THE microReport

micromeritics®
The Science and Technology of Small Particles™

MICROMERITICS INSTRUMENT CORPORATION

VOLUME 19 NO. 1

In This Issue

CERTIFIED ISO9001 MANUFACTURER NORCROSS, GA

Micromeritics to Showcase a Variety of New Instruments at the 2008 Pittsburgh Conference

Pittcon is one of the world's premier annual conferences devoted to laboratory science and instrumentation. This year, Pittcon takes place March 2 through March 7 at the Ernest N. Morial Convention Center in New Orleans, Louisiana.

Micromeritics, located in Booths 2620 and 2621, will be introducing some of the latest developments in particle characterization instrumentation. This year's exhibition presents a great opportunity to get a hands-on look at a variety of new instruments available from Micromeritics, as well as the opportunity to speak personally with our technical, sales, and customer support associates.

TriStar II 3020

The **TriStar II 3020 Surface Area and Porosity Analyzer** is a fully automated, three-station analyzer that can measure surface areas as low as 0.01 m^2/g using nitrogen and features a new krypton option that can measure surface areas down to 0.001 m^2/g. A dedicated P_0 port is standard, allowing the user to measure the saturation pressure on a continuous basis. The TriStar II can collect up to 1000 data points. Minute details of the isotherm can be observed and recorded providing high resolution and revealing pore structure details. A 2.75-liter Dewar and extended length sample tubes allow complete adsorption and desorption isotherms to be collected without operator intervention. The TriStar II packs all these features and more into an intelligently designed instrument that combines a small footprint with easy accessibility.

continued on page 2

Pittcon continued

The new **ASAP 2050 Xtended Pressure Sorption Analyzer** is an easy-to-use instrument capable of collecting physisorption data in an elevated-pressure environment. While retaining design elements of Micromeritics' popular ASAP 2020, the ASAP 2050 analysis manifold is equipped with a pressure transducer capable of operating from vacuum to 10 atmospheres. Custom straight-walled, stainless-steel sample tubes are capable of safely withstanding temperatures and pressures well beyond the system's specifications. Two independent vacuum systems allow simultaneous preparation of two samples and analysis of another. Sample preparation and analysis can occur concurrently without interruption and without the risk of cross-contamination. The instrument is particularly well suited for fuel cell, hydrogen storage, and pressure swing adsorption research applications.

The **AccuPyc II 1340 1-cm^3 Gas Displacement Pycnometer** is designed for those who typically analyze a very limited amount of sample. The AccuPyc features improvements in design that allow the volume of small amounts of sample to be accurately detected and measured with enhanced reliability to within a fraction of a microliter. A novel manifold design utilizes high-reliability, high-precision valves that seal more securely so that small leaks do not contribute to variability of results. High-compression, minimum-volume seals around the pressure transducer minimize helium storage and leakage. This shortens the time to complete a run cycle and practically eliminates the development of temperature gradients that would otherwise compromise results. The AccuPyc II 1340 1-cm^3 also features an ultra-low noise transducer amplifier that results in faster equilibration.

AccuPyc II 1340 1-cm^3 Gas Displacement Pycnometer

The **AutoChem 2950 HP High Pressure Chemical Adsorption Analyzer** is an automated catalyst characterization system capable of preparing and analyzing samples at elevated pressures up to 1000 psia and at temperatures from -100 to 1100 ºC. The instrument can perform a variety of experiments including pulse chemisorption, BET surface area, temperature-programmed reduction (TPR), desorption (TPD), oxidation (TPO), and reaction analyses. Equipped with all the features of Micromeritics' AutoChem II 2920, this instrument is suitable for a variety of applications including fuel cell and hydrogen storage research. This micro-reactor, combined with a mass spectrometer, can also be used to determine product yields and catalytic activity under commercially viable conditions.

Pittcon 2008

Booths: 2620 and 2621

Micromeritics Website Offers Premium Information for Registered Customers

Home page Log-in button

Micromeritics has recently added to its website a special premium section dedicated to our instrument users. It is our goal to add value to our products by providing our customers with a knowledge base of not only Micromeritics instrument information, but industry and academic information as well. This evolving area will eventually provide customers with free downloadable software upgrades, product-specific user information, operator training videos, power point presentations, and other useful offerings.

This section currently contains Operator manuals, Material Safety Data Sheets, product bulletins, technical articles and papers, a FAQ (frequently asked questions) section, a bibliography of technical papers referencing Micromeritics instruments, archived issues of The microReport, technical tips, and more.

To access this database simply click on the "Log-in" button in the left column of the Micromeritics home page (micromeritics.com) and register your Micromeritics instrument information. You will be emailed a confirmation within two business days.

We are excited about this database of Micromeritics technical and product information for our customers and look forward to making micromeritics.com an even more valuable resource in the future.

Announcing the Fifth Instrument Grant Award Winner

A Pulse ChemiSorb 2705 Catalyst Characterization System has been awarded to the Department of Chemistry and Geology at Columbus State University, Columbus, Georgia.

According to Dr. Anil Banerjee, Principal Investigator, "The Pulse ChemiSorb 2705 system will be used for chemisorption and physisorption studies on a number of projects. We are setting up a catalytic research group that will conduct research in the areas of hydrogen storage and catalytic oxidation of carbon monoxide using platinum, non-platinum group metals, oxides, and alloys. Our plan also includes the training of a selected number of high school chemistry teachers (working in our federal Teacher Quality project) and very talented high school students."

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 pleased to present this award in an ongoing grant program to support important research."

Part I: Characterization of Supported Palladium, Hydrogen Sorption

By
Jason Exley

Introduction

The ASAP 2020 Chemisorption instrument can be used for many different analyses. One of the uses of the 2020 can be to analyze the chemisorption of hydrogen onto supported metal catalysts. In this study, 5 weight % palladium on activated carbon was used. During chemisorption, the bonds between H_2 molecules dissociate on the metal and the individual hydrogen atoms chemically bond to the surface atoms of the palladium[1]. As the hydrogen bonds to the surface of the metal, the 2020 measures the quantity of hydrogen adsorbed at certain pressures. In this study, a wide range of pressures were employed, ranging from 0.01 mmHg to 500 mmHg. A detailed activation procedure is given in Table 1. The palladium is reduced in flowing H_2 to produce a clean catalytically active surface. This removes all impurities from the sample that would produce unwanted effects on the hydrogen adsorption. The analysis is ready to begin when the pressure in the sample tube has been held at or below 10 µmHg for 30 minutes. To acquire the quantity adsorbed at the low pressures, very small volumes of hydrogen are dosed to the sample via the ASAP 2020's low pressure dosing option. This provides a detailed chemisorption isotherm in low pressure (< mmHg). This occurs until all low-pressure data points are acquired and then larger volumes of hydrogen are dosed to create the higher pressure environment for the sample. The overall analysis is performed at multiple temperatures, ranging from 35 ºC to 120 ºC. The varying temperatures cause different quantities of hydrogen to be adsorbed onto the sample at specific pressures. As the temperature of the sample increases, the shape of the isotherm changes and less hydrogen is adsorbed as temperature is increased.

Task Number	Task Name	Gas	Temp (ºC)	Rate (ºC/min)	Time (min)
1	Evacuation		100	10.0	30
2	Flow	H_2	100	10.0	5
3	Flow	H_2	300	10.0	120
4	Evacuation		300	10.0	30
5	Evacuation		70	10.0	30
6	Analysis	H_2	70	10.0	

Table 1: Preparation Analysis

Results

As seen in Figure 1, the isotherm forms two major steps with a plateau between them. A majority of the total hydrogen chemisorption occurs in these two narrow pressure ranges. The low-pressure adsorption, below 0.1 mmHg is the first major adsorption step; this is where the first layer of hydrogen is bonded to the surface of the palladium. A monolayer of hydrogen is sorbed to the surface. The second step of the isotherm, between 30 mmHg and 75 mmHg, is where additional hydrogen is absorbed into the palladium and forms palladium hydride. These

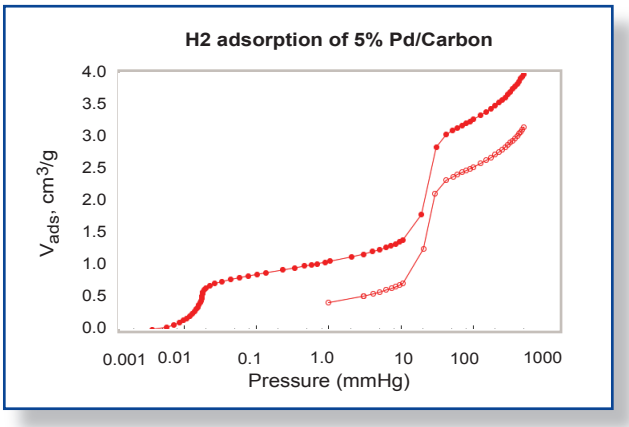


Figure 1: Palladium Hydride Isotherm (50 ºC)

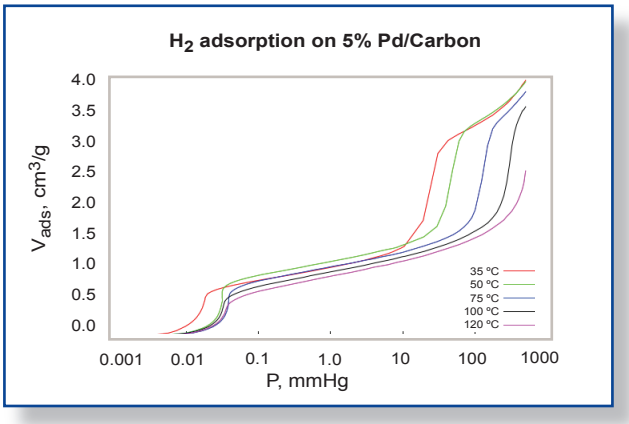


Figure 2: Hydrogen sorption on 5% Pd supported on carbon at temperatures ranging from 35 to 120 ºC

pressure ranges where gas adsorption occurs are temperature dependent. This specific isotherm is formed when the palladium is heated to 50 °C. If the sample was at a warmer or cooler temperature, the isotherm would shift right or left, respectively. This pressure-temperature relationship can be seen using the van't Hoff equation.

$$\ln(P) = (\Delta H/RT) - (\Delta S/R)$$

Where ΔH is the enthalpy of the hydride in kJ/mol, ΔS is the entropy of the hydride in kJ/mol • K, R is the gas constant, 8.314472 J/(K*mol), T is the temperature in Kelvin, and P (in atmospheres) is the mean pressure of the second step of the isotherm in atmospheres.

After performing multiple analyses at different temperatures (Figure 2), the isotherm data can be used in conjunction with the van't Hoff equation to calculate the enthalpy and entropy of the hydride formation, as has been reported by Sandia National Laboratory[2]. To do so, for each temperature at which the sample was analyzed, the mean pressure of the second step of the isotherm (in atmospheres), and its corresponding temperature (in Kelvin) must be noted. With this data, the logarithm of the pressure is plotted versus the inverse of the temperature and a linear regression can be formed, as seen in Figure 3.

The slope of the line is equal to ΔH/R (the Enthalpy) and the y-intercept is equal to ΔS/R (Entropy). Once these two values are calculated, the mean pressure at which hydrogen will sorb onto the palladium sample can easily be predicted.

Furthermore, from the temperature and pressure data acquired through the isotherms or through calculation of the van't Hoff equation, the heat of adsorption (the energy required for adsorption to take place) can be calculated for a specific quantity of gas adsorbed[3]. This is done using the Clausius-Clapyron equation

$$(\partial \ln P / \partial T)_x = q / RT^2$$

or the more commonly employed form:

$$\ln(P)/m = q/R * 1/T$$

The Clausius-Clapyron equation provides a convenient technique for determining the isosteric heat of adsorption. When several isotherms are available (Figure 2), a plot of the ln(P) versus 1/T at constant quantity adsorbed provides a linear relationship. The slope of that line is q/R where q is the isosteric heat of adsorption and R is the gas constant. A range of adsorbed quatities may be used to develop a plot of isosteric heat versus coverage as seen in Figure 4.

Comparing Figure 3, the van't Hoff plot vs. Figure 4, the isosteric heat, it is clear the isosteric heat of adsorption provides a detailed analysis of the heat released during the formation of PdH_x. The van't Hoff plot only provides an appropriate or average enthalpy.

Resources:
1.Webb, Paul A. and Orr, Clyde. Analytical Methods in Fine Particle Technology. Micromeritics Instrument Corp., 1997.
2.Sandia National Laboratories Hydride Properties Database.
3.Gregg, S. J. The Adsorption of Gases by Solids. New York, N.Y: Chemical Publishing Company, Inc., 1934.

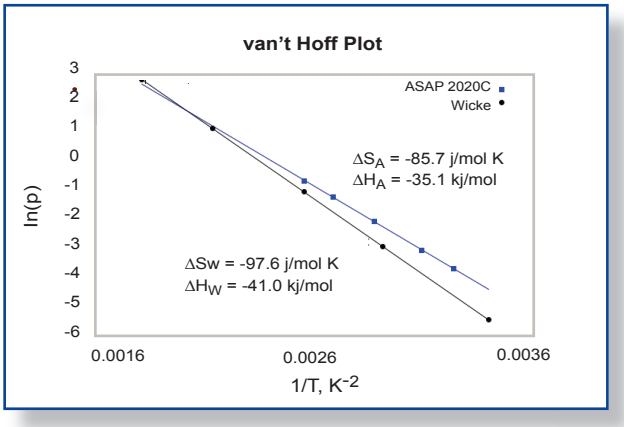


Figure 3: The linear regression of the tabulated data using the van't Hoff equation wth the enthalpy and entropy values displayed

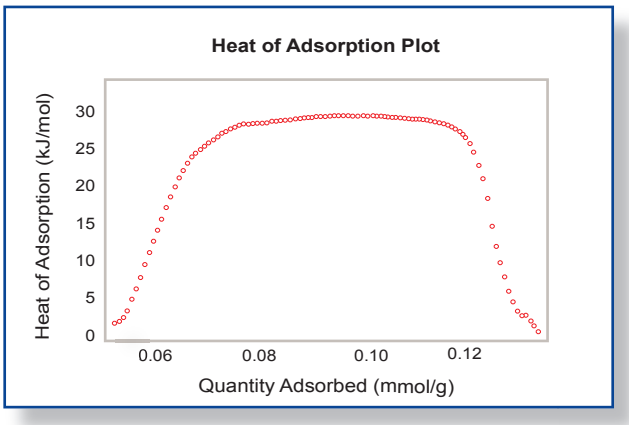


Figure 4: The isosteric heat of adsorption of Hydrogen on Palladium

What's New at MAS

MAS Presents a Poster at Pittcon

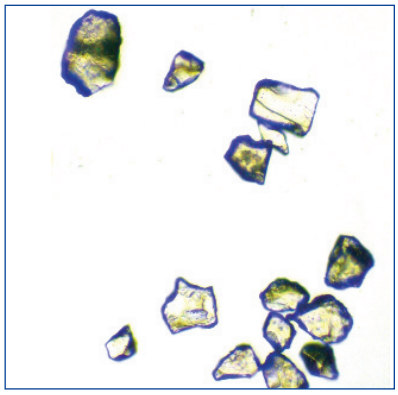
MAS will be presenting a poster on particle shape and the effect on reported particle size at Pittcon. Samples of glass spheres, wollastonite, garnet, and mica were selected because of their differing geometries. The particle size of each material was determined using a variety of techniques including x-ray sedimentation, laser light scattering, electrical sensing zone, dynamic image analysis, and microscopy.

As expected, the various particle size techniques produced different particle size results. The poster explains why that is to be expected and why each result may be "correct" according to the theories and assumptions of the instrumentation.

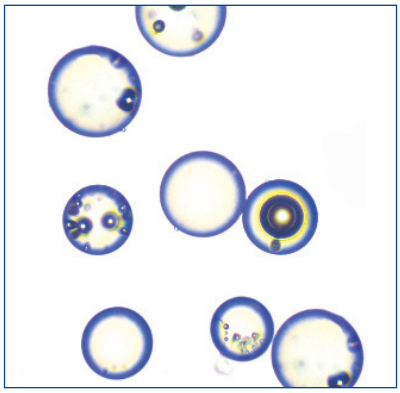
Be sure to stop by the poster session on Wednesday morning to view this poster or send an email to mas@particletesting.com to request a reprint. Poster details are as follows:

"Particle shape and the effect on the reported particle size"

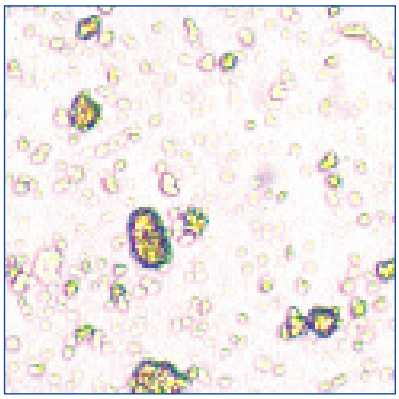
Abstract Number: 1970 - 25P
Session 1970
General Interest:
Chemical Methods
March 5, 2008
Morning Session



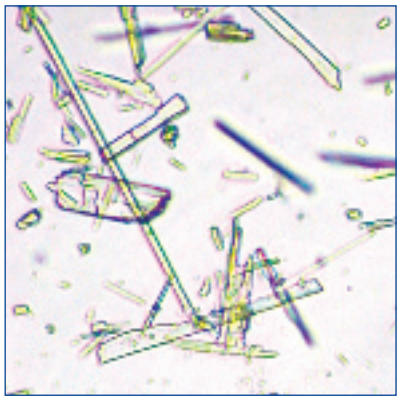
Garnet



Glass spheres



Mica



Wollastonite

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

Pittcon
March 2 - 7, 2008
Booths 2620 -2621
Ernest N. Morial Convention Center
New Orleans, LA

Interphex
March 26 - 28, 2008
Booth 307
Pennsylvania Convention Center
Philadelphia, PA

Powder and Bulk Solids
May 6 - 8, 2008
Booth 2402
Donald E. Stephens Convention Center
Chicago, IL

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.

Instrument Training Course Schedule

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.

2008 Training

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 II 2920
June 3 - 5

AutoPore IV 9500 Series
June 10 - 12

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.

See our website for a complete course schedule.
www.micromeritics.com

Events

Pittcon 2008
March 2 - 7, 2008
Ernest N. Morial Convention Center
Booths 2620 and 2621
New Orleans, LA

Interphex 2008
March 26 - 28, 2008
Pennsylvania Convention Center
Booth 307
Philadelphia, PA

ACS Spring 2008
April 6 - 10, 2008
Ernest N. Morial Convention Center
Booth 322
New Orleans, LA

Hydrogen Symposium
April 24 - 25, 2008
Indiana University
Lafayette , IN

See our website for a complete Event schedule 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 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:
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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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