstrate and explain how different techniques can report different particle size results, each with accuracy and repeatability.

MICROMERITICS ANALYTICAL SERVICES The Particle Testing Authority

Look for Micromeritics Analytical Services at these upcoming events and meetings:

AAPS – Annual Meeting
November 11-15, 2007
San Diego, CA

Pittcon
March 2-6, 2008
New Orleans, LA

Interphex
March 26-28, 2008
New York, NY

Micromeritics Analytical Services is pleased to introduce two new employees.

Amanda Thompson is a recent graduate from Georgia Southern University with a major in Chemistry. Amanda is primarily responsible for measuring gas adsorption isotherms and BET surface area analyses.

Mona Bishop is a recent graduate from Georgia State University with a major in Chemistry. Mona's main responsibilities include particle size analyses using laser light scattering and sedimentation techniques.

2008 Instrument Training Course Schedule

Elzone II 5390
February 12 - 14

SediGraph 5120
February 19 - 21

Gemini Series
March 18 - 19

ASAP Physisorption and Chemisorption
April 1 - 4

ASAP Physisorption
April 1 - 3

TriStar 3000
April 15 - 17

Saturn DigiSizer 5200
April 22 - 24

AutoChem 2920
June 3 - 5

AutoPore IV 9500 Series
June 10 - 12

ASAP 2420
August 19 - 21

SediGraph 5120
August 26 - 28

TriStar Series
September 9 - 11

Gemini Series
September 16 - 17

AutoChem 2920
November 4 - 6

AutoPore IV 9500 Series
November 11 - 13

ASAP Physisorption and Chemisorption
December 2 - 5

ASAP Physisorption
December 2 - 4

Above: Students attending a recent training course

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.

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

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 4 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.

Attention Authors

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

Send your article to:
James Kerce, Editor
The microReport
MICROMERITICS
One Micromeritics Drive
Norcross, GA 30093-1877
james.kerce@micromeritics.com

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:

HEADQUARTERS:

Micromeritics Instrument Corporation
One Micromeritics Drive
Norcross, GA 30093-1877
USA
Telephone:
U.S. Sales (770) 662-3633
International Sales (770) 662-3660
Fax: (770) 662-3696
WEB: www.micromeritics.com

SUBSIDIARIES:

Micromeritics China
Apt. 5H, No. 1 Building
Hua-Ao (Epoch Center)
No. 31 Zi Zhu Yuan Road, Hai Dian District
Beijing 100089, CHINA
Tel: (+86) (0)10-6848-9371
Fax: (+86) (0)10-6848-9371

Micromeritics Shanghai China
Room 15M, J Building, Ladoll International
No. 831 XinZha Road, JingAn District,
Shanghai 200041, CHINA
Tel: (+86) (0)21-6217-9208
Fax: (+86) (0)21-6217-9180

Micromeritics France S.A.
Parc Alata
Rue Antoine Laurent Lavoisier
60550 - Verneuil en Halatte, FRANCE
Tel: (+33) (0)33-3-44-64-6080
Fax: (+33) (0)33-3-44-64-6089

Micromeritics GmbH
Erftstrasse 54
D-41238 Mönchengladbach, GERMANY
Tel: (+49) (0)2166-98708-00
Fax: (+49) (0)2166-98708-88

Micromeritics Ltd.
Unit 2, Chestnut House
178-182 High Street North
Dunstable, Bedfordshire LU6 1AT
ENGLAND
Tel: (+44) (0)1582-475248
Fax: (+44) (0)1582-475252

Micromeritics N.V./S.A.
Eugene Plaskylaan 140B
1030 Brussels, BELGIUM
Tel: (+32) 2-743-39-74
Fax: (+32) 2-743-39-79

Micromeritics SRL
Via W. Tobagi n. 26/7
20068 Peschiera Borromeo
Milano, ITALY
Tel: (+39) (0)2 553 02833
Fax: (+39) (0)2 553 02843

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(770) 662-3654

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Micromeritics
One Micromeritics Drive
Norcross, GA 30093-1877, U.S.A.

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