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## Micromeritics to Showcase a Variety of New Instruments and Instrument Options at Pittcon 2010

Pittcon, one of the world's premier annual conferences devoted to laboratory science and instrumentation, takes place from February 28 through March 5, 2010, at the Orange County Convention Center in Orlando, FL.

Micromeritics, located in Booth 1721, will introduce significant developments in material characterization instrumentation. Attendees who visit our booth will have the opportunity to see firsthand our new instruments and instrument options, as well as the opportunity to speak personally with our technical and sales representatives.

The new generation **Saturn DigiSizer® II High-Definition Digital Particle Size Analyzer** utilizes a state-of-the-art CCD detector containing over three million detector elements, Mie theory, and unique design and data reduction features, to provide an extremely high level of resolution and sensitivity not avail-



able in other laser particle sizing systems. The Saturn DigiSizer II analyzes and measures particles over a range of 40 nanometers to 2.5 millimeters. Features such as fully automated sampling and low-volume sample handling units and a complete system for circulating the dispersing liquid/sample mixture through the cell and reservoir make the Saturn DigiSizer ideal for a wide range of production, research, and quality control applications.

The **MicroStar Residual Gas Analyzer** features a quadrupole mass spectrometer that complements the Micromeritics AutoChem 2910, 2920, and 2950 dynamic chemisorption analyzers and is ideally suited for pulse chemisorption studies and temperature-programmed reactions. It allows the user to follow the evolution of residual gases from the AutoChem and use the resulting data to determine reaction kinetics, product selectivity, and reaction yields; all of which are vital components of a successful catalyst development program. Unlike traditional temperature-programmed desorption or reduction experiments where only one active gas is monitored, the MicroStar provides continuous monitoring of reaction products (up to 128 different masses).

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Vapor / Water Vapor Option  
for the ASAP 2020

Micromeritics' ASAP 2020 is now available with new vapor and water vapor options. This added versatility provides researchers and formulators the capability to characterize the surface area and porosity of materials both before and after exposure to vapors or moisture. The **ASAP 2020 vapor option** allows characterization of large samples including tablets, pellets, granulated food, powders, and desiccant packs and is compatible with a wide range of vapors including benzene, toluene, xylene, ethers, aldehydes, alcohols, and amines. The ASAP 2020 water vapor option is an ideal tool for performing a detailed characterization of pharmaceuticals, food, cosmetics, and many other materials that sorb moisture. The moisture adsorption isotherm may be used to understand the water activity of a material which may influence chemical stability, microbial activity, flow properties, compaction, hardness, and dissolution rates. Unlike traditional gravimetric moisture analysis, the ASAP 2020 water vapor option allows the user to characterize large samples.

Micromeritics' **ASAP 2420 enhanced micropore option** extends the low-pressure measurement capabilities of the instrument and allows enhanced performance for characterizing microporous materials using nitrogen, argon, carbon dioxide, hydrogen, and other fixed gases. The ASAP 2420 design allows for high-performance/high sample throughput

with six independently operated ports for adsorption analysis and a fully automated sample preparation module with twelve independently controlled stations. The multiport capabilities of the ASAP 2420 provide an ideal platform for research and quality control laboratories requiring high-throughput material characterization including: BET surface area, pore volume distributions, pore area distributions, micropore analysis, and NLDFIT modeling of adsorption isotherms.



HPVA-100  
High-Pressure Volumetric Analyzer

Since the formation of Particulate Systems and its acquisition of the Particle Insight Dynamic Image Analyzer, this Micromeritics brand has added a number of new products and announced a strategic collaboration with Surface Measurement Systems Limited.

The **HPVA-100 High-Pressure Volumetric Analyzer** uses the static volumetric method to measure high-pressure adsorption isotherms with hydrogen, methane, carbon dioxide, and other gases. Applications include catalysts, zeolites, activated carbons, carbon nanotubes, hydrides and metal-organic frameworks. The operating pressure of the HPVA ranges from high vacuum to 200 bars. Sample temperatures during analysis can range from cryogenic to 500 °C.



The **Magnetic Analyzer MA-1040** detects minute quantities of iron in a wide range of materials including high-purity glass used in fiber optics to plastics used in wiring insulation. With sensitivity as low as 0.00001% magnetic content, the MA-1040 can also be used to detect low magnetic iron levels in food, precious gems, pharmaceuticals, and many other materials.



DVS - Dynamic Vapor Sorption  
Intrinsic

The collaboration of Particulate Systems with Surface Measurement Systems Limited gives distribution rights to Particulate Systems for key Surface Measurement Systems products in selected regions of the world. These products include the **DVS Dynamic Vapor Sorption Systems**, which utilize dynamic gas flow and the gravimetric technique to produce high-resolution adsorption and desorption isotherms of water and organic vapors on solid materials.



## Outsourcing Analytical Services

By: Adam Keith, Business Manager  
Micromeritics Pharmaceutical Services

Strategic outsourcing partnerships are transforming the way that companies conduct business in this era of rapid technological advances and global competition. Outsourcing allows a company to focus, and stay focused, on its core competencies while contracting the expert services of another business on an as-needed basis. The old adage that any one person cannot be an expert in everything is proving to be true for companies as well.

During these uncertain economic times, the initial motivation for establishing outsourcing partnerships is often to achieve cost savings. Over time, however, it becomes clear that there is an even more significant value to the partnership relationship. Companies are finding that while reducing costs, they experience gains in productivity as well. They are able to utilize the best talent and technology available for the job at hand – without being limited to their own in-house expertise and resources. In essence, outsourcing offers “a better bang for the buck.”

With this in mind, Micromeritics established Micromeritics Pharmaceutical Services (MPS) as an outsourcing partner to the pharmaceutical industry. MPS utilizes the instrument technologies and expert scientific services of Micromeritics, with its 45+ years of experience, and incorporates complementary analytical techniques from other sources as well. The benefit to our customers is that MPS now provides analytical services as a DEA-licensed, FDA-registered, cGMP/GLP-compliant contract analytical laboratory.

During the last ten years, the pharmaceutical industry has experienced significant changes. While pharmaceutical companies are reorganizing and consolidating to trim overhead and cut costs, their research is actually expanding. The exploration of new, more targeted drug candidates is exploding

in the wake of advances in technology and increasing consumer demand. The result is more potential products that must undergo stringent and newer testing. In light of these changes, the FDA is finding that it looks favorably on the outsourcing of analytical testing, due to the high standards that independent laboratories can establish and maintain.

While many activities involved in analytical testing are easily outsourced, successful outsourcing ultimately depends on the outsourcing partner’s expertise in the required areas of analysis. MPS provides expertise in various areas, including preformulation material characterization and excipient screening, as well as follow-up assistance to researchers in overcoming any obstacles related to these basic material properties. MPS makes available many characterization tools that can lead to predicting in-process behavior and to determining physicochemical properties, bioavailability, and other pharmacokinetic phenomena. MPS also makes available the early preformulation analytical testing that is critical to the development of the numerous new drug candidates and the challenges they introduce.

As important as the science itself is a methodology that produces robust and precise documentation and validation. In all of its services, MPS collects and produces data throughout the analytical testing process. Much of the data collected early in the process can lead to significant cost savings to the customer during later stages of the development cycle. As standard procedure, MPS processes can be reliably replicated. In fact, MPS and the customer can make arrangements for testing methods to be transferred as intellectual property to the customer, when circumstances warrant.

As an independent lab, MPS houses a full array of instrument

technology from top manufacturers. With its state-of-the-art instrumentation, MPS is already the material characterization outsourcing partner of choice for many pharmaceutical researchers. Please contact us if your organization is interested in learning more about the analytical services we provide through an outsourcing partnership. Information about Micromeritics Pharmaceutical Services is available online at [www.micRx.com](http://www.micRx.com) or call 770-624-3393.

### Micromeritics Introduces Materials Characterization Webinar Series

Micromeritics has begun offering webinars that focus on applications, theory, and instrumentation for a variety of materials characterization techniques. The first in this series of technical presentations highlighted the Elzone electrical sensing zone particle size/count technique and its pharmaceutical applications. For those who missed it, an archived video of the webinar can be found on our website. Visit our home page weekly for announcements on Micromeritics’ future webinars and a link to our archived webinar page.

## Volumetric Gas/Vapor Sorption Analytical Technique – A Useful Tool in the Development and Production of Pharmaceutical Powders and Tablets

By: Radha Vippagunta  
Novartis Pharmaceuticals Corporation

In today's pharmaceutical development environment, scientists are looking to new analytical test methods for help in resolving numerous issues that can arise in all phases of drug development and production. For example, an accurate prediction of the stability of finished products must be determined, therefore it is necessary to identify and quantify any batch-to-batch variations that occur in the processing of powders. It is also important to study the capping effect on coated tablets and variations in dissolution rates of intact tablets. The volumetric gas adsorption analytical technique can help resolve a variety of issues that may arise early and continue in the drug development process through production.

Commonly, different batches of the same active pharmaceutical ingredients (APIs) can exhibit different physical and mechanical properties. The source of batch-to-batch variability of these APIs can be difficult to ascertain. We used a Micromeritics ASAP 2020 Accelerated Surface Area and Porosimetry analyzer and the BET surface area test method to characterize different batches of API that exhibited different flow properties. We determined that the amorphous content of the batches varied. From this information, we were able to conclude that the varying amorphous content of the batches was the source of the variability in flow behavior.

Using the ASAP 2020 water sorption accessory, one can study the effect of accelerated stability conditions on intact tablets by examining isotherms measured at different temperatures. The capping effect on coated tablets or

variations in rate of dissolution rates can easily be determined.

### Batch-To-Batch Variation (1)

As particle size distribution of most APIs and excipients are controlled by milling, it is reasonable to assume that each face of a crystal, each edge and defect, will have different fracture patterns and surface properties due to different distribution of various functional groups on these fracture surfaces. It is known that amorphous materials or crystalline materials with some amorphous content will relax with changes in surface energy as a function of time. Hence the surface energy of a powder will be continually changing as the forces in the system change, and thus determine the behavior and property of the material.

Surface energy is commonly quantified using a contact angle goniometer. The contact angle is large if the surface is hydrophobic. Hydrophilicity is indicated by smaller contact angles and higher surface energy. Although contact angle methods for powders have limitations, the mean values obtained are still meaningful when correlated to the functionality. However, concerns over this methodology have resulted in the growth of the use of gas and vapor-based probes to assess surface energy for powders.

Powder vapor interactions can be studied chromatographically using

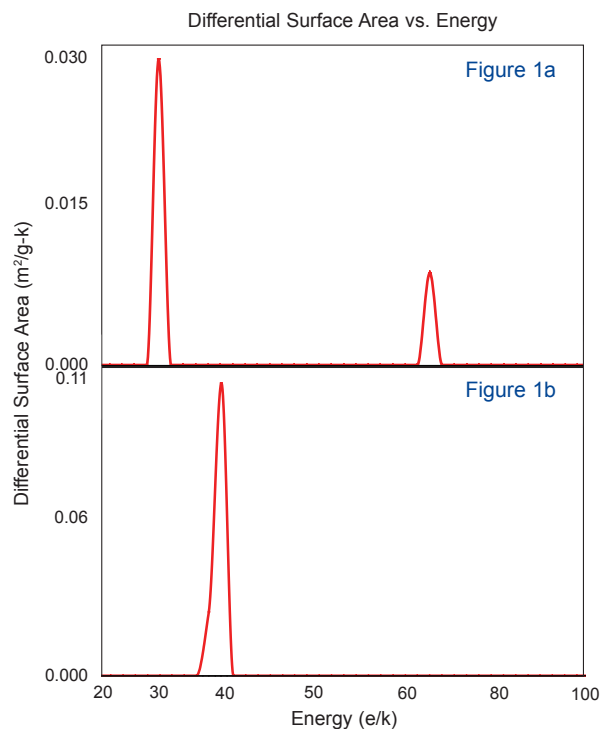


Figure 1: Surface energy difference between different batches of same compound as estimated by DFT model  
a) Freshly prepared Metformin HCl batch  
b) Thermodynamically stable Metformin HCl batch

inverse gas chromatography. IGC is an interesting technique which can give information on the dispersive and polar surface energy components. Thus, IGC can be applied to solids with multiphase mixtures and chemical heterogeneity. In such cases, the variation of the surface energy arises due to the influence of impurities or the limited surface accessibility.

Using the nitrogen gas adsorption isotherm, the accessibility problem can be nullified and also the influence of surface morphology of the chemically similar solid at nanometer scale can be easily studied

with accuracy. Besides just determining surface area by application of the well known Brunauer-Emmett-Teller (BET) model to gas adsorption isotherms, additional information (e.g., porosity and surface energy due to heterogeneity) pertaining to surface properties can be obtained. Density functional theory can be applied to calculate the surface heterogeneity in energy of low surface area or non-porous materials, which are common in pharmaceutical materials. The relevance of surface heterogeneity is likely only in monitoring relative changes from lot-to-lot and differences between crystal and amorphous forms. In this example, a study was carried out to determine the cause for the batch-to-batch variation in flowability of a Metformin HCl drug substance during processing, despite no apparent differences in physical or chemical properties. On investigating available batches, it was noticed that time between synthesis of drug substance and

use in drug product manufacture (age) could be a factor. The surface energy estimated by density functional theory (DFT) showed that the freshly manufactured batch has a higher energy than the thermodynamically stable batch. The surface energy values indicate that the freshly manufactured material has more active surface, making it interact better with other surfaces as an activated powder, thus showing poor flowability.

#### Predicting the effect of accelerated stability conditions on the intact tablets

Using the ASAP 2020 water sorption accessory, one can easily study the effect of accelerated stability conditions such as 25 °C/60RH or 40 °C/75RH on intact tablets by carrying out the isotherms at these temperatures. The capping effect on the coated tablets or variations in rate of dissolution rates can be studied well in advance. In this example, the increase in water uptake at

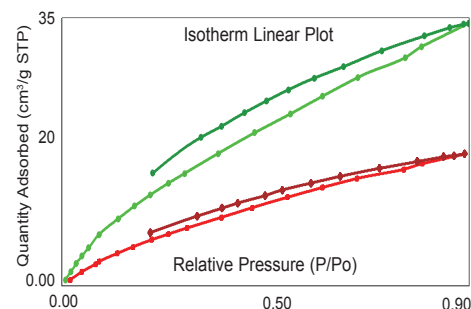


Figure 2: Water sorption isotherms for intact tablets at 25 °C and 40 °C

higher temperature resulted in lowering the T<sub>g</sub> (glass transition) of the drug substance and gelling occurred in the dissolution media, thus drastically lowering the dissolution rates.

(1) "Application of surface area measurement for identifying the source of batch-to-batch variation in processability," Radha R. Vippagunta, Changkang Pan, Ronak Vakil, Vindhya Meda, Richard Vivilecchia, and Michael Motto; *Pharmaceutical Development and Technology*, 1097-9867, first published on 25 February 2009.

## Micromeritics Announces Instrument Grant to the Department of Chemical and Biomolecular Engineering at Rice University

Micromeritics' Grant Selection Committee has selected the recipient of its grant award for the fourth quarter of 2009. An AutoChem II Catalyst Characterization System has been awarded to the Department of Chemical and Biomolecular Engineering at Rice University, Houston, TX.

According to Dr. Michael Wong, Principal Investigator and Associate Professor of Chemical and Biomolecular Engineering at Rice University, "My laboratory works at the interface of Chemical Engineering, Chemistry, and Materials Science, with a focus on designing functional nanoparticle-based materials for catalytic, encapsulation/delivery, and energy applications. The materials of interest that will be studied with the AutoChem II include specially synthesized supported metal oxide nanoclusters for heterogeneous

gas-phase catalysis, bimetallic catalysts for liquid-phase chemical reactions, and carbon nanotube-based structures and metal-organic framework/coordination polymer materials for gas adsorption. Projects include the synthesis and characterization of novel metal oxide supported material with enhanced acidic properties, Pd/Au nanoparticles for water remediation applications, catalytic oxidation of carbon monoxide by gold nanoparticles, and many others."

According to Preston Hendrix, president of Micromeritics, "This program is designed to promote and advance the acquisition and use of particle characterization instrumentation in non-profit universities and institutions where other means of funding are not generally available. We are very proud and excited to present this award in an ongoing grant program to support impor-

tant research." Mr. Hendrix has appointed a special Grant Selection Committee to ensure the success and effectiveness of this program.

Micromeritics' Instrument Grant Program is intended to provide particle characterization instruments to non-profit universities and research organizations for the purpose of fostering and supporting meritorious research projects. A maximum of one instrument/integrated system will be awarded per calendar quarter. The next grant decision will be made prior to 03/31/10. The next application deadline is also 03/31/10 for a 06/30/10 grant decision.

Applications may be submitted at any time in accordance with the application instructions and will remain active for a period of one year from the date of submission. Visit Micromeritics' website for a detailed grant description, application requirements, and application.

MAS recently acquired a new particle size analyzer utilizing a light obscuration technique. This instrument reports particle size distribution of most materials between 0.5  $\mu\text{m}$  and 400  $\mu\text{m}$ . This instrument technique is specifically required for determining the particulate matter in injectable solutions as described in USP methods <788> and <789> and measures particle size in both aqueous and organic solutions. Its accuracy is extremely sensitive with the capability to detect just a few particles in solution. The test number for this new application available from MAS is 005-73. Contact us today if you have questions or need more information.

**Good News - Prices for MAS services in 2010 will remain the same as in 2009 and 2008.** MAS is dedicated to helping customers find solutions to their problems, removing roadblocks as we can. Cost is an issue for all companies in this challenging economic environment, and we have looked for ways to manage our costs so as not to raise your cost of using our services.

Last year MAS exhibited at Pittcon for the first time, and our booth was a huge success! Once again, we will attend this annual event, and look forward to seeing you at **Pittcon**, in Orlando, FL, from February 28 through March 5. Stop by **Booth 3920** to say hello.

## MAS Events

**Informex**  
Feb 15 - 19  
San Francisco, CA

**Pittcon**  
Feb 28 - March 5  
Orlando, FL

**Interphex**  
April 20 - 22  
New York, NY

**Powder and Bulk Solids**  
May 3 - 6  
Chicago, IL

## Meet the newest member of MAS

Thao Nguyen is the most recent Lab Analyst to join MAS. Originally from Vietnam, Thao has lived in Atlanta since 1994. She earned a Bachelor of Science degree in Biology from Georgia Tech in 2007. While studying at Georgia Tech, she worked as a Pharmacy Technician at CVS pharmacy, where her responsibilities sparked her interest in material characterization and analysis. Initially, Thao managed projects utilizing BET surface area and pore size analysis by gas adsorption for MAS. Currently, her work focuses on projects related to particle sizing. Thao is a meticulous scientist whose analytical skills provide benefits to both customers and her MAS co-workers. Please feel free to contact Thao with any questions regarding particle size analysis.



## Free Surface Energy and Water Vapor Sorption Analyses Offers

For a limited time, Micromeritics Analytical Services is offering either **one free surface energy analysis** or **one free volumetric water vapor sorption analysis** of your sample on a Micromeritics gas adsorption analyzer. This is an excellent, no-obligation opportunity to determine the surface free energy or water uptake of your sample in a controlled environment. We invite you to take this opportunity to explore

some of the advantages that gas adsorption technology offers over other techniques. For the surface energy offer, visit Micromeritics Analytical Services website (www.particletesting.com), complete our Sample Submission Form, and simply write "free surface energy offer" as the type of payment. For the water sorption analysis, complete the form, include an analysis temperature (typically 0 - 40 °C), and enter "free water vapor offer" as the type of payment. We'll take

care of the rest. Once you receive your results, please feel free to contact us for a complimentary data consultation. Additionally, we are currently offering a 25% discount on our surface energy analysis service via inverse gas chromatography (IGC). We are also offering a 25% discount for gravimetric water vapor sorption on the Surface Measurement Systems DVS. Contact us at (770) 662-3630 or [mas@particletesting.com](mailto:mas@particletesting.com) for more details.

## Instrument Training

Micromeritics provides basic start-up training for most instruments during installation. However, when operators wish to maximize their proficiency and broaden their capability with their Micromeritics instrument, more advanced training is needed. To achieve this goal, Micromeritics offers for most instruments targeted classes in which customers may expand their ability and improve their understanding by learning from the experts who designed their instruments. These classes, periodically held at Micromeritics headquarters in suburban Atlanta, Georgia, include:

### Theory Overview

Learn about the science upon which each instrument is based and how this science applies to successful sample preparation, analysis, and results interpretation.

### Detailed Operations

Effective sample file creation, use of analysis parameters, and manual sample entry are all covered. Increase efficiency and learn to use the full power and flexibility of the operating software.

### Automatic Analyses

Develop correct analysis procedures to optimize collection of accurate, reproducible data. Much class time is spent performing hands-on analyses in a controlled, tutorial environment.

### System Utilities

Learn instrument software utilities which help manage sample files and directories, protect data, and select system options.

### Report Optimization

Learn to configure reports and obtain the most useful information, as well as improve report comprehension.

### Troubleshooting

Learn techniques to quickly locate and resolve instrument and software problems.

### User Maintenance

Under the guidance of a Micromeritics Maintenance Specialist, practice routine maintenance procedures which improve operation, reduce downtime, and increase data accuracy.

### Course Enrollment

Training courses last from two to four days and are designed to provide hands-on, performance-based instrument skills and knowledge. Small classes allow individual instruction and peer interaction. Course materials include a Study Guide, a complimentary copy of *Analytical Methods in Fine Particle Technology*, and a wealth of other educational material. A Certificate of Completion is awarded to each trainee.

Visit [www.micromeritics.com](http://www.micromeritics.com), select Service Center, and then Training Center for additional information, to enroll for a specific course, or for a complete course schedule.

Visit our website for the complete 2010 course schedule and registration.  
[www.micromeritics.com](http://www.micromeritics.com)

## 2010 Training

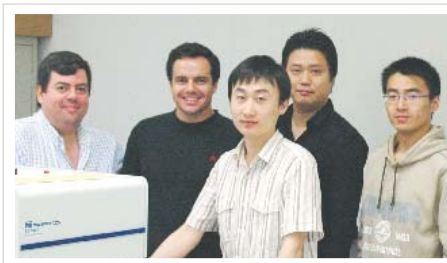
Gemini™ 2390  
March 23 - 24

ASAP™ 2020  
Chemi and Physi  
April 13 -16

AutoPore™ IV 9500  
April 20 - 22

Saturn DigiSizer® II  
June 8 - 10

For additional information or to register for the class of your choice, visit [www.micromeritics.com/Service-Center/Training-Class-List.aspx](http://www.micromeritics.com/Service-Center/Training-Class-List.aspx) or phone 770.662.3607. Early registration is recommended since class space is limited.



Students attending a recent Elzone course

## Events

### Pittcon 2010

February 28 - March 5, 2010  
Orange County Convention Center  
Orlando, FL  
Booth #1721

### ACS Spring 2010

March 21 - 25, 2010  
The Moscone Center  
San Francisco, CA  
Booth #1116

### Analytica 2010

March 23 - 26, 2010  
Messe München GmbH  
Munich, Germany

### Interphex 2010

April 20 - 22, 2010  
Jacob K. Javits Convention Center  
New York, NY  
Booth #426

Visit our website for a complete Event schedule [www.micromeritics.com](http://www.micromeritics.com)

## THE microReport

The microReport newsletter is published by Micromeritics Instrument Corporation.  
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(770) 662-3654

Subscriptions are free to particle technology users and can be started simply by writing to The microReport editor.

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## 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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The microReport  
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4356 Communications Drive  
Norcross, GA 30093-1877  
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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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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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