In This Issue

CERTIFIED ISO 9001 MANUFACTURER
NORCROSS, GA

New TriStar™ 3000 Software Offers New Report Options

Micromeritics announces the release of Version 6.00 software for the TriStar 3000 Surface Area and Pore Size Analyzer. This release incorporates a new report system that provides both increased functionality and versatility. Reports include alpha-s and t-plot. The t-plot report includes Broekhoff-de Boer, Carbon Black STSA, and user-defined thickness curves. BJH adsorption and desorption reports also support the same thickness curve flexibility as the t-plot.

All reports can be manually or automatically rescaled with the added ability to zoom in on fine details. Graphics, corporate logos, and other images can be used to customize reports for papers and presentations, and the all new cut-and-paste feature

The TriStar 3000 provides high-quality surface area and porosimetry measurements on solid materials by using the technique of gas adsorption. This easy-to-operate, tabletop instrument is designed to analyze up to three samples simultaneously for optimum throughput. Its speed and accuracy make it an ideal instrument for research and quality control appli-

cations, as well as for industries that include pharmaceuticals, cosmetics, paints, pigments, food products, and high-tech ceramics. The TriStar 3000 features include:

- Three analysis ports, up to three samples can be run simultaneously

- A 2.75-liter dewar that allows for an extended overnight analysis

- Performs three BET surface area measurements in approximately 20 minutes

- Measures surface areas as low as 0.01 m^2/g

- Incremental or fixed dosing routines can be used to specify up to 1000 data points, allowing minute details of the isotherm to be recorded

ISO 9001-2000 Customer Satisfaction Surveys

Micromeritics is soon to be recertified as an ISO 9001-2000 certified company. ISO is the short name for the International Organization for Standardization, a worldwide organization whose goal is to promote the development of standardization to facilitate the international exchange of goods and services. ISO sets operational and quality control standards for businesses in over 125 countries around the world.

ISO certification is important because it is recognized world-wide as an accepted standard of quality. When companies can accurately document their quality systems, they can compare their systems against a recognized quality standard and improve what they offer to customers. Also, because ISO certification is an understood standard, companies can use it to gauge and select the vendors or subcontractors they work with.

One requirement of ISO 9001-2000 certification is to track customer satisfaction so that we can continue to improve our process-es through customer feedback. This is why 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:
http://micromeritics.com/customer_service/customer_surveys.aspx. A valid Micromeritics product serial number will be required to submit the survey.

DataMaster™ Software for the ASAP 2020

Micromeritics' DataMaster software allows you to perform data reduction offline, using data collected from Micromeritics gas adsorption analyzers, including the new ASAP 2020. DataMaster allows you to compare and analyze data from different instruments and overlay the information on the same graph.

It provides great flexibility in choosing reduction constants that best fit a particular application. Data reductions such as BET and Langmuir multipoint surface area, BJH mesopore structure, and micropore structure by t-Plot, MP Method, Dubinin-Ashtakov, Dubinin-Radushkevich, or Horvath-Kawazoe may be performed and reported. If installed, DFT Plus® data reduction may be done for the entire adsorption isotherm.

Micromeritics Worldwide Sales & Marketing Meeting

Early this March Micromeritics played host to our international distributors and direct sales representatives at our corporate headquaters in Norcross (Atlanta) Georgia. The purpose of this meeting was to provide updated training and information about many of our new products, and to discuss our future goals for the company.

Micromeritics has recently chosen a new color scheme for our instrumentation and has developed a new website that is designed to provide more information about applications. Plans are also underway to provide a website directed to customers using the services of our Materials Analysis Laboratory.

The sales meeting, along with the many other changes underway at Micromeritics, is an effort to continue to provide the quality products, information, and services that we have become known for over the last 40 years.

Micromeritics 21 CFR Part 11 Software

Micromeritics is pleased to introduce 21 CFR (Code of Federal Regulations) Part 11 software. This regulation was developed in cooperation with pharmaceutical companies and the U.S. Food and Drug Administration (FDA) to reduce Time-to-Market of new pharmaceutical products.
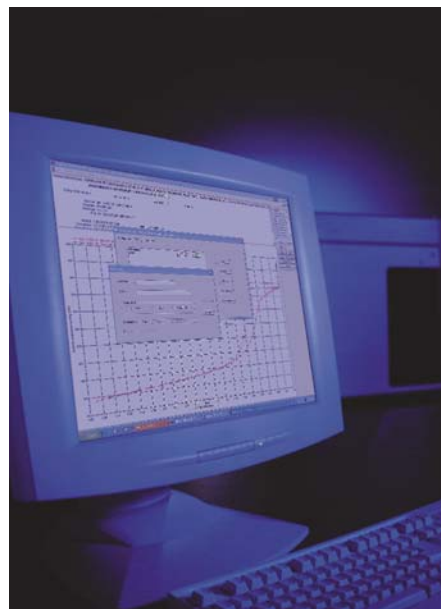
The regulation applies to all systems that manage "records in electronic form that are created, modified, maintained, archived, retrieved, or transmitted, under any requirements set forth in agency regulations." It applies to all data management systems used in pharmaceutical applications covered by the rule; therefore, it applies to all instrumentation that produces data managed by these systems.

The regulation requires the creation of Electronic Records and the implementation of Electronic Signatures in order to provide validation of the authenticity and integrity of the analytical data, and also to provide electronic signatures for verification and certification of that validation. Micromeritics software assures that the integrity of the compliance process is not compromised. User levels are assigned at the appropriate level (usually a lab manager or systems administrator), and once an operator logs in, he or she can only perform analytical functions appropriate for their particular level of security.

Micromeritics 21 CFR Part 11 software helps meet the requirement that all data systems be validated to ensure accuracy, reliability, consistent intended performance, and the ability to discern invalid or altered records. System access is limited to authorized individuals. Secure, computer-generated, time-stamped audit trails are an integral part of the software program.

Individuals are assigned a unique, electronic signature which is applied to electronic records. These signatures cannot be removed, copied, or transferred to falsify an electronic record.

IQ/OQ Validation

Micromeriticcs is also taking an active part in instrument validation by providing Installation Qualification (IQ) and Operational Qualification (OQ) documentation. The IQ/OQ protocol help assure that analytical instruments are manufactured within a reliable validation program, that they are properly installed, and that they function according to the manufacturer's specifications.

Determination of the Density of Small and Irregular-Shaped Samples of Sound and Degraded Waterlogged Woods

Authors: Ines Dorina Donato, Marcella Mulè Cascio
University of Palermo – Dipartimento Di Chimica Fisica, Viale delle Scienze - Parco d'Orleans II -
Pad. 17, 90128 Palermo - Italy

Determining the density of wood can often prove to be difficult. Wood is a porous material which may include gases (i.e. air or water vapour), liquids (i.e. water or preservative agents), solids (i.e. salts, reinforcing agents), or substances that cause an alteration either in the sample mass or its volume. Humidity affects all the properties of wood, especially its density, so it is necessary to specify the percent of humidity in the wood submitted for density analysis.

If degradation has been provoked by insects or fungus, it is possible to determine the volume of a sample of known mass and afterwards measure the density of the damaged wood (Donato 2002). Determining density is more difficult if the degraded wood has been impregnated with water from submerged finds or wet sites as the sample undergoes a substantial shrinkage process during the drying phase. The evaluation of the degradation level is a fundamental parameter to start an effective conservation process; the experts in the field used to refer to a parameter named "conventional density," R, expressed in g/cm^3 and obtained by the followings:

$$R = (u_{max}/100 + 1/1.5)^{-1}$$

where u_{max} is the maximum humidity percent incorporated by the sample and 1.5 is the density of the cellular walls obtained by measurement of sound woods (Roger 1990).

Thus defined, the conventional density does not require a knowledge of the volume; it is based on the assumptions that all voids are filled with water, all the cellular structures shrink the same way (corresponding to the amount of water), and, ultimately, the density of the cellular walls is the same for all the wood species. Some experimental observations emphasize that the density of the cellular structures of wet woods is lower than that of sound woods. This is due to the loss of some substances related to the specificity of the residing place. The chemical composition, the percent of lignin, cellulose and hemicellulose and their densities are all meaningful issues.

Although the difference in the density of the cellular walls is generally small for woods showing high humidity levels, obtaining a rapid and accurate "effective" density of a small and irregular-shaped sample is difficult. The aim of this work is to develop a method for accurately determining the density of wood that is either sound or degraded, and of either small size or irregular shape.

The Experiment

The apparent density (d_{app}) is the ratio between the mass of a prismatic core sample in humidity environmental equilibrium condition, and its volume obtained by means of calibration; the density of the cellular structure (d_p) is given by the ratio between the mass of the sample and the volume effectively occupied by the cellular structure. In the past, pycnometers for solid materials used mercury to determine density, but mercury had the disadvantage of being highly toxic.

Using low surface tension liquids, such as ethanol, can lead to false results because of its penetration inside the wood, provoking shrinkage of the cellular cavities. Recently some modifications to the most commonly used techniques have been proposed: a) silica powder has been used instead of liquids and b) the sample has been coated with paraffin to avoid liquid absorption (Park 2000). Either way, the measurements have reflected scarce reproducibility and large mistakes.

Volume and density determinations of wood samples have been performed by the University of Palermo (Department of Chimica – Fisica) using the AccuPyc 1330 from Micromeritics. The AccuPyc

is based upon the gas (He) displacement technique. The sample cell of the instrument can be kept at controlled temperature; and it is possible to perform measurements on small irregular-shaped fragments whose weight is not lower than 0.5 g. Volume and density of the sample can be obtained with reproducibility of $\pm 2*10^{-3}$ g/cm^3. Thanks to the rapidity of the measurement, it has been possible to perform 30 determinations for each sample for a more appropriate elaboration of the experimental data. The samples were adequately prepared accordingly to either their nature (sound or degraded) and to the parameter to evaluate (d_{app} or d_p).

Methodology developed for the determination of the density of irregular-shaped sound woods for which it is difficult to determine the macroscopic volume.

The d_p value for samples of irregular shape is provided automatically by the AccuPyc. The instrument determines the volume and calculates density using the entered sample mass.

In the same way d_{app} can be determined provided the measurement is performed only after filling all the pores and cavities of the wood structures with proper blends of impregnating agents. The samples, previously weighed, were impregnated with a blend, 40% by weight of polypropylene glycol 425 in polyethylene glycol 1500 at 45 °C. Volume determinations were then made at 25 °C as the blend used became a solid. The effect of the impregnation is observed with a

microscope, performing a "spot test in situ" with cobalt thiocyanate ammonium, while using an image analyser (Image, Pro Plus). No variation in the size of the sample subsequent to the impregnation is observed. The "real" volume of the impregnated sample was next determined by theAccuPyc. D_{app} is obtained by the ratio between the weight of the wood sample and its volume after the impregnation. In order to have good reproducibility of the measurements, it is necessary to quickly remove, prior to solidification, the external layer of the reinforcing agent so that the volume is only related to that of the impregnated wood sample.

Determinations of d_{app} have been performed over wood cores of various taxon; the values are consistent with the ones obtained by traditional methods, that is, the determination of the macroscopic volume of prismatic cores.

Methodology used for the determination of the density of wet degraded wood cores.

The samples of wet degraded woods are retained in deionised water at 4 °C, in sealed vessels to avoid contamination due to bacteria or spores. These woods are generally very difficult to treat due to their low consistency.

D_{app} is determined by the ratio between the mass of the wood material and the volume of the sample impregnated by water, as determined by the AccuPyc. It is possible to determine the amount of wood material contained in the core under analysis, symbolized

by u_{max}. u_{max} is determined by the ratio between the weight loss of the sample due to dehydration (18 hours at 105 °C) and the mass of the dry sample.

Over the same sample, dried using the procedure described above, d_p is determined. In fact, using the mass of the wood sample, the instrument determines the volume and density. The determinations have been performed with samples of wet wood, coming from the lacustrine area of Fiavè-Carera and kindly supplied by Servizio Beni Culturali della Provincia Autonoma di Trento.

Results and Discussion

The volume of the cavities (V_c) and the porosity (Z %) percent are obtained from the values of d_{app} and d_p; Z %, defined as the ratio between V_c and the volume of one gram of wood (V) permits an estimation of the voids in the wood matrix:

$$V_c = (1/d_{app} - 1/d_p)$$

Z % = 100 * (V_c/V) from which Z % = 100 * (1 − d_{app}/d_p)

Furthermore, it is possible to determine the effectiveness of the impregnation technique (E %) from the percent of the voids filled by the impregnant agent:

$$E \% = 100 * (m_{impr}/d_{impr})/V_c$$

where m_{impr} and d_{impr} are, respectively, the mass and the density of the impregnant.

The values of d_{app}, d_p, V_c, Z % and E %, obtained from measurements on core sound woods of

different taxon, are reported in Table 1.

In evaluating the results, it can be observed that differences between various species of woods are related to either the d_{app} values and the d_p values, confirming that these latter ones depend on the nature and the amounts of the substances of the cellular walls.

The porosity of the wood is not only that of the cellular lumen but also of the inner voids of the walls, of the micro fibril and the elemental fibril. The determination of this parameter is fundamental for predicting the permeability of the wood to liquid products such as preservatives, paints, and glues.

The percent of the voids filled has to be correlated to either the chemical characteristics of the impregnating liquid or to the porosity and the anatomy of the wood (omoxilo, eteroxilo).

Table 2 reports the values of d_{app}, d_p, Z %, u_{max}, R and Δ % relevant to the samples of degraded wood. One sample (not identified) is very much degraded and it has not been possible to identify the species.

In evaluating the results, it can be observed that the values of d_{app} of specimens of degraded wood are functions of the humidity percent values: the higher the values of u_{max} the lower the values of d_{app}. The values of d_p depend on the nature of the wood and on the level of degradation and it is not absolutely correct to consider only one value for d_p. The differences observed, Δ %, between the value of the density experimentally determined and conventionally determined have to be correlated with either u_{max} or the type of wood. Since the determinations require very small amounts of sample, it will be possible to perform them on various points of the body of the find in order to get more detailed information about its state of conservation.

Conclusions
The results obtained show that the AccuPyc 1330 He pycnometer from Micromeritics is an ideal instrument for analysing those small wood finds. By this method, it is possible to remove only a small piece of material for which precise investigation can be performed.

Table 1. Data of sound woods.

Taxon	d_{app} gcm^{-3}	d_p gcm^{-3}	Vc cm^{-3}	Z%	E%
Fir Tree	0.505 ± 0.002	1.433 ± 0.002	1.28	65	41
Chestnut	0.558 ± 0.001	1.398 ± 0.005	1.08	60	48
Oak (not aged)	0.749 ± 0.001	1.354 ± 0.005	0.60	45	70
Oak (aged)	0.724 ± 0.001	1.413 ± 0.003	0.67	49	76
Beech	0.701 ± 0.001	1.427 ± 0.003	0.73	51	52
Beech (evaporated)	0.701 ± 0.001	1.396 ± 0.004	0.71	50	41

Table 2. Data of degraded woods.

Taxon	d_{app} gcm^{-3}	d_p gcm^{-3}	Z%	u_{max}	R	Δ %
Not Identified	0.080 ± 0.001	1.340 ± 0.001	94	1224	0.077	3.75
Oak	0.090 ± 0.002	1.390 ± 01770.002	94	1073	0.088	2.22
Beech	0.088 ± 0.002	1.418 ± 0.002	94	1095	0.086	2.27
Larch	0.168 ±0.003	1.410 ± 0.001	88	548	0.163	2.98
Pine	0,140 ± 0.003	1.441 ± 0.002	90	696	0.131	6.43
Yew	0.159 ± 0.003	1.372 ± 0.001	88	600	0.150	5.66

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.

Training

AutoChem II 2920
September 9-11

Gemini
September 16-17

SediGraph 5100
November 4-6

AutoPore IV
October 7-9

ASAP 2020 Chemisorption
June 10-12, November 11-13

ASAP 2020 Physisorption
June 3-5, November 11-13

TriStar 3000
August 26-28

Events

American Ceramic Society
April 27 - 30, 2003
Gaylord Opryland
Nashville, TN

AAPS Workshop on Particle Size Analysis
April 30 - May 2, 2003
Crystal Gateway Marriott
Arlington, VA

Catcon 2003
May 5 - May 6, 2003
The Westin Oaks
Houston, TX

International Powder & Bulk Solids Technology Forum
May 6 - May 8, 2003
Pheasant Run Resort
St. Charles, IL

ACHEMA
May 19-24, 2003
Country D Hall 5.1 G8
Frankfurt Germany

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:
Laurel Whitmire, Editor
The microReport
MICROMERITICS
One Micromeritics Drive
Norcross, GA 30093-1877
laurel.whitmire@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. 4 Bei Wa Road, Hai Dian District
Beijing 100081, P.R., CHINA
Tel: (+86) (0)10-6848-9371
Fax   (+86) (0)10-6848-9371

Micromeritics France
Zaet St. Maximin
181, rue Henri Bessemer
F-60100 Creil, 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
Telephone   (+49) (0)2166-98708-0
Fax   (+49) (0)2166-98708-88

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

Micromeritics N.V./S.A.
Oude Haachtsesteenweg 107, C3
B-1931 Brussels, Belgium
Tel: 011-32-2-743-3974
Fax: 011-32-2-743-3979

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

mi micromeritics®
The Science and Technology of Small Particles™

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

CERTIFIED ISO9001 MANUFACTURER
NORCROSS, GA