

# AUTOCHEM III

A Catalyst Characterization  
Laboratory in a Single  
Analytical Instrument

[micromeritics.com/AutoChem](http://micromeritics.com/AutoChem)



**mi** micromeritics®

The AutoChem from Micromeritics is the most widely used and highly cited system for catalyst reactivity characterization because it is also the most automated, highly accurate system for chemisorption and temperature-programmed reactions.

The all new AutoChem III meets and exceeds that performance with a design that will **save you hours a day, make the most sensitive, reproducible measurement, and enhance operator safety.**

Pulse Chemisorption	Metal Dispersion	Metal-Supported Catalysts
TPR	Metal Surface Area	Acid or Base Catalyzed Reactions
TPO	Active Surface Area	Oxide or Zeolite Catalysts
TPD	Crystallite Size	Advanced Battery Anode Materials
TPSR	Heat of Desorption	Fuel Cell Catalysts
Dynamic B.E.T.	Activation Energy	
Breakthrough Curve	B.E.T. Surface Area	



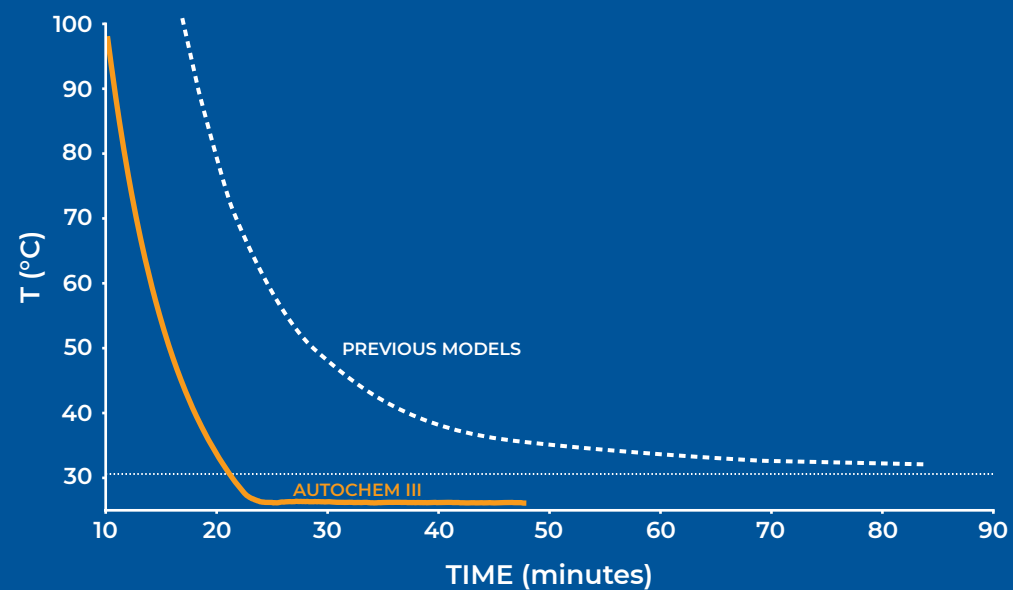


# QUICK & EASY

The new AutoChem III is designed to make key operations quick and easy, saving you **hours a day** so you spend less time making measurements and more time making progress.

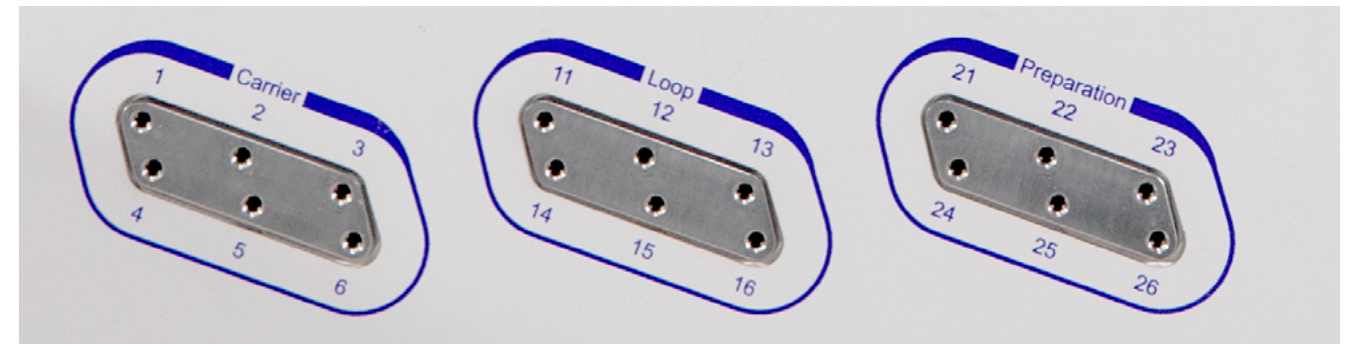
## RAPID TURNAROUND TIME WITH AUTOCOOL

The NEW AutoCool is an integrated gas-fed system that rapidly cools sample tubes before and during experiments. AutoCool is typically **30 minutes faster** than alternative systems and requires no liquids or external support.



## NEVER PREPARE ANOTHER VAPOR CAPTURE SLUSH BATH

The NEW AutoTrap effectively traps vapor and requires **no manual slush bath preparation**. Traditional methods for vapor capture require the time-intensive process of slush-bath preparation through the manual mixing of liquid nitrogen with isopropanol. The zeolite bed of the AutoTrap effectively captures vapors, can be used for several experiments without interruption, and can be regenerated *in situ*.



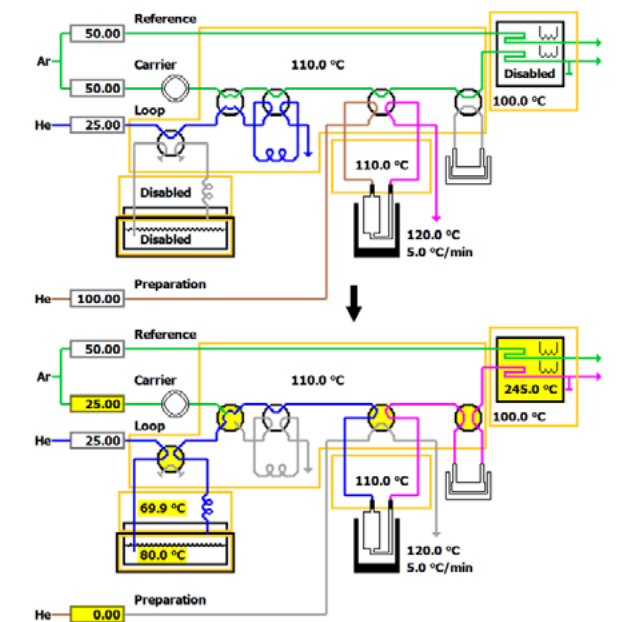
## READY TO RUN WITH 18 AVAILABLE GAS STREAMS

Don't waste time reconnecting and switching gas lines: have what you need ready when you need it. The AutoChem III has 18 available gas streams so you're **always ready to run your next reaction**.

Having the right blended gas ready also means you won't introduce errors from poorly designed external gas connections, and you won't compromise data accuracy by blending gases, which unnecessarily introduces error from mass flow controllers.

## PROGRAM WHAT YOU ENVISION, VISUALIZE WHAT YOU HAVE PROGRAMMED

The new MicroActive method editor features an intuitive process illustration that shows the programmed state of the instrument at every step of the method so you can see that your method matches your vision.



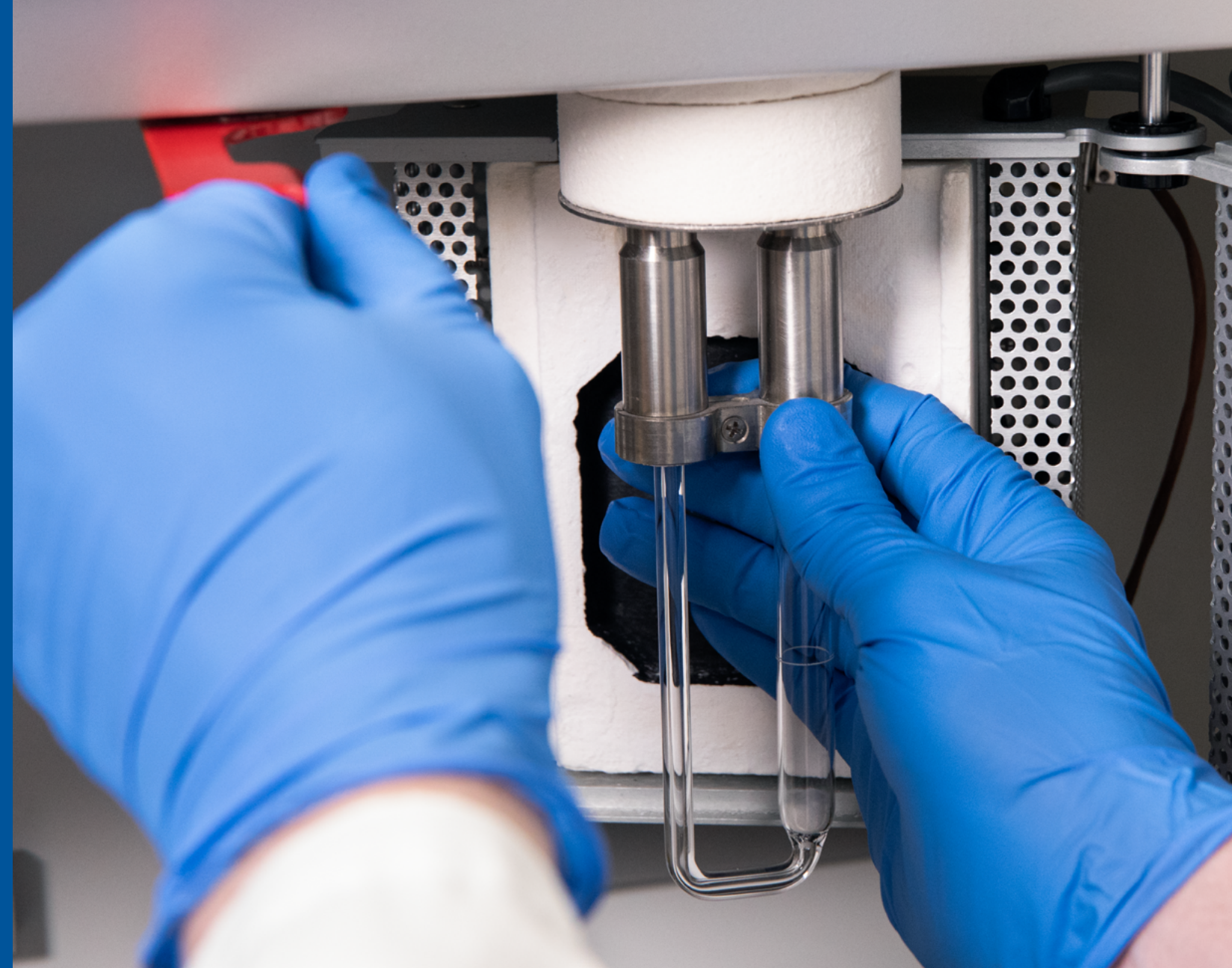
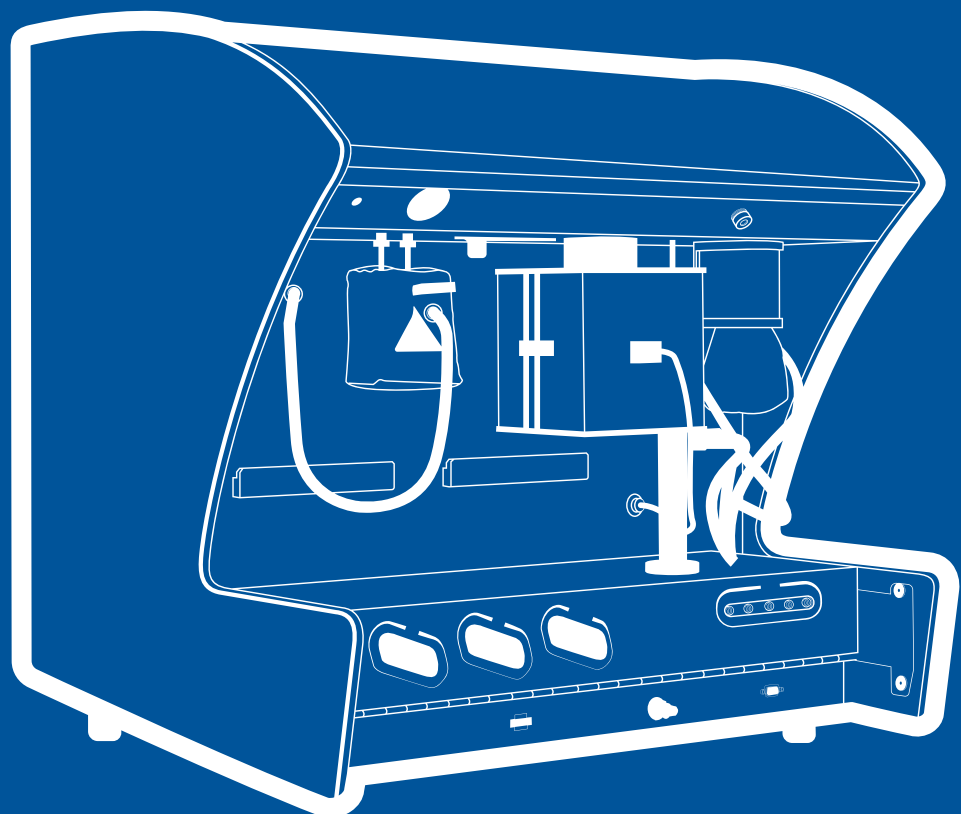


## MAKING ACCURACY EASY: EXCLUSIVE AUTOMATED DETECTOR CALIBRATION

The AutoChem III makes accuracy simple with automated detector calibration. Traditional systems require calibration by multiple runs of reference materials or single-point offsets that ignore changes in temperature or pressure.

The AutoChem III makes the most accurate gas volume measurement through an automated multi-point calibration using patented gas blending and a dedicated reference stream flow controller. Injection loop temperature control and pressure measurement reduce error by an additional 5% compared to inferior designs. The process is **fast, automated, requires no operator intervention, and produces more accurate results than alternative designs.**

US Pat #10,487,954 B2



## LOADING SAMPLES IS A SNAP

The patented new KwikConnect makes sample tube installation faster, easier, and more reliable than traditional designs with **half as many separate pieces and no threaded fittings**. Installation and removal is easier and quicker, reduces the risk of breaking sample tubes, and provides peace-of-mind that the snap lock closure has completely sealed the system.

US PAT #11,105,825 B2



# AUTOCHEM III FEATURES

[LEARN MORE →](#)

**Internal gas temperature control**  
in four separate zones prevents condensation during studies with vapor and improves overall signal stability

**Lowest internal gas volume**  
provides the highest peak resolution and minimizes tailing when changing gas stream composition.

**High sensitivity thermal conductivity detector (TCD)**  
is 2x as sensitive as alternatives so you can measure smaller samples, detect secondary reactions, and have greater confidence in your results

**KwikConnect**  
retention system makes sample tube mounting quick, easy, and safe with no threaded connections and half as many separate pieces as traditional designs

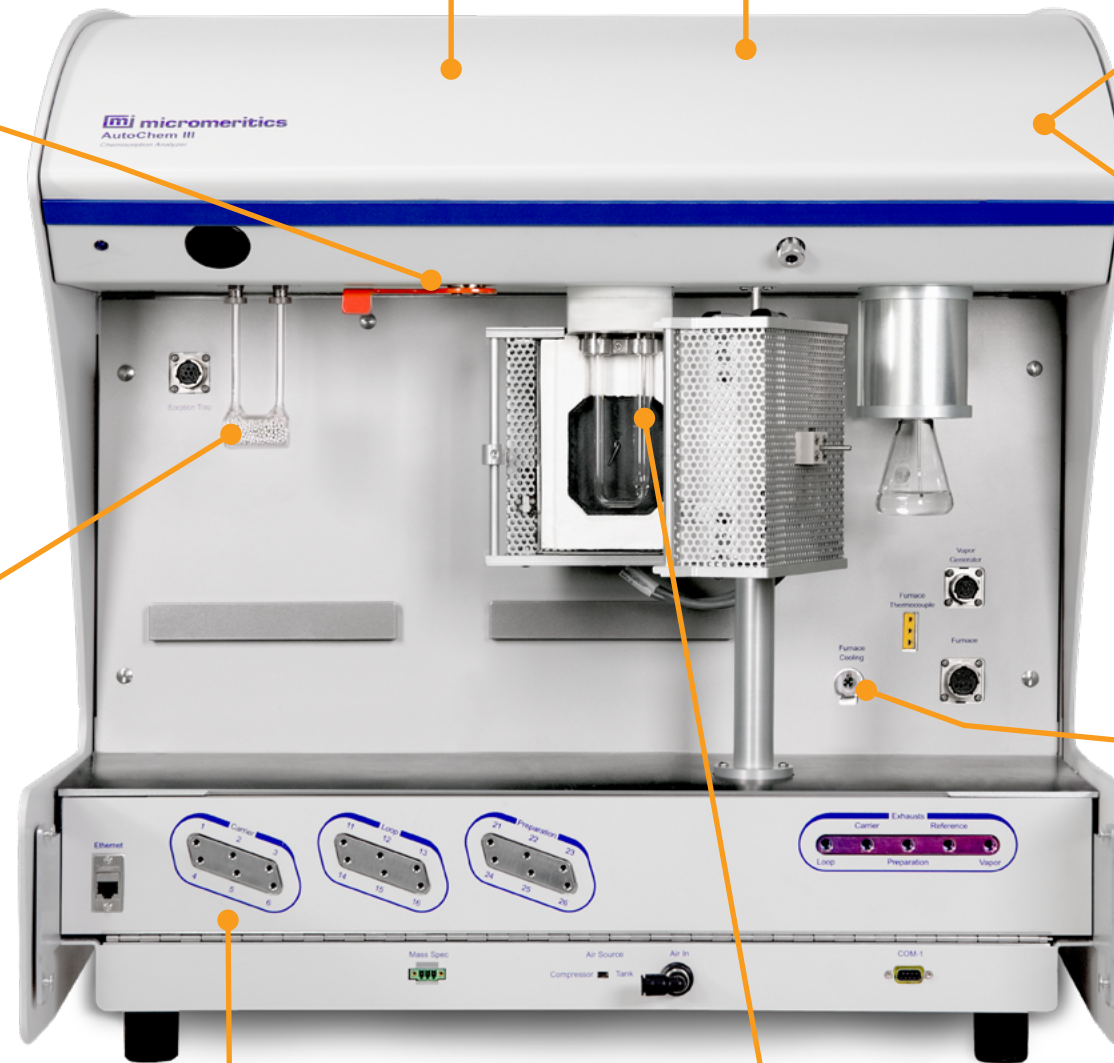
**Temperature-controlled corrosion-resistant detector**  
is compatible with corrosive gases and inherently protected from gas leaks that can destroy alternative designs, providing high reliability and long operational life

**Exclusive AutoTrap**  
provides superior moisture removal for TPR experiments with a system that is effortless to use and saves hours per day

**Integrated AutoCool**  
cools the furnace and sample quicker than forced air alone without the use of cryogenic liquids, saving an average of 30 minutes per experiment

**18 total gas inlets**  
six each for preparation, carrier, and loop gases permit sequential experiments of different types and saves time between experiments

**Dynamic clamshell furnace**  
provides temperature control up to 1200°C and controlled heating rates from 0.1°C/min to 100°C/min with the lowest available temperature overshoot



# BETTER MEASUREMENTS FOR MORE CONFIDENT DECISIONS

The AutoChem III provides results that drive confident decisions. The highest available measurement accuracy and repeatability - made under conditions that match your reaction environment - give you certainty to act with confidence.

## PRECISION TEMPERATURE CONTROL

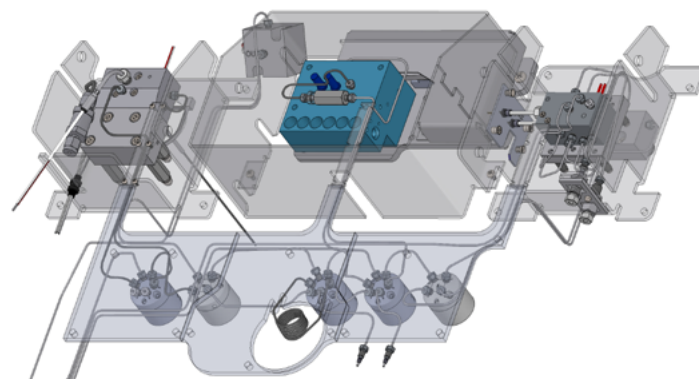
Exact thermal accuracy is essential to simulate reaction conditions while preventing deactivation of precious catalyst materials. The AutoChem III exceeds all available systems in every key performance characteristic

- Widest range of temperatures: -100°C to 1200°C
- Widest range of heating rates: 0.1 °C/min to 100 °C/min  
Repeatable temperature profiles  
Accurate determination of activation energy,  $E_a$
- Local sample temperature measurement  
Superior control accuracy without overshoots
- Four independently controlled gas stream temperature zones eliminate vapor condensation and enhance measurement stability

## MOST ACCURATE GAS STREAM COMPOSITION

The AutoChem III features the lowest gas flow path volume to **eliminate carryover and signal tailing when changing gas flow conditions**. This guarantees precise gas stream composition, even when switching configurations from one experiment to another.

And with 18 available gas inlets, you will have the gas composition that you need ready, without introducing errors associated with blending gases *in situ*.



## BETTER TEMPERATURE CONTROL AT EVERY STEP

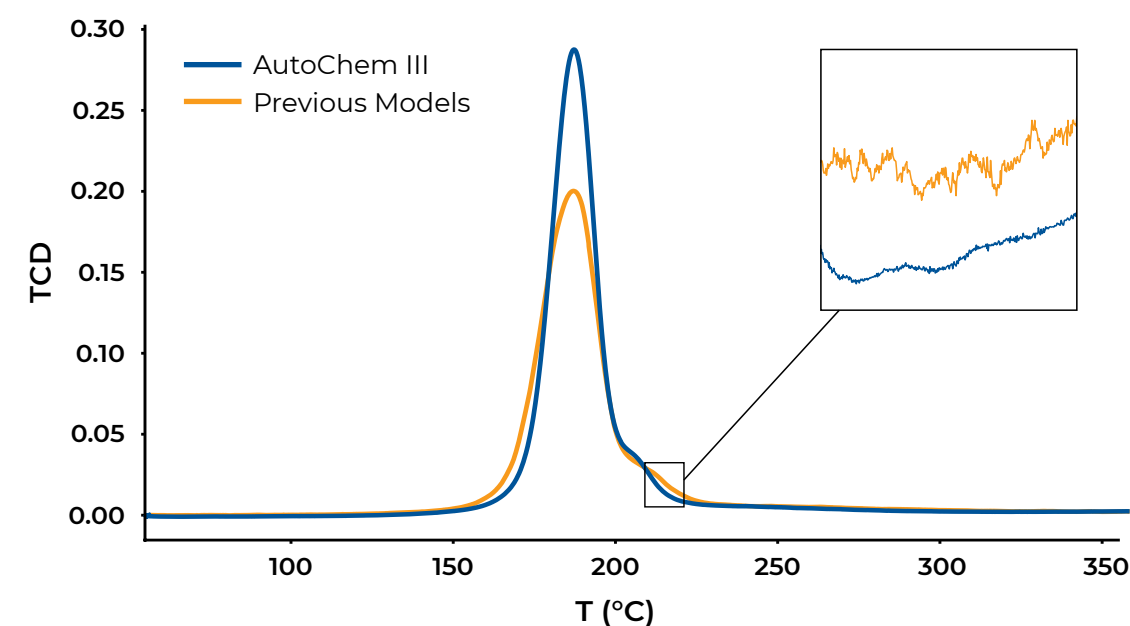
<b>FURNACE</b> to simulate reaction conditions	<b>VAPOR</b> to control vapor composition	<b>GAS STREAM</b> to maximize detection sensitivity	<b>DETECTOR</b> to ensure robustness
--	---	---	--

## SEE MORE OF YOUR REACTION WITH THE MOST SENSITIVE CHEMISORPTION SYSTEM IN THE WORLD

The new AutoChem III features a new thermal conductivity detector (TCD) that is **110% more sensitive** than previous designs. This enables you to use **lower sample masses**, accurately detect **secondary reactions**, and achieve **greater accuracy** of catalyst traits like site coverage.

Detector sensitivity is enhanced by a reference stream with a dedicated mass flow controller (MFC) that provides a stable reference to the sample stream. Alternative designs use a common carrier stream for both the reference and signal paths, resulting in interference between the measurement and reference stream leading to signal instability.

The temperature-controlled TCD is a robust sensor with a long operating life and intrinsic protection from operational errors like gas flow leaks that cause premature failure of 4-element detectors used in inferior designs.







## CONTINUOUS CONTROLLED VAPOR DOSING

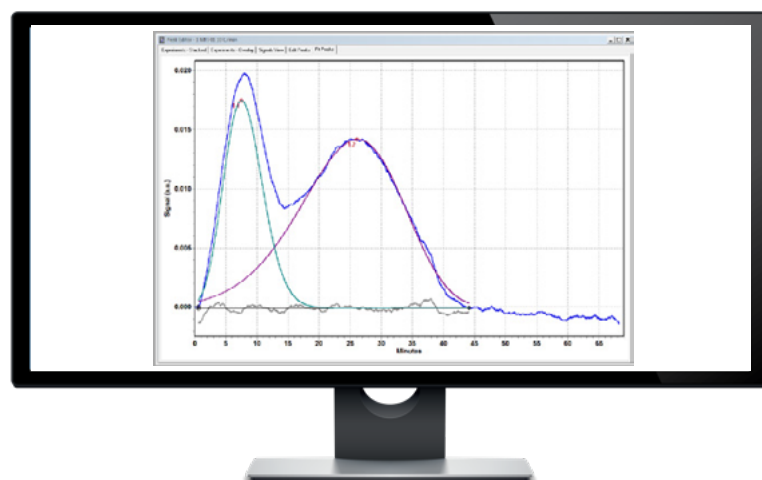
Achieve faster analysis and more complete characterization of surface selectivity and functionality with the available vapor generator featuring automated vapor calibration, injection repeatability better than 1%, and **all new continuous dosing** capabilities. This system creates uniform streams of saturated vapors such as water, alcohols, amines, or organics that are used to prepare samples for TPD or as the reaction gas stream.

The new continuous dosing capability enables **faster and more uniform vapor dosing** than legacy systems that are limited to discrete vapor stream pulses.

## MOVE QUICKLY FROM DATA TO DECISION

Rapidly transition from experimental data to material characteristics with Micromeritics' own AutoChem data analysis software. Get all the answers you need with:

- Interactive peak analysis including: limit selection, baseline definition, integration, and deconvolution
- Built-in analysis models for: pulse chemisorption, % dispersion, metal surface area, crystallite size, First-order kinetics, heat of desorption, activation energy, BET, Langmuir, total pore volume, and more.
- Seamless integration of mass spec data
- Detailed, configurable graphical reports



## IMPROVING OPERATOR SAFETY

**The AutoChem III enhances operator safety at every stage of the measurement, reducing opportunities for exposure and potential for hazardous conditions.**

### NO CRYOGENIC LIQUIDS

The new AutoTrap removes moisture **without cryogenic liquids** such as liquid nitrogen. The AutoTrap also eliminates the need for slush bath preparation, which requires vigorous mixing of alcohols and other solvents in glass vacuum flasks.

### COOL TO THE TOUCH

The new AutoCool brings sample tubes to room temperature so fast after every experiment that you can change samples and **start the next experiment quickly, without handling hot glass sample tubes**. And the KwikConnect sample tube retention system allows you to release the tube in one motion without fumbling with threaded connections and separate adapter pieces.

### THIRD-PARTY TESTED AND VERIFIED

Micromeritics products are third-party-tested to conform to the highest level of regulatory compliance and operational safety. Install and run with the confidence that the system will meet or exceed requirements for electrical safety and compatibility with the need for separate qualifications or assessments.



# INSTRUMENT CAPABILITIES

## AutoChem III

Temperature	Ambient to 1200°C
Temperature Ramp Rates	-100°C to 800°C: up to 100°C/min 800°C to 1000°C: up to 50°C/min 1000°C to 1200°C: up to 25°C/min
Preparation gases	6 inlets: H <sub>2</sub> , O <sub>2</sub> , He, Ar, H <sub>2</sub> /Ar, and more
Carrier gases	6 inlets: He, Ar, H <sub>2</sub> /Ar, and more
Analysis (loop) gases	He, H <sub>2</sub> , CO, O <sub>2</sub> , N <sub>2</sub> O, NH <sub>3</sub> /He, and more
Pulse Chemisorption	
Temperature-programmed reactions: TPR, TPO, TPD, TPSR	
Strong Chemisorption: Reactive metal area, dispersion, crystallite size	
Active site surface concentration	
Reduction, Oxidation Temperatures	
Acid site strength distribution: Lewis/Brønsted acid site distribution	
Breakthrough Curve Measurement	
Activation Energy	
<b>Optional Capabilities</b>	
CryoCooler	-100°C to 1200°C
Detection by Mass Spec	
Continuous or Pulsed Vapor Dosing: water, alcohol, amines, aromatic organics, and more	
Enhanced Chemical Resistance	
B.E.T. Surface Area	

# OPTIONS AND ACCESSORIES

## MASS SPECTROMETER

Mass spec provides a direct probe for the identity and quantity of specific reaction products. This is especially valuable when investigating an unknown reaction, or a reaction that creates multiple products. The single quadrupole mass spec with heated transfer line provides detection of mass fragments up to 200 amu and data collection that is integrated with the operation of the AutoChem III.

The AutoChem III also includes a general mass spec communication port for coordination with a lab's existing mass spec.

## CRYOCOOLER

Begin experiments at temperatures as low as -100 °C with controlled liquid-nitrogen-based cooling.

## VAPOR GENERATOR

Prepare samples for analysis or perform measurements in the presence of pulsed or continuous vapor streams such as water, alcohol, pyridine, aromatic organics, and more.

## ENHANCED CORROSION RESISTANCE (ECR)

For reaction chemistries that requires particularly aggressive gas compositions, a special version of the AutoChem III is available with enhanced corrosion resistance. Wetted materials are constructed from highly resistant Hastelloy, highly stable perfluoroelastomers, and inert-coated stainless steel to provide the greatest stability under the harshest working conditions.



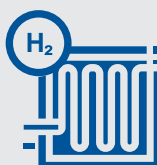


# RELEVANT APPLICATIONS



## NET ZERO TECHNOLOGIES

The development of efficient and effective catalysts is necessary to the continued development of CO<sub>2</sub> mitigation and the hydrogen economy that will enable a sustainable energy future. The AutoChem III is a useful tool to optimize adsorption and dissociation of H<sub>2</sub>/O<sub>2</sub> on electrolysis electrodes, show whether desorption occurs near reaction conditions, quantify acid or base sites to optimize reactivity and selectivity, and more.



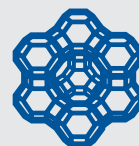
## FUEL CELLS

Platinum-based catalysts including Pt/C, PtRu/C, and PtRuIr/C are often characterized by temperature-programmed reduction to determine the number of oxide phases and pulse chemisorption to calculate: metal surface area, metal dispersion, and average crystallite size.



## PARTIAL OXIDATION

Manganese, cobalt, bismuth, iron, copper, and silver catalysts used for the gas-phase oxidation of ammonia, methane, ethylene, and propylene are characterized using: Temperature-programmed oxidation and desorption, heat of desorption & dissociation of oxygen.



## CATALYTIC CRACKING

Acid catalysts such as zeolites are used to convert large hydrocarbons to gasoline and diesel fuel. The characterization of these materials includes: Ammonia chemisorption and temperature-programmed desorption.



## CATALYTIC REFORMING

Catalysts containing platinum, rhenium, tin, etc. on silica, alumina, or silica alumina are used for the production of hydrogen, aromatics, and olefins.



## ISOMERIZATION

Catalysts such as small-pore zeolites (mordenite and ZSM-5) containing noble metals (typically platinum) are used to convert linear paraffins to branched paraffins.



## HYDROCRACKING: HYDRODESULFURIZATION, AND HYDRODENITROGENATION

Hydrocracking catalysts typically composed of metal sulfides (nickel, tungsten, cobalt, and molybdenum) are used for processing feeds containing polycyclic aromatics that are not suitable for typical catalytic cracking processes.



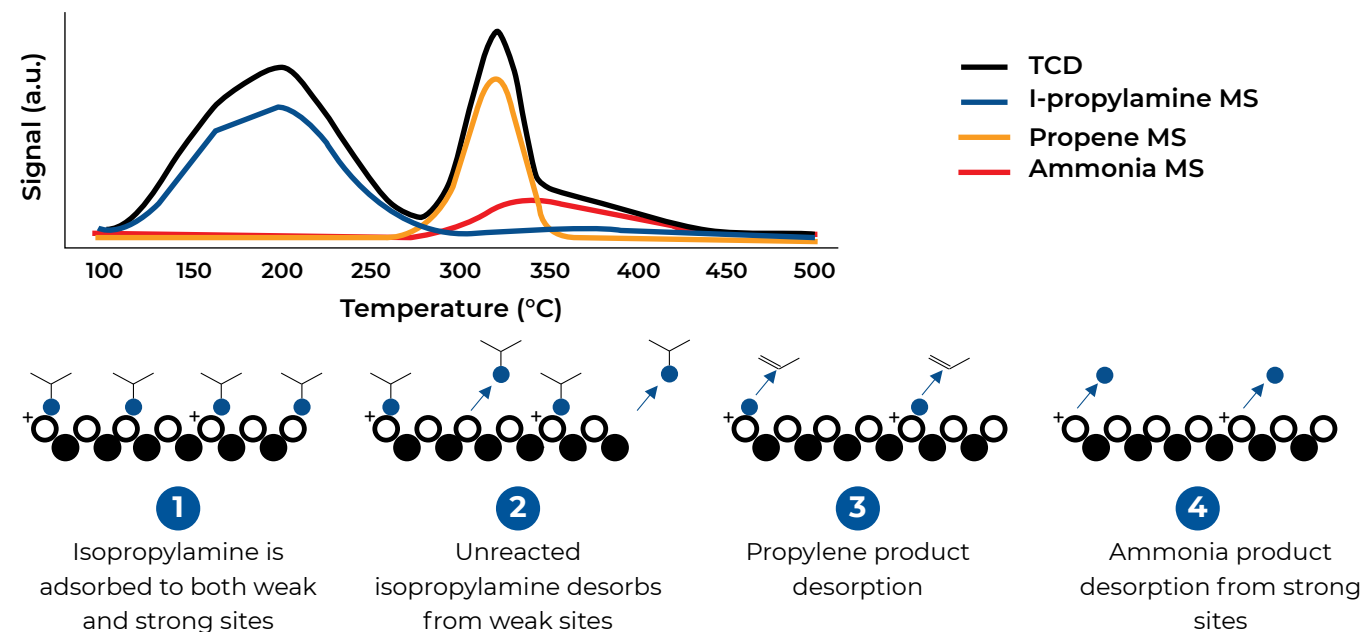
## WATER GAS SHIFT REACTION

The water gas shift reaction is an important element in the hydrogen lifecycle and the push toward net zero technologies. The combination of catalysts, often copper-zinc-alumina and iron-chromia, are characterized by TPR and pulse chemisorption maximize activity.

# AUTOCHEM METHODS

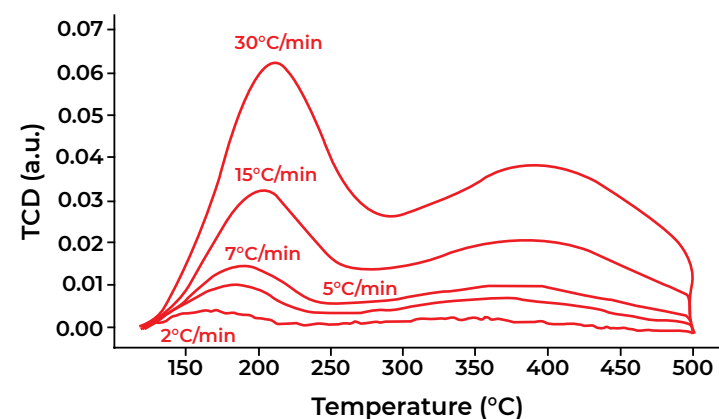
## TEMPERATURE PROGRAMMED REACTIONS

The suite of temperature programmed reactions are collectively used to measure reactivity as a function of temperature through change in gas stream composition. As temperature is increased, gas stream composition is changed through: consumption of reactive gases, creation of reaction products, and desorption of bound species.



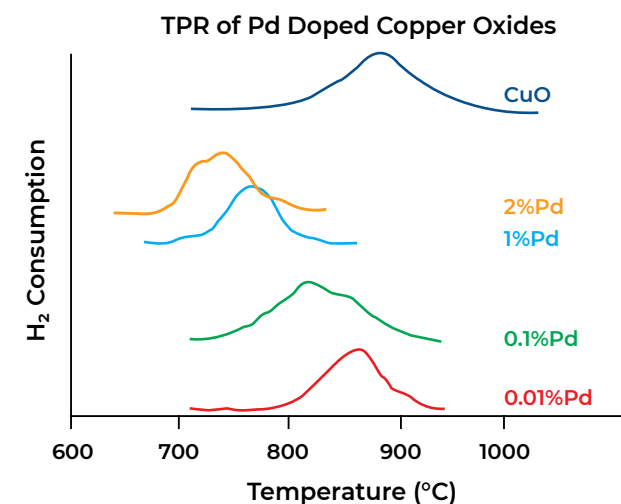
## TEMPERATURE-PROGRAMMED DESORPTION (TPD)

Species previously adsorbed can be desorbed by increasing sample temperature under flowing inert gas. Ammonia TPD is one of the most common applications. A sample is first saturated with ammonia during the preparation step, then heated to desorb bound ammonia, revealing **relative acid site strength for materials such as zeolites**. Similarly, desorption of carbon dioxide indicates the **strength of basic sites**.



Bulk decomposition into the gas phase can also be used to characterize carbonates for CO<sub>2</sub> removal or hydrides for hydrogen storage.

## TEMPERATURE PROGRAMMED REDUCTION (TPR)



The TPR measurement is a specific case of temperature programmed reaction in which a gas blend of hydrogen and an inert carrier – usually Argon – are passed over an oxide sample. Hydrogen is removed from the gas stream and water vapor is generated. The water vapor is trapped by the AutoTrap and the depletion of hydrogen from the carrier stream is measured.

In particular, this measurement **provides the required conditions (temperature, time, and activation energy) required to prepare a heterogeneous catalyst** from its native oxide state into the active zero-valence metal.

## TEMPERATURE PROGRAMMED OXIDATION (TPO)

In a TPO experiment, oxygen in the sample gas stream is consumed, generally reacting with different forms of carbon to produce CO or CO<sub>2</sub>. TPO experiments are important to characterize the **reactivity of metal oxide catalysts** and optimize process conditions.

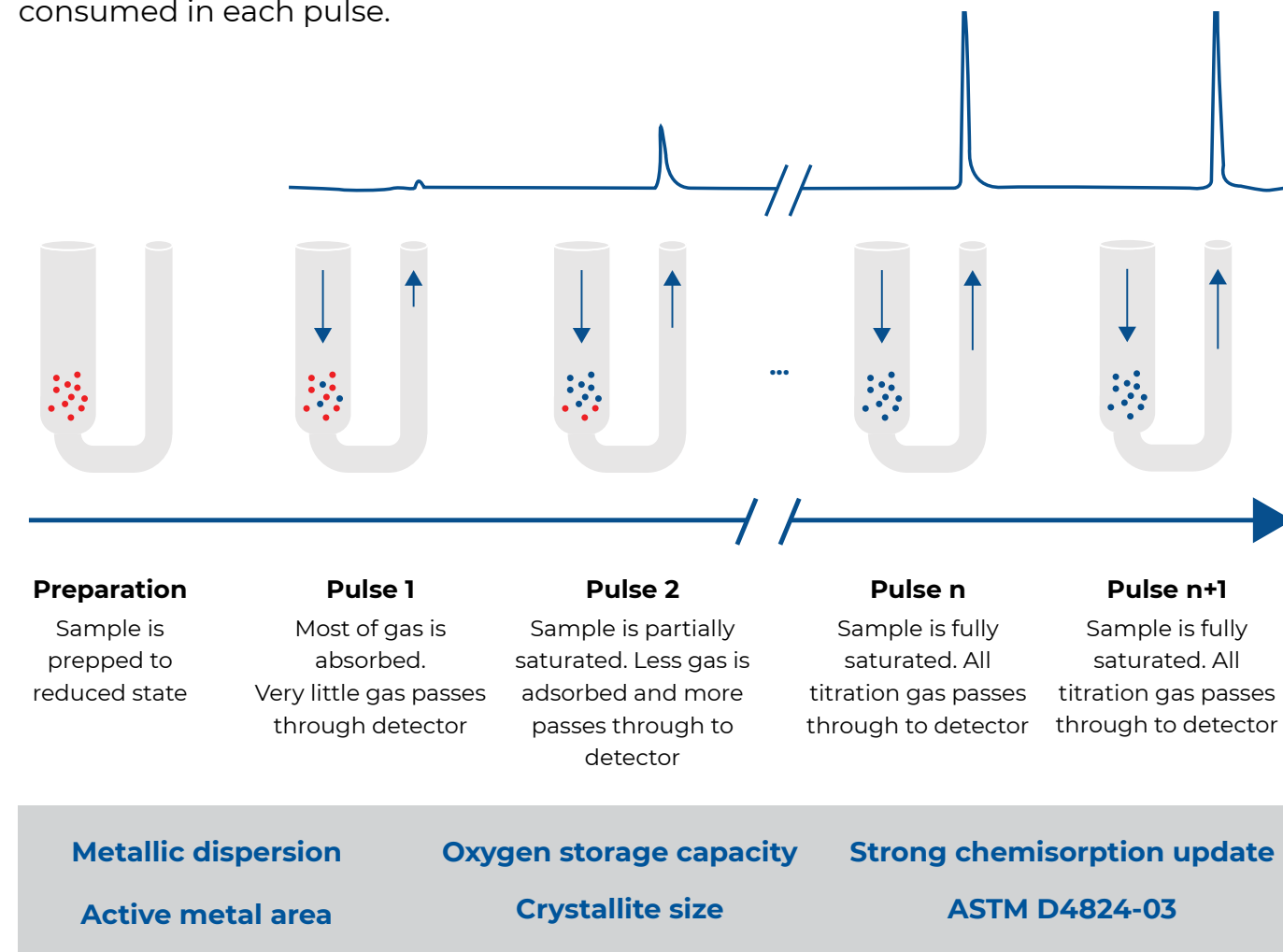
The temperature of oxidation is also associated with the reactivity of carbon-carbon bonds, and is therefore an effective means for **differentiating forms of carbon**. Using TPO, amorphous, nanotube, filament and graphitic carbon can be distinguished, especially those that form on catalysts.



# AUTOCHEM METHODS

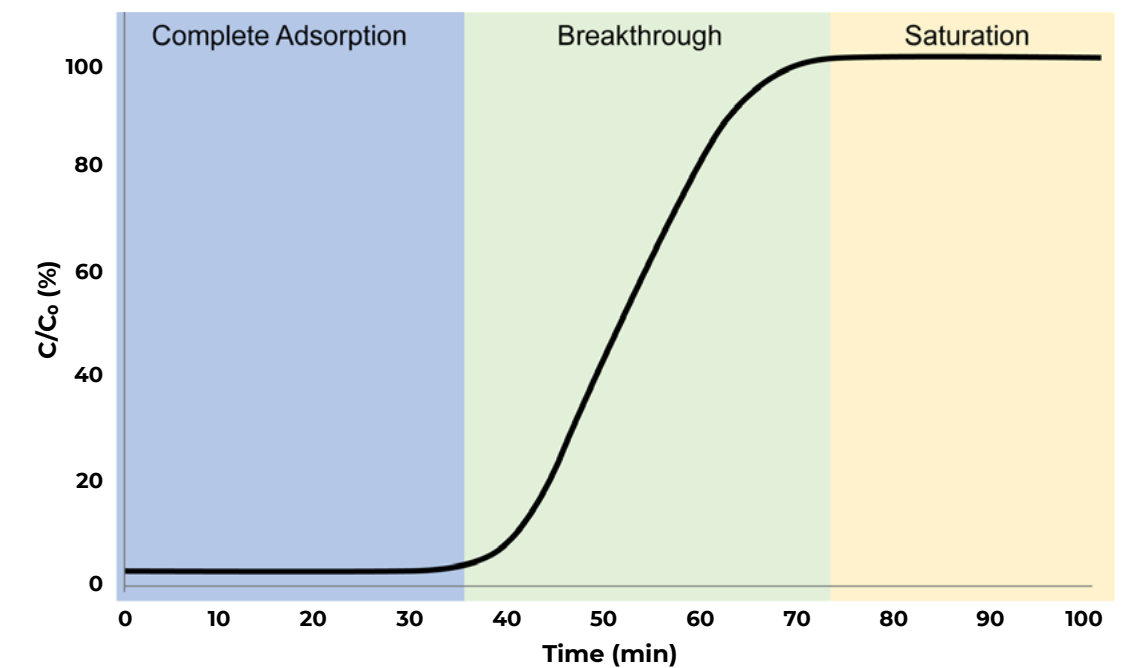
## PULSE CHEMISORPTION

A sample is prepared in situ to a known initial state (e.g. pure oxide or valence metal) through temperature and gas exposure. Reactive gas pulses of known volume are delivered through to the sample and the system measures the volume of gas consumed in each pulse.



## BREAKTHROUGH CURVE ANALYSIS

Breakthrough analysis is a powerful technique for determining the adsorption capacity of a material under dynamic flow conditions. Breakthrough analysis allows users to precisely control temperature, pressure, and gas flowrates during an experiment. This allows users to analyze adsorbates under process relevant conditions, giving them the tools necessary to optimize their systems and adsorbent materials for their application. Additionally, breakthrough allows users to easily collect multicomponent equilibrium adsorption data allowing them to determine the selectivity and adsorption kinetics of their materials.



## B.E.T. SURFACE AREA: PHYSISORPTION

The AutoChem III can measure B.E.T surface area by the flowing or dynamic method in which  $N_2$  depleted from, or added to, a flowing gas stream is measured at liquid nitrogen, or ambient, temperatures, respectively. Basic physical surface area measurements are important for catalyst development as they reflect the basic physical form of the catalyst and/or support and the available contact area for reactivity. This is also an important basic measurement for porous and granular materials of all types.



## SERVICE SUPPORT

[micromeritics.com/service](http://micromeritics.com/service)

Customer service is at the heart of what we do with over 10,000 installations during the past 60 years.

### ISO-9001 CERTIFIED SERVICES



1 Year Parts and Labor Warranty



Preventative Maintenance



Maximized uptime



Reduced cost of ownership



Well-trained users



Predictable, easy-to-budget-for expenses with protection from unexpected operating costs

Micromeritics offers a full range of instrument installation, preventive maintenance and repair services to support instruments through their full life cycle. On-site and factory services are provided through our global network of factory trained and certified service engineers.

## APPLICATION SUPPORT

[micromeritics.com/apps](http://micromeritics.com/apps)

Micromeritics' team provides industry-leading, high-quality application support and training to assist scientists, engineers, and analysts in the field of material characterization. Our application support team is composed of scientists and engineers to help users obtain the highest quality data and information about their material from Micromeritics Instruments. The Micromeritics team is dedicated to helping users successfully use their Micromeritics Instruments for the life of their instrument.



Expert, lifetime, applications support for Micromeritics customers.



Free training courses, application notes and how-to videos available on [www.micromeritics.com](http://www.micromeritics.com)

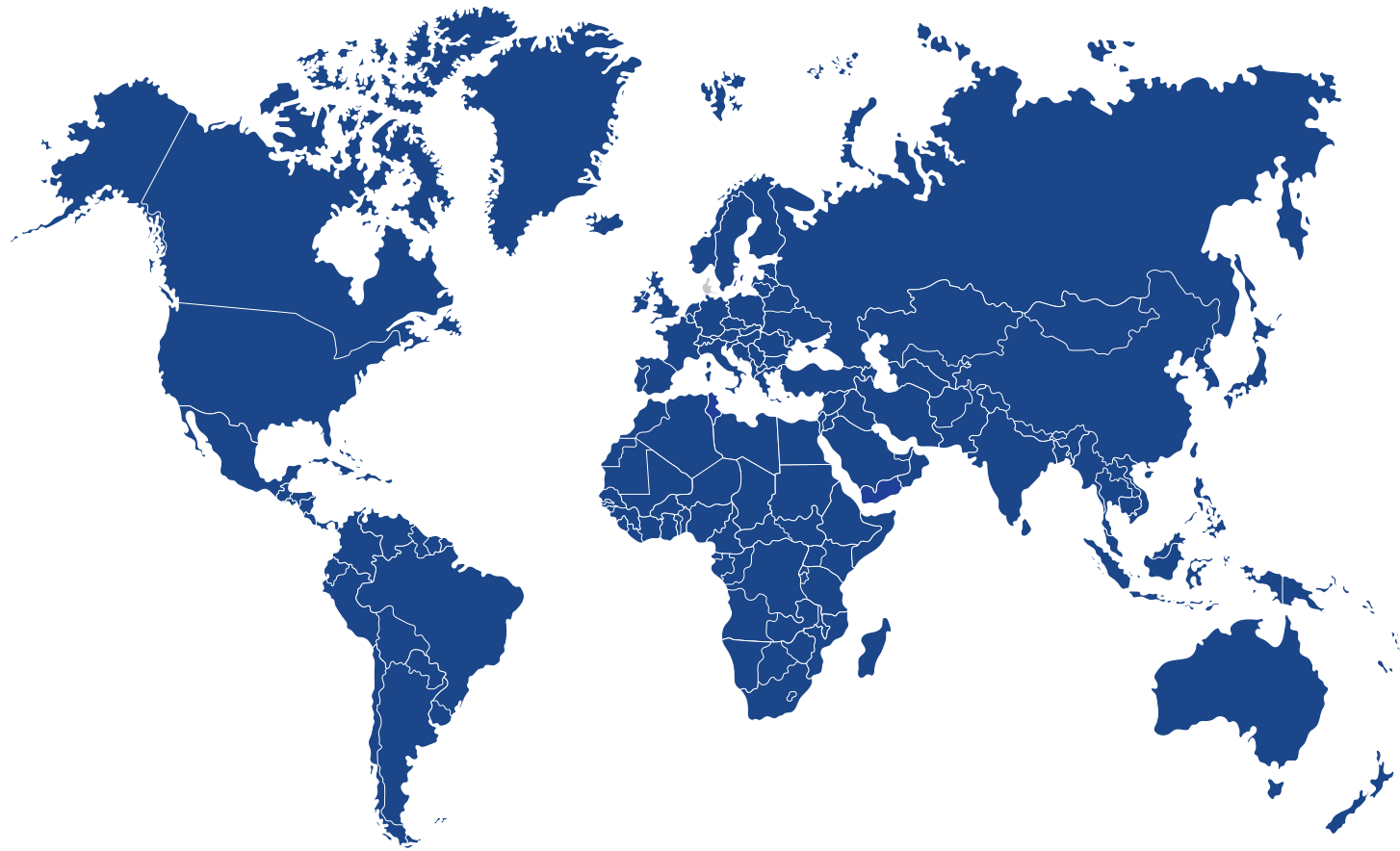


Applications specific, hands-on training available in Micromeritics USA, Germany, and China facilities



Collaborations with industrial and academic partners to continually improve the quality of measurements and interpretation of material characterization data





## WORLDWIDE PRESENCE

[micromeritics.com/worldwide](https://micromeritics.com/worldwide) →

**MORE** than **12,000** installations in **100** different countries are used every day in the labs of the most innovative companies and the most prestigious government and academic institutions.

Customers choose Micromeritics for their **world leading** high-performance systems, expert application staff, and factory-trained engineers around the globe.

Get **MORE**  
from Micromeritics



## MATERIALS CHARACTERIZATION SERVICES WORLD-CLASS ACCREDITED LABORATORY

**PARTICLE  
TESTING  
AUTHORITY**



Need to characterize your materials or supplement your current lab's capabilities? Want access to top-of-the-line instruments and expert scientists?

The Micromeritics PTA lab is the leading contract laboratory for the characterization of catalysts, supports, and adsorbents. The same engineers and scientists that develop and support our market-leading technologies are available to help you develop methods, test samples, and analyze the results.

- ISO 17025 accredited and FDA registered
- Globally recognized scientists
- Typical turnaround time: 7 business days
- Over 25 analytical techniques

Contact PTA today to learn how our world-class laboratory can advance the development of your next generation catalysts.



Micromeritics products are 3rd party tested to conform to the highest level of compliance and safety. Visit [micromeritics.com/compliance](https://micromeritics.com/compliance) for full details by product.



## Micromeritics Instrument Corporation

4356 Communications Drive, Norcross, GA 30093 USA

Tel.: +1 770-662-3636