

# microREPORT

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# Micromeritics Product Spotlight

## ASAP 2460 Surface Area and Porosity Analyzer

The ASAP 2460 Surface Area and Porosity Analyzer incorporates a unique expandable system designed for high-performance and high sample throughput. The base ASAP 2460 is a two-port master control unit. For more throughput, additional two-port units can be connected to the master unit expanding the system to either a four-port or six-port analyzer.

### Analytical Versatility with Superior Throughput

- All analysis ports can be operated independently and concurrently, allowing the user to load and unload samples at any time, regardless of the analysis stage.
- An analysis in excess of 60 hours can be performed without refilling the dewar.
- With a master unit and two auxiliary units, BET surface area analyses utilizing six parallel runs can be achieved in as little as 30 minutes.
- Servo control for dosing and evacuation provides a higher degree of gas management and speeds collection of data points through reduction of dosing overshoot.
- Up to five different non-reactive adsorptives, plus helium for free space, can be attached to the analyzer simultaneously.
- Long-duration dewars and isothermal jackets assure a stable thermal profile along the length of both the sample and saturation pressure (PO) tubes throughout lengthy analyses.
- Intuitive MicroActive software combines user defined reports with the ability to interactively evaluate isotherm data.
- User selectable data ranges through the graphic interface allow direct modeling for BET, t-plot, Langmuir, DFT interpretation, and new advanced NLDFT methods.
- An innovative dashboard monitors and provides convenient access to real-time instrument performance indicators and maintenance scheduling.

[Click here to read the new ASAP 2460 brochure.](#)

## Low Surface Area Measurement (Krypton) and Dedicated Micropore Options

In addition to the standard 2460, low surface area krypton and micropore models are available. Low surface area (Krypton) model includes the addition of a 10-mm Hg transducer and permits accurate measurement of very low surface area on materials such as API (active pharmaceutical ingredient), powdered metals, etc.

The micropore model includes the addition of a 1-mm Hg transducer which extends the low pressure measurement capabilities and allows enhanced performance for characterizing microporous materials using nitrogen, argon, carbon dioxide, hydrogen, and other fixed gases. The transducer also increases pressure resolution in the range necessary for micropore analysis.



### Benefits:

- Fully automated modular system optimized for walk-up sample screening
- High throughput with two, four, or six independent analysis stations
- BET surface area measurements in as little as 30 minutes
- Dosing options of maximum volume increment or dosing over specified pressure ranges
- Analysis temperature can be entered, calculated, or measured
- Equilibration option allows user to specify equilibration times for different parts of the isotherm
- Low surface area and micropore options
- Innovative MicroActive software with advanced NLDFT modeling

# Magnesium Stearate: The Effects of Hydration State and Degassing on Isotherms and BET Surface Area

Darren Lapham  
Operations Manager, MCA Services

Magnesium stearate, MgSt, is one of the most commonly used excipients in solid dosage pharmaceuticals, added to powder blends primarily as a lubricant. However, the addition of MgSt also causes unfavourable effects, such as alteration of dissolution characteristics, reduced tablet hardness and strength. Therefore, it is critical to ascertain the optimum MgSt loading for a particular powder blend. This is complicated by variations in both the physical and chemical nature of commercial MgSt. MgSt exists in several crystalline phases and a number of hydration states: anhydrous and the mono-, di- and tri-hydrates. MgSt di-hydrate in a plate-like morphology appears to provide particularly good lubricity.

The measurement of BET surface area ( $S_{BET}$ ) is commonplace in the pharmaceutical industry and is routinely applied to characterisation, formulation, manufacture and QC. The most common analytical technique used for  $S_{BET}$  measurement is volumetric nitrogen adsorption followed by application of the Brunauer-Emmett-Teller (BET) equation to a low pressure region of a gas adsorption isotherm. The measurement of MgSt  $S_{BET}$  is, however, known to be problematic.

In this work, nitrogen adsorption/desorption isotherms of commercial MgSt batches of different hydration states have been investigated. Atypical characteristics, i.e. those deviating from usual isotherm characteristics, have been identified and depend on MgSt hydration state. Specifically these are:

1. Poor linearity of the adsorption isotherm below approximately 0.2 P/Po. For calculation of  $S_{BET}$  this results in BET transform plots of poor linearity, thus low correlation coefficients. BET C-values also tend to be low ( $<10$ ).
2. Low pressure hysteresis between adsorption and desorption isotherms continuing below 0.45 P/Po.
3. Deviation of isotherm shape from BDDT classifications with increasing adsorption volume toward saturation commencing at lower P/Po than usually observed.



### Experimental

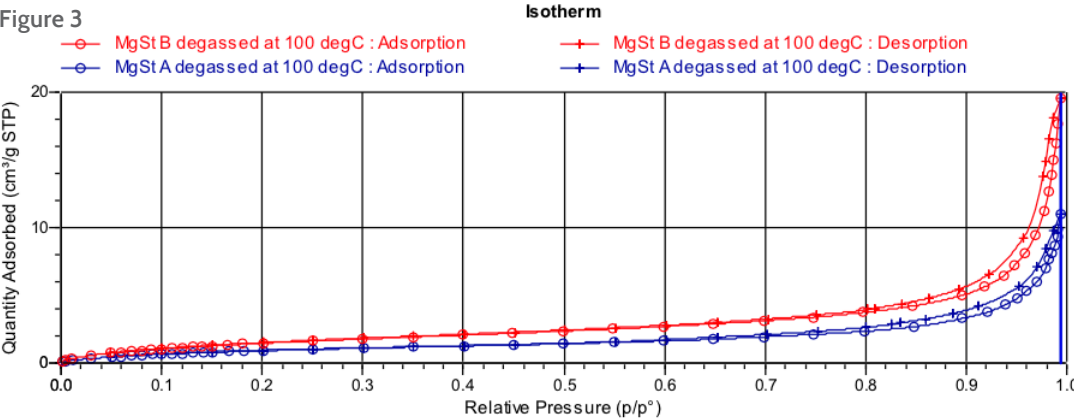
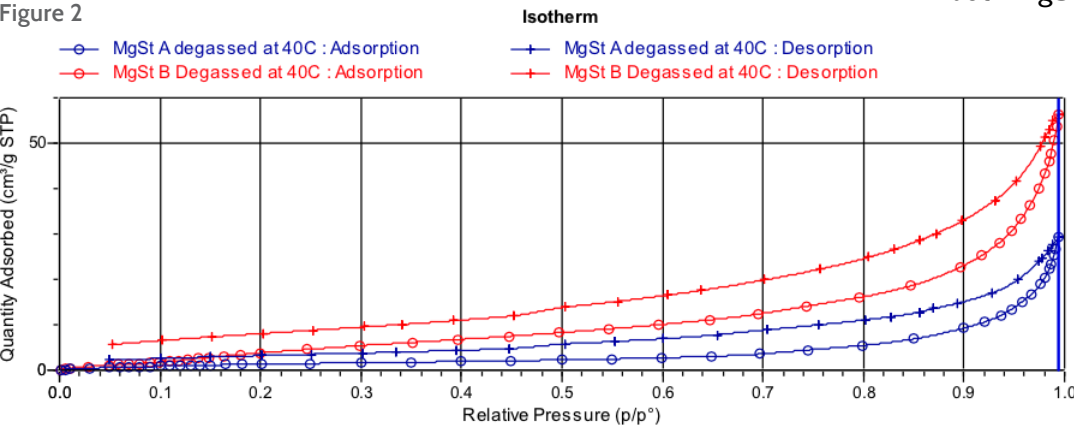
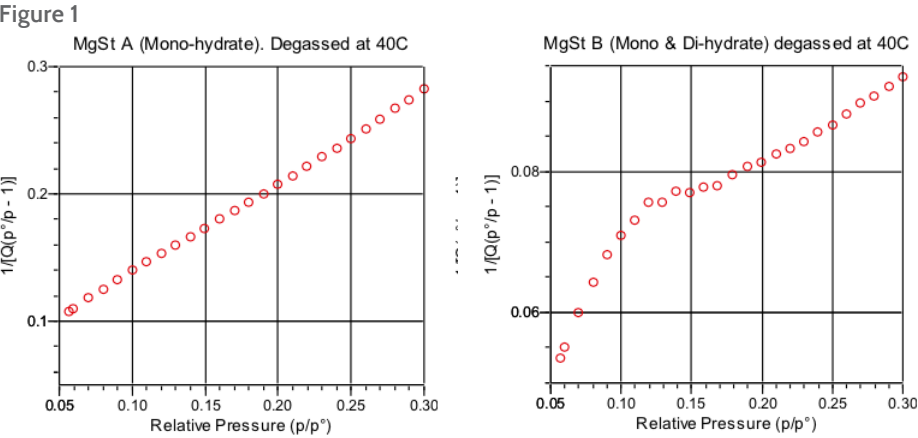
Two commercial MgSt batches, A and B, have been analysed. These were first characterised for hydration state using thermogravimetric analysis (TG). TG was undertaken at Micromeritics Analytical Services, using a TA Instruments Q5000 IR instrument. MgSt A was found to be predominantly mono-hydrated whilst MgSt B is of mixed mono- and di-hydrated material.

### Gas Adsorption

Nitrogen adsorption/desorption isotherms were measured using a Micromeritics TriStar II Plus instrument. Sample masses were 0.3 g +/- 0.01 g and samples were degassed to constant mass using a Micromeritics VacPrep 061 having an ultimate applied vacuum of 100 mTorr. Filler rods and isothermal jackets were used and equilibration times were 5 seconds throughout.



$S_{BET}$  measurement of MgSt is known to be problematic. Specifically this is due to difficulty in obtaining a linear BET transform plot within the classical BET P/Po range, resulting in plots of low correlation coefficient. Figure 1 shows BET transform plots for samples degassed to constant mass at 40°C to remove unbound water but without dehydration. The mono-hydrated sample, MgSt A, shows a BET transform plot of good linearity throughout the P/Po range, with a correlation coefficient of 0.9997. Conversely, the mixed mono- and di-hydrate sample, MgSt B, shows a plot of poor linearity over



the same P/Po range with a correlation coefficient of 0.9502. The highest correlation coefficient for this sample (0.9988) is only obtained when the BET range is limited to 0.22 – 0.30 P/Po. Figure 2 shows full isotherms of both samples degassed at 40 °C. Classification of the adsorption isotherms according to BDDT models is not straightforward. Above approximately 0.2 P/Po both are most similar to a type II isotherm, although the increase in adsorption volume towards saturation commences at a lower P/Po than would usually be expected. Below 0.2 P/Po MgSt A exhibits type II character which allows for easily-constructed BET transform data. However, MgSt B is non-linear in this region and appears to be intermediate between types II and III.

A common feature of both isotherms is the failure of adsorption and desorption isotherms to close at lower P/Po, resulting in low pressure hysteresis (LPH) which is of greater magnitude for MgSt B. It has been shown previously (Lapham, 2017) that these atypical features are caused by the presence of hydrated water within the MgSt structure. When both MgSt A and B are dehydrated

through degassing at 100°C, typical type II isotherms of consistently lower sorption volumes and with very little LPH are obtained, as shown in figure 3.

Samples of each batch were analysed for  $S_{BET}$  calculated in the range 0.05 – 0.30 P/Po after degassing at temperatures in the range 30 – 100 °C.

For both samples,  $S_{BET}$  decreases as samples become dehydrated and is accompanied by an increase in BET C-value and, for MgSt B, improved correlation coefficient. These values are detailed below.

The reliability of  $S_{BET}$  data must also be

assessed. The correlation coefficient provides a good indication of the suitability of the P/Po range applied, a value >0.999 being expected in most cases. The BET C-value provides an indication of the strength of adsorption, typically a value <10 is considered to show weak interaction and thus potential inapplicability of BET theory to  $S_{BET}$  calculation.

Conclusions

The presence of hydrated water within the MgSt structure causes atypical isotherm characteristics. However, the effects on  $S_{BET}$  depend on hydration state. The adsorption isotherm within the BET range of MgSt mono-hydrate is of typical character and  $S_{BET}$  can be calculated with confidence. However, adsorption within the BET range of di-hydrated MgSt is atypical, resulting in BET transform plots of low correlation coefficient

and BET C-value.  $S_{BET}$  must therefore be considered with caution. Given that  $S_{BET}$  is significantly higher for MgSt B in the hydrated state, the possibility of an over-estimation of  $S_{BET}$  must also be considered. Calculating  $S_{BET}$  from a portion of the adsorption isotherm providing reasonable linearity (> 0.22 P/Po), as recommended by various pharmacopeia improves correlation coefficient but without significantly changing  $S_{BET}$  or BET C-value. Again  $S_{BET}$  should be considered with caution. These concerns are of particular importance given that di-hydrated MgSt is favoured for lubricity effects. Further, this work demonstrates the importance of establishing characterisation methodologies for use in the QBD approach to development and manufacturing in pharmaceutical processes and products.

Reference: Darren P Lapham and Julie L Lapham. Gas adsorption on commercial magnesium stearate: Effects of degassing conditions on nitrogen BET surface area and isotherm characteristics. International Journal of Pharmaceutics 530 (2017) 364-376.

Degassing Temp(°C)	MgSt A			MgSt B		
	$S_{BET}$ (m²g⁻¹)	BET C-value	Correlation Coefficient	$S_{BET}$ (m²g⁻¹)	BET C-value	Correlation Coefficient
30	5.4	12	0.999	23.9	3	0.949
40	5.6	12	0.999	24.0	5	0.943
50	5.6	12	0.999	19.7	4	0.953
60	5.3	12	0.999	15.7	5	0.961
70	4.3	16	0.999	7.7	18	0.999
80	3.5	18	0.999	6.6	18	0.999
90	3.5	20	0.999	6.3	20	0.998
100	3.2	19	0.999	5.9	19	0.998



Dr Darren Lapham Ph.D has been Operations Manager at MCA Services, a UK contract analysis laboratory and consultancy for physical materials characterization since 2002. Darren specializes in techniques of gas adsorption, mercury porosimetry and chemisorption. Darren obtained his Ph.D in Physical Chemistry from the University of Essex in 2000 and has undertaken post-doctoral research at Greenwich University and the University of Cambridge, working on semi-conducting and solid state gas sensors and rechargeable battery technology. Currently, Darren is researching the physical characterization of pharmaceuticals and battery components.

For more information on pharmaceutical characterization click here.

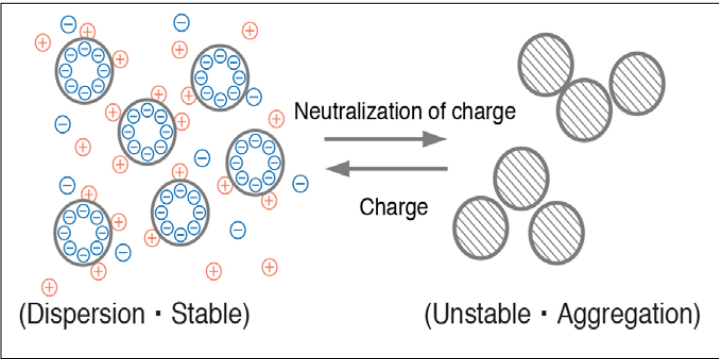
# Measuring Zeta Potential of Non-aqueous Suspensions

Jack G. Saad

Submicron particles suspended in liquid medium are regularly utilized in many industries, from food and pharmaceutical production to lubricants for machinery. The stability of these suspensions is vital to their function and shelf-life. The typical way to characterize the stability of a suspension is to measure the zeta potential of the suspension.

So what is zeta potential and how is it used to determine suspension stability?

Particles and the suspending medium always have some kind of charge associated with them. For the particles, it is the surface charge and surrounding ions. For the medium, it is either polar forces of the molecules or the presence of surfactants or other charged molecules. In the event the particles are suspended in a non-polar solvent, there is still a chance that the particles will be attracted to each other and form agglomerates, or larger particles. The formation of larger particles will eventually lead to the separation of the suspension. Some stabilizing additive may be needed to cause the particles to repel each other, thereby stabilizing the suspension.

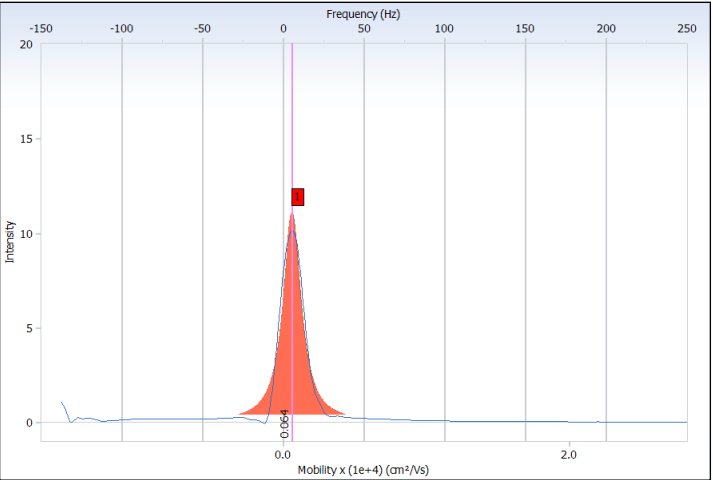


[Click here to read the new NanoPlus HD brochure.](#)

To measure zeta potential, a current is passed through the suspension and the electrophoretic mobility of the particles is measured by electrophoretic light scattering (ELS). Since it is difficult to pass a current through low polarity to non-polar organic solvent, i.e. low dielectric constants, an analysis cell must be optimized to make this measurement.

It is not appropriate to treat organic media, such as benzene, toluene, or tetrahydrofuran, as if they are as conductive as water.

The NanoPlus sold by Particulate Systems, a Micromeritics brand, has a specialized accessory cell compatible with organic media and optimized electrode positioning to make these types of zeta potential measurements possible. It is made of high quality quartz for maximized optical performance. The user friendly software calculates zeta potential using the Hückel calculation model. This differs from measurements in aqueous media, where zeta potential is calculated using the Smoluchowski model.



Diluent	Temperature (°C)	Refractive Index	Viscosity (cP)	Dielectric Constant
Water	25	1.3330	0.8904	78.3
Acetone	20	1.3589	0.326	20.7
Isopropanol	30	1.3770	1.77	18
Methanol	25	1.3312	0.547	33.6
Benzene	20	1.5011	0.652	2.28
Toluene	20	1.4969	0.590	2.4
Cyclohexane	20	1.4264	0.696	2.02

\*Property data presented in NanoPlus Operator's Manual from multiple sources. Please see manual for additional values and references.





# Using the HPVA to Analyze Hydrogen Storage Potential of Metal Organic Frameworks at High Pressures

Determining the hydrogen storage capabilities of materials such as Metal Organic Frameworks (MOFs) and other highly microporous materials is an important undertaking in the modern push for a hydrogen economy. An efficient method of hydrogen storage is a critical aspect in the development of hydrogen fuel cells. Hydrogen gas has a high energy density by mass but a low energy density by volume when stored as a compressed gas, making it unfavorable for hydrogen storage. Maintaining hydrogen in a liquid state (20 K at atmospheric pressure) also is not energy efficient. Storing hydrogen in a solid material by adsorption is the best alternative, requiring less volume than compressed gaseous hydrogen and consuming far less energy than required to liquefy hydrogen. Dosing high pressure hydrogen onto high surface area MOFs for storage as an adsorbed gas is a highly desirable process due to the high hydrogen energy density obtained and the availability of reversible adsorption.

Four commercially available MOFs produced by BASF were analyzed with Particulate Systems' High Pressure Volumetric Analyzer (HPVA) to determine their hydrogen storage potential. Those MOFs are: Basolite C300, a copper-based organic framework; Basolite F300, an iron-based organic framework; Basolite Z1200, a zinc-based organic framework; and Basolite A100, an aluminum-based organic framework. Approximately 500 mg of each MOF was placed under vacuum and slowly heated up to 200 °C for a period of 12 hours (Z1200 was only heated to 100 °C to prevent degradation of the sample) using the HPVA degas

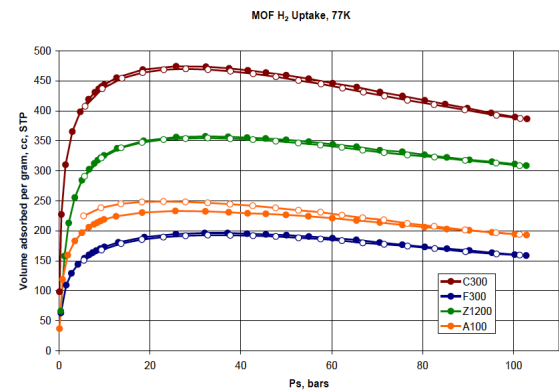


Figure 1: An overlay of the excess isotherms generated from the analysis of various MOFs with hydrogen at 77 K. The solid circles represent the adsorption isotherms and the hollow circles represent the desorption isotherms.

port. All four samples were analyzed at liquid nitrogen temperature (77 K) in a liquid nitrogen bath, utilizing the cryogenic option for the HPVA, up to pressures of 100 bar. An isothermal jacket was used to maintain the cryogenic temperature zone of the samples during analysis. At 77 K, each MOF showed different amounts of hydrogen uptake; C300 adsorbed the most while F300 adsorbed the least. A plot of the isotherms generated from the analyses is shown in Figure 1.

The isotherms displayed in Figure 1 exhibit a phenomenon in which the adsorption reaches a maximum and then declines as the pressure increases. This phenomenon is due to the increasing density of the hydrogen in the pores of the material at elevated pressures. The density of the adsorbing gas (H<sub>2</sub>) inside the pores (a function of pore size) is far greater than the density of a non-adsorbing gas (He). Since the calculated amount of gas in the sample cell is based on the density of helium and its resulting free-space volume (including the volume inside the pores), the amount of free gas in the sample cell is overestimated. When using the static volumetric method, like that of the

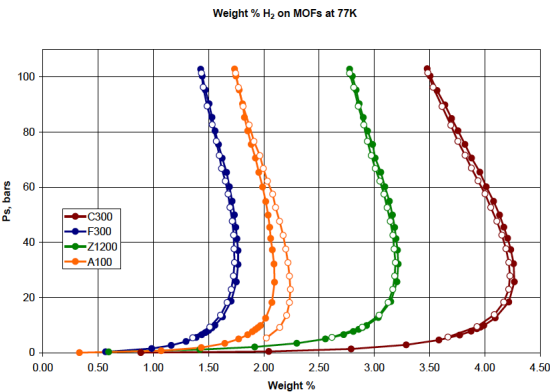


Figure 2: Weight percentage plots of various MOFs analyzed with hydrogen at 77 K.

HPVA, a maximum in the isotherm may be observed. This is used to create the excess isotherm as shown in Figure 1. To generate the absolute isotherm, the density of the gas and the volume of the pores must be included in the calculations. Since the pore size and distribution of these types of materials are not readily

available to most users, the excess isotherm will suffice and is commonly reported for adsorption isotherms.

An alternative method to see the storage capacity of materials from the excess isotherm is to view the amount of gas adsorbed as a function of the sample weight. The target weight percentage of hydrogen uptake for storage purposes is between 7% and 8%. Figure 2 shows an overlay of the weight percentage plots based on the isotherms displayed in Figure 1.

Since the Basolite C300 adsorbed the most hydrogen at 77 K, it was also analyzed at two additional temperatures. For one analysis, an ice bath was used to maintain the sample at 0 °C. For the second, a recirculating water vessel was used to maintain the sample temperature at 30 °C. For these two experiments, the sample was dosed with hydrogen to pressures up to 200 bar, the full extent of the pressure range obtainable with the HPVA. The excess isotherms are shown in Figure 3 and the weight percentage plots in Figure 4.

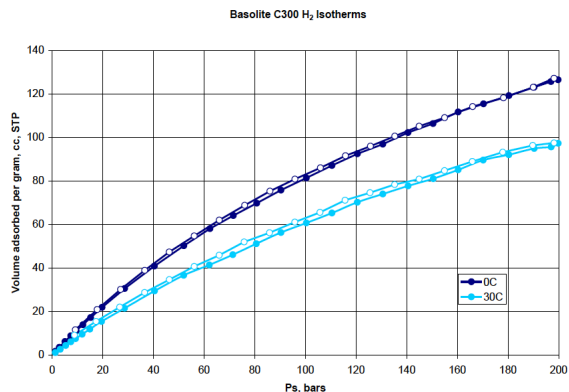


Figure 3: Hydrogen uptake on Basolite C300 at 0 °C (dark blue) and 30 °C (light blue).

When reviewing the data in Figures 1 through 4, it is clear that the HPVA is a powerful tool for evaluating the hydrogen storage potential in MOFs and other microporous powders. The HPVA, with its wide temperature range (from cryogenic to 500 °C) and its ability to dose up to 200 bar of pressure, is perfect for analyzing samples under extreme conditions while providing accurate data.

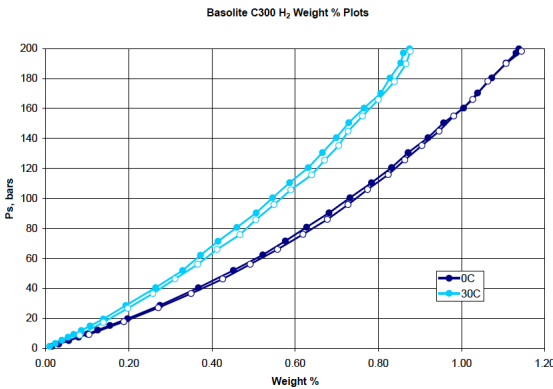


Figure 4: Weight percentage plots of hydrogen on Basolite C300 at 0 °C (dark blue) and 30 °C (light blue).



For more information on HPVA, [click here](#).

# Using the Modified PID EFFI Microactivity Reactor for the Characterization of Catalysts via Thermo-Programmed Techniques

Simon Yunes, Andrew D'Amico, and Jason Exley

The PID EFFI (Efficiency) microactivity reactor can be re-configured and connected to a mass spectrometer for temperature-programmed and pulse chemisorption techniques used to characterize and test catalysts in situ. This innovative research tool, known as the “Micro Catalyst Characterization and Testing Center” (MCCTC), allows for multiple catalyst characterization experiments and activity testing at pressures ranging from atmospheric up to 100 bar or 200 bar (depending on instrument configuration). Characterization techniques including temperature-programmed reduction, oxidation, and desorption can be used as a point of reference prior to testing the performance of the catalyst. The capability to then follow the transient activity of the catalyst on-line provides important information about the behavior of the catalyst’s active life and deactivation. Recharacterization of the catalyst after activity testing provides critical information about the parameters which potentially cause loss in activity of the catalyst. The combination of characterization and performance testing in a single apparatus provides important and accurate information about catalytic behavior for specific applications.

## The Instrument

Figure 1 shows the MCCTC configuration of the PID EFFI, designed for catalyst characterization and catalytic testing to determine activity and selectivity of the specimen. The essential components of this instrument include mass flow controllers (MFCs) for the carrier and active gases, three (3) six-port valves to dictate flow pathways and pulsing through the reactor, a heated reactor zone able to reach temperatures up to 1100oC using various ramping rates, and a quadrupole mass spectrometer for on-line analysis of gas mixtures. All plumbing, valves, and the liquid evaporator (for cases involving liquid feeds), are enclosed in a hot box heated up to 200°C ensuring excellent mixing of

gases and the prevention of condensation within the system. Heated vapors which condense at atmospheric conditions are cooled and separated from the gas phase using the patented Liquid/Gas separator. These liquid products can be collected for further studies and the dry gas proceeds to the mass spectrometer for composition analysis.

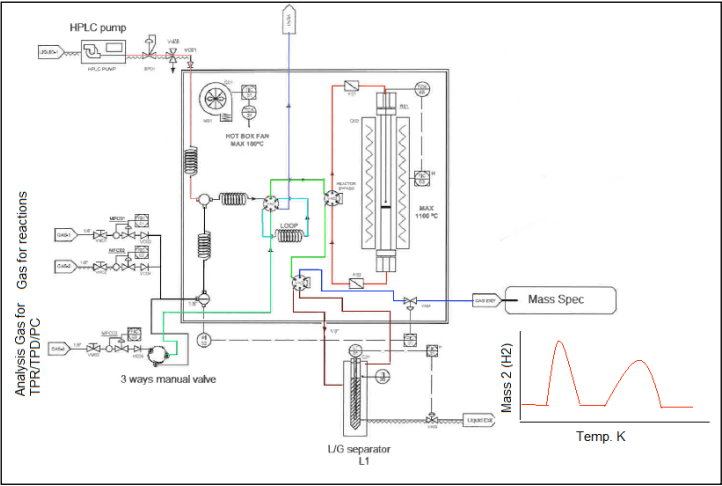


Figure 1: Flow Diagram of the MCCTC Connected to a Mass Spectrometer

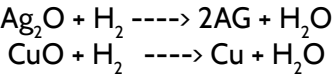
## Experiment 1: Temperature-Programmed Reduction (TPR)

Temperature-programmed reduction is used for characterizing the reducible species existing in the catalyst as well as determining the effect of the catalyst support on the dispersion of active metal(s). In this particular case, the effect of the H2 pressure on the reduction profile of a catalyst was studied by acquiring TPR profiles at two distinctly different pressures, 1 bar and 25 bar. Approximately 80mg of a silver (I) oxide and copper (II) oxide catalyst mixture (50/50 approx.) was selected for this study. A flow of 50 sccm of a 10% hydrogen balance nitrogen mixture was used for species reduction and a flow of 100 sccm nitrogen was supplied as a carrier gas and diluting agent. The

furnace temperature was ramped at a rate of 10°C/min up to 500°C to generate complete TPR profiles.

First, temperature-programmed reduction was performed on the silver/copper oxide mixture at atmospheric pressure like most TPR studies are traditionally executed. The atmospheric pressure TPR profile can be seen in Figure 2 and portrays a sharp inverted peak at 163°C and a broader inverted peak at 320°C, indicating the activation temperatures of the silver oxide and copper oxide, respectively. Next, the same TPR analysis was performed at a pressure of 25 bar.

The 25 bar pressure TPR profile can be seen in Figure 3 and includes a sharp inverted peak at 150°C and a broader inverted peak near 250°C. The inverted peaks show the decrease of H2 in the exhaust gas downstream of the catalyst being reduced. During reduction hydrogen is consumed and combines with the oxygen atoms in the metal oxide following the chemical reactions:



It is clear that the high pressure of H2 over the catalyst significantly decreases the reduction temperature as expected. The increased pressure provides a higher driving force for hydrogen and oxide interaction, specifically regarding porous catalyst supports. This principle is very valuable in industry because reduction of catalysts at lower temperatures greatly decreases the chance of sintering and ultimately allows for higher catalytic activity. Since TPR is a bulk reaction, a quantitative analysis can be made with the mass spectrometer data to determine the total amount of reducible species present in the catalyst.

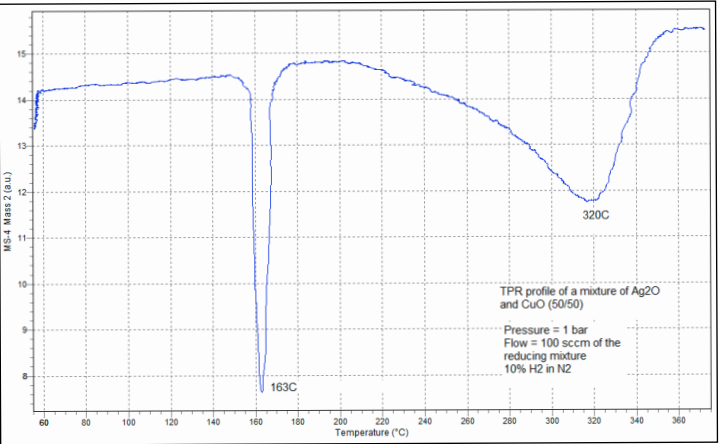


Figure 2: TPR Profile Performed at Atmospheric Pressure

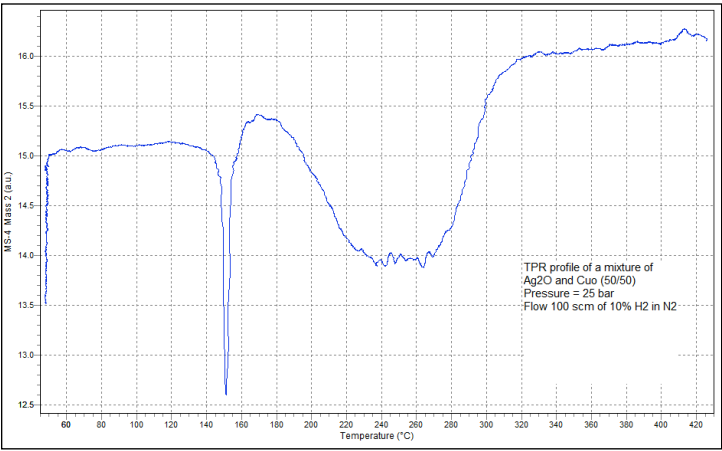


Figure 3: TPR Profile Performed at 25 bar Pressure

## Experiment 2: Pulse Chemisorption (PC)

Pulse chemisorption (PC) is an analytical technique used to determine the dispersion, or accessible active species present on the surface, of a catalyst. An active gas such as CO or, in this case, H2, is pulsed onto the catalyst at room temperature and the quantity of active gas adsorbed is determined. For this experiment, the active gas was pulsed by using a six-way valve with a 0.5 mL STP loop in intervals of approximately 3 minutes. The 10% hydrogen balance nitrogen mixture was fed through the loop at a flow rate of 20 sccm to ensure complete filling within 1 minute. Atmospheric pressure nitrogen was fed at a constant rate of 50 sccm through the system to act as a carrier gas.

Approximately 800mg of 0.5 wt% Pt/Al2O3 was the studied catalyst. Figure 4 is the data obtained from the mass spectrometer during the duration of the experiment and the five separate peaks indicate the five separate pulses. The last three peaks are identical and represent pulses after the catalyst has been saturated and are used as a baseline to backcalculate the amount of gas adsorbed from the first two pulses. Almost all of the active gas is adsorbed in the first pulse and some of the active gas is adsorbed in the second pulse. By integrating the peaks and combining this data with the known pulsed volume, the total amount of adsorbed gas is known. The percent dispersion of the catalyst is determined using the following equation:

$$D_{\%} = \frac{V_{\text{ads}} F W_a}{V_{\text{mol}} M_{\%}} \cdot 100 \cdot 100$$



Where D% is the percent dispersion,  $V_{ads}$  is the total volume of gas chemisorbed,  $F_s$  is the stoichiometry factor, in this case 2 for diatomic hydrogen,  $W_a$  is the atomic weight of the active metal, 195 AMU for platinum,  $V_{mol}$  is the molar volume of the adsorptive (22.414 liters/mol), and M% is the percent of active metal by weight as grams of metal per gram of sample.

The studied catalyst, 0.5 wt% Pt/Al<sub>2</sub>O<sub>3</sub>, is a well known standard used for Micromeritics Chemisorption instruments such as the Autochem II 2920 and has a reported dispersion of 35% ± 5%. The percent dispersion calculated from the data collected by the MCCTC and mass spectrometer yielded 36%.

If the PC experiment is performed at elevated pressures, more active species will be adsorbed and thus, the percent dispersion of the catalyst will be proportionally higher. It is known that catalysts characterized at atmospheric pressure exhibit higher activity than indicated when screened at elevated pressures required for industrial chemical reactions.

Furthermore, other parameters can be determined by the PC technique such as active metal surface area and the size of the active particles. Large active particle size indicates sintering, or growth, of the active particle. Generally, as the active particle size increases, the activity of the catalyst decreases.

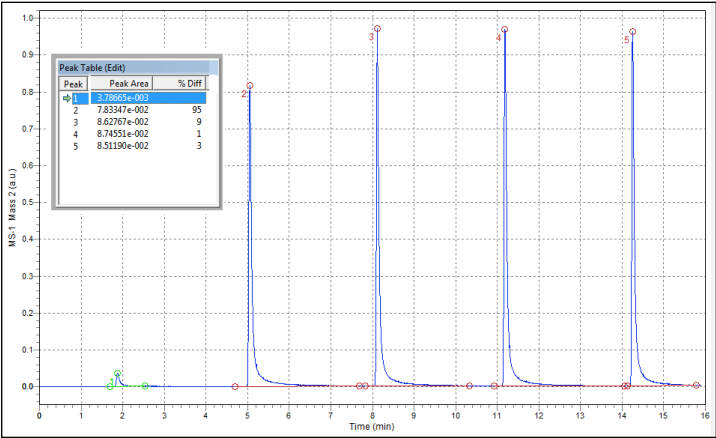


Figure 4: Pulse Chemisorption Profile of H<sub>2</sub> on 0.5 wt% Pt/Al<sub>2</sub>O<sub>3</sub>

Additional Catalyst Characterization Experiments Using the MCCTC Temperature-Programmed Desorption (TPD)

This technique monitors the amount of gas desorbed from the surface of a catalyst during a linear temperature

ramp after the surface has been saturated by an active gas. The strength of interaction of adsorbed molecules bonded to the active species is evaluated and surface coverage may also be quantified. In addition, the portion of adsorbed molecules present on the catalyst surface up to the reaction temperature can be determined. For example, if hydrogen is retained on a catalyst such as platinum up to the desired reaction temperature, the catalyst will be capable of performing the hydrogenation reaction.

To illustrate this analysis, a TPD profile of hydrogen on platinum collected on the Autochem II 2920 is considered here in Figure 5. The Autochem II 2920 uses a thermal conductivity detector (TCD) to quantify the amount of hydrogen in the exhaust stream and the MCCTC paired with a mass spectrometer will provide the same TPD profile. The increasing signal intensity indicates the release of hydrogen from the surface of the catalyst, seen here occurring at three separate temperature peaks. The multiple peaks indicate different active sites of the catalyst; peaks occurring at higher temperatures reflect sites with higher adsorption enthalpies. TPD can be performed at elevated pressures using the MCCTC but should only be considered if the catalyst was reduced at the same pressure.

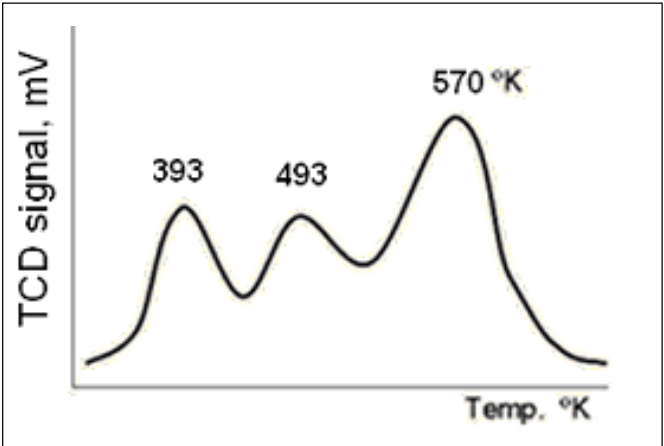


Figure 5: TPD Profile of H<sub>2</sub> on a Platinum Supported Alumina Catalyst

Temperature-Programmed Oxidation (TPO)

This technique is mainly used to determine the degree of reduction of active species present on the catalyst. Combining this technique with TPR allows for estimating the number of regeneration cycles that a catalyst can undergo before completely deactivating.

TPO is also used for characterizing de-NO<sub>x</sub> catalysts

such as V<sub>2</sub>O<sub>5</sub> and TiO<sub>2</sub>. These catalysts are widely used in industry for transforming dangerous NO<sub>x</sub> contamination into other non-pollutant components. Typically these catalysts promote the reaction of NH<sub>3</sub> with NO<sub>x</sub> to safely produce water and nitrogen.

Figure 6 shows an example of a typical TPO profile of the pre-reduced de-NO<sub>x</sub> catalyst, V<sub>2</sub>O<sub>5</sub>. The TPO profile includes three oxidation peaks, two of them well defined at 500°C and 860°C and the third is a “shoulder” near 960°C. Peaks appearing at such high temperatures indicate stability of the reduced species that will be active for the reaction. This particular catalyst was reported to have a 95% conversion rate during activity testing, an exceptional yield for a de-NO<sub>x</sub> catalyst.

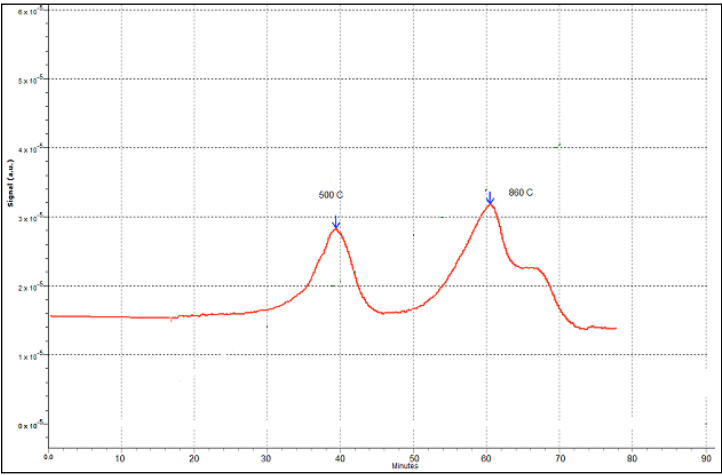


Figure 6: TPO Profile of V<sub>2</sub>O<sub>5</sub>, a de-NO<sub>x</sub> Catalyst



Conclusion

The MCCTC is an innovative piece of equipment that combines almost every simple catalyst characterization technique with catalytic activity testing. This combination reduces overall costs by eliminating the need for multiple pieces of equipment by performing the same tasks in just one instrument. Also, the ability to characterize catalysts at different experimental pressures and temperatures is extremely valuable due to the fact that the catalyst can undergo preliminary characterization and testing using the same industrial reaction conditions.

This fact predicts the catalyst behavior at the pre-set experimental conditions prior to activity testing and can determine the cause of catalyst deactivation through post activity testing characterization. Ultimately the user saves time and money by electing to characterize and test catalytic activity using one system.



For more information on the PID Effi, [click here](#).





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## PARTICLE TESTING AUTHORITY



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Micromeritics Analytic Services serves the needs of the Battery and Fuel Cell industries, chemical, catalyst, coatings, petrochemical, paper, polymer, coatings, minerals, industrial powders and many other application areas.

We provide a comprehensive range of analytical characterization techniques from a single sample to complex method development to new product assessments or to address large scale manufacturing projects.

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### Available Analytical Techniques:

- Particle Size Distribution
- Particulate Count
- Nanoparticle Size
- B.E.T Surface Area
- Pore Size Distribution
- Micropore Analysis
- Pore Volume Distribution
- Total Pore Volume
- Density
- Surface Energy
- Dynamic Water Vapor Sorption
- TGA
- DSC
- Active Surface Area
- Crystallite Size
- High Pressure Adsorption
- Magnetic Content
- Zeta Potential
- Isothermic Heat of Adsorption
- Microscopy
- Method Development
- Method Validation
- Consulting Services
- Percent Metal Dispersion



# The Particle Testing Authority Pharmaceutical Service



Understanding the physical and chemical properties of your drug substances and products is crucial. In today's age of Process Analytical Techniques (PAT) and Quality by Design (QbD), regulatory authorities look favorably upon drug developer partnerships with Contract Research Organizations (CROs) that have specific areas of expertise.

Backed by Micromeritics with 50 years of experience, MPS can be trusted as your materials characterization outsourcing partner for determining the physical properties of pharmaceutical materials, medical devices, nutraceuticals, and other FDA-regulated products. Through the use of advanced analytical testing systems, MPS provides information on your materials that enable you to define and achieve high-quality drug products while optimizing your drug development and production process.

Early identification and understanding of Critical Quality Attributes (CQAs) are essential steps in the process. From a QbD perspective, scientists, researchers, and regulators look for consistency, both in physical and chemical attributes. MPS is here to help you define, specify, and control the critical quality attributes of your materials.

Our areas of expertise include particle size distribution analysis (micron and nano- particles), particle shape and morphology, surface area, surface energy, vapor sorption, porosity, density, thermal analysis, and material flow properties.

Our analytical systems are linked to secure and validated data-handling networks (LIMS), ensuring that results are compiled and delivered as quickly, reliably, and accurately as possible while maintaining compliance with the appropriate standards for analytical methodology, security, and data integrity. Through a secured web portal, our customers can submit paperwork, view project status, and retrieve results.

MPS is a DEA-licensed, FDA-registered, cGMP/GLP-compliant contract characterization laboratory.



# PoroTechnology

## We Are Recognized Experts in Core Analysis

Poro Technology was founded by John Neasham Ph.D in 1995 and has for over 22 years, been providing MICP analysis for a broad spectrum of reservoir and seal rock types. Today Porotech's experienced team of analysts continue to provide the highest quality results for our customers. PoroTechnology provides full evaluations to make the critical decisions necessary for successful recovery of oil and gas potential.

## Industry Expertise

Our staff is comprised of recognized leaders in the evaluation of core sample

## Advanced Capabilities

Includes MICP, Grain Density, Pore Size Analysis, XRD analysis, SEM, Soxhlet Extraction

## Expedited Full Reporting

You will receive a full evaluation report with a turn-around, in most cases, of less than 2 weeks



**MICP- Mercury Injection Capillary Pressure:** Analysis to evaluate the porosity and matrix permeability distributions in unconventional reservoirs



**Sample Preparation - Soxhlet Extraction:** Full service cleaning laboratory with toluene and methanol extractions for proper sample prep prior to instrumental analysis



**Grain Density By Gas Pycnometry:** Obtain the true, absolute, skeletal density of a core sample through non-destructive helium pycnometry



**Pore Size By Gas Adsorption:** Adsorption isotherms will indicate reservoir capacity as well as reveal important information about pore volume, area, and pore distribution



**XRD X-Ray Diffraction:** Provides critical information about rock composition and bulk mineralogy to help in improving the choice of drilling and stimulation fluids



**SEM Microscopy:** Analysis used to understand pore types and their effects on permeability and porosity as well as to investigate the difference between natural and induced fractures





# Tradeshows and Events in 2018

EVENT	DATE
PACCON 2018- Thailand	2/07/2018 - 2/09/2018
Analytica 2018- Germany	4/10/2018 - 4/13/2018
Interphex 2018	4/17/2018 - 4/19/2018
AIChE 2018	4/22/2018 - 4/26/2018
PB&S 2018	4/24/2018 - 4/26/2018
Achema 2018- Germany	6/11/2018 - 6/15/2018
Powdermet 2018	6/17/2018 - 6/20/2018
Ceramics Expo 2018	5/01/2018 - 5/03/2018
GFEC- France	5/15/2018 - 5/18/2018
University of Mississippi Hands on Tableting Technology Course	5/20/2018 - 5/25/2018

# Check Us Out on Social Media

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# How To Reach Us

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