INTRODUCING THE BREAKTHROUGH ANALYZER (BTA)

A Compact, Versatile, High-Performance Selective Adsorption System.

micromeritics.com/BTA





THE BREAKTHROUGH ANALYZER

When your research requires high-performance selective adsorption data, choose the new BreakThrough Analyzer from Micromeritics.



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The new BreakThrough Analyzer (BTA) is a flexible gas delivery and management system for the **precise characterization** of adsorbent performance under process relevant conditions. It delivers reliable adsorption data for gas/vapor mixtures using a **flow-through system**.

A safe and highly optimized device for collecting both transient and equilibrium adsorption data for multi component systems. The BTA can be configured with up to six precision mass flow controllers and patented high performance blending valves, delivering unparalleled flexibility in experimental design. The superior gas-delivery design ensures the precise control of both composition and flow rate, while **minimizing** dead volume.

The high-quality, stainless-steel column can hold 0.05 to 2.5 grams of adsorbent. Automated sample activation up to 1050°C is possible with the precise, rugged, and reliable resistance furnace.

Operating pressures are controlled from atmospheric to 30 bar via a servo positioned controlled valve. The thermostated environmental chamber delivers **uniform temperature** control of the entire system up to 200°C, eliminating cold spots. The BTA secure door lock system ensures operator safety throughout the analysis.

Vapor generators can be added to the BTA to enable the use of important probe molecules such as water for experimental studies. The BTA easily **connects** to commercially available Fourier Transform Infrared Analyzer (FTIR) and Mass Spectrometer (MS) systems for gas identification and quantification.



PID

GAS 5

GAS 6

user safety

Thermostated Environmental Chamber A prevents condensation of vapor streams



BTA

Electropolished 316 SS Sample Column

with a capacity of up to 2.5g and is suitable for use with powders, other diameters are available for pellets or extrudates



Column Furnace rugged, resistance furnace with high temperature capabilities up to 1050°C

Addition Of Detectors and G Other Optional Accessories

System scalability enables the expasion of capabilities over time through addition of detectors and other optional accessories (e.g. mass spectrometer, GC/MS, additional vapor sources, vacuum activation, others available upon request)

Fully Automated Experimental Design

allows for easy experimental setup



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Touch Screen allows for easy instrument operation and monitoring of experimental conditions

Proprietary Blending Valves

provide remarkable advantages for gas mixing and minimization of system dead volume

Up to 6 Gas Inlets and 2 Vapor Sources

to offer a wide range of analysis options with exceptional flow control and blending of multiple gases

Automatic Door Lock

ensures temperature stability during analysis and



WHY THE MICROMERITICS BTA?

The BreakThrough Analyzer allows for the widest range of experimental conditions with unmatched automation from sample activation to analysis. The BTA offers several advantages over any competitive adsorption measurement system including:

- Configurations with up to 2 vapor sources available
- Proprietary zero volume blending valves with unmatched minimization of dead times
- Unparalleled touch screen control
- Thermostated environmental chamber delivers uniform temperate control up to 200°C exclusive to the BTA
- MicroActive software, the most intuitive, flexible, and comprehensive analysis software for adsorption studies

INSTRUMENT SPECIFICATIONS

Furnace Temp Max Convection Oven Temp Max Sample Mass Sample Volume Features

Investigation of kinetic performance of adsorbents Investigation of co-adsorption and displacement Determination of sorption selectivity High resolution separations using small sample quantities Dynamic adsorption and desorption experiments Determination of single- and multi- component adsorption data quartz column

Fully automated control via PC

Up to 6 high precision mass flow controllers

Ultra-low dead volume for rapid signal response

Automated switching between purge and process gases

Configurations for gas-vapor and vapor-vapor separation

altered temperature conditions

Touch Screen

Patented "No Dead Volume" mixing valve with rapid switching

6 WHY BREAKTHROUGH

1050°C

200°C

- Up to 2.5 g
- Up to 2.5 mL
- CE

- Determination of breakthrough curves
- In-situ sample preparation up to 450°C with a SS column & 1050°C with a
- Programmable total pressure, flow rate, composition and temperature
- Optimised for research-scale sample sizes with interchangeable reactor beds
- Door remains locked during analysis to protect user and the analysis from

RELEVANT APPLICATIONS

DIRECT AIR CAPTURE

DAC is difficult due to low concentrations of carbon dioxide in air along with other impurities including moisture, and the captured CO_2 may be sequestered underground, sold, or converted into value added chemicals to offset carbon emissions.

OLEFIN/PARAFFIN SEPARATONS

Olefin/Paraffin Separations are a core part of the petrochemical industry and used to in the production of polymers such as polyethylene and polypropylene; these separations are energy intensive and increase CO₂ emissions.

CO₂ ADSORPTION

CO₂ Adsorption – power generation, chemical plants, and refineries are significant point sources for carbon dioxide emissions and the higher concentrations often require different operating conditions when compared to direct air capture.

NATURAL GAS SEPARATION

Natural gas is a mixture of hydrocarbons and other gases that must be purified prior to use in industrial applications and households for heating and food preparation.

WATER ADSORPTION

Harvesting water from the air may be a critical technology for many parts of the world where the fresh water supply is limited due to an arid climate or the increasing usage of water for agriculture.

TOXIC GAS ADSORPTION

Toxic Gas Adsorption porous solids are used for personal protection and also under development for the capture of toxic gases including sulfur dioxide, hydrogen sulfide, and nitrogen dioxide from natural gas or other process feeds.

APPLICATION MATERIALS



ZEOLITES

Pressure swing adsorption using Zeolite 5A, 13X, or LiX, which have high selectivity for adsorbing nitrogen are used commercially for air separation and producing oxygen.

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#### POROUS MEMBRANES/ MONOLITHS

Porous membranes and monoliths coated zeolites or MOFs are commonly used to improve the operational efficiency of separation processes.



### **POROUS ALUMINAS**

Alumina – Supported Ionic Liquids are effective adsorbents with potential applications for the separation of CO₂ from natural gas.



#### SILICAS

Amine functionalized silicas are effective and highly selective adsorbents and used for the direct air capture (DAC) of CO₂.



#### **ACTIVATED CARBON**

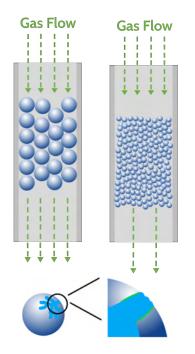
Volatile organic component (VOC) from automobile fuel systems are captured by canisters filled activated carbon and these VOC emissions are minimized.



#### METAL-ORGANIC FRAMEWORKS

MOFs are highly selective adsorbents which are effective for demanding commercial applications including alkanes & olefins, olefins & alkynes, DAC, CO₂ & CH₄.

## **BREAKTHROUGH ADSORPTION** DYNAMIC ANALYSIS



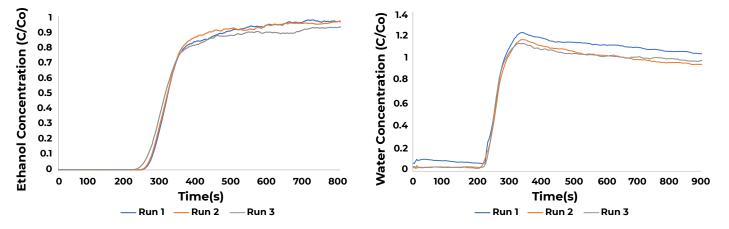
Breakthrough analysis is a powerful technique for determining the adsorption capacity of an adsorbent under flow conditions. Dynamic breakthrough adsorption provides many advantages over static adsorption measurements.

- Easily collect multicomponent adsorption data
- Determine adsorbate selectivity
- **Replicate process conditions**

When conducting breakthrough analysis, sample preparation is a critical step in the analysis process to prevent pressure drop and mass transfer limitations. Pressure drop occurs when the interstitial space between particles is too small to accommodate the flowrate of gas. Mass transfer limitations occur when the pore size of the material is similar to the kinetic diameter of the adsorbate. Appropriately sizing particles is therefore critical to obtain the best results.

## **MULTICOMPONENT VAPOR ANALYSIS**

The Micromeritics BTA is capable of flowing up to two vapor streams simultaneously through its packed column. The thermostated environmental chamber prevents condensation of these vapor streams during analysis and ensures that all gases and vapors maintain a constant temperature within the instrument. Vapor streams are generated using a bubbler which allows for a carrier gas to reach saturation with the vapor of choice. The figure below displays multicomponent ethanol/water breakthrough measurements conducted on zeolite 13X.



Figures: Ethanol (left) and water (right) multicomponent breakthrough curves collected at 50°C on zeolite 13X.

## **EXAMINING** A BREAKTHROUGH CURVE

## **COMPLETE ADSORPTION**

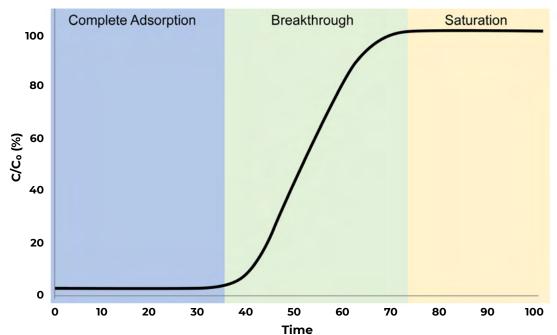
The adsorbent completely adsorbs the adsorbate gas such that none can be detected at the outlet of the breakthrough column

## **BREAKTHROUGH**

The adsorbate gas is first detected at the outlet of the breakthrough column. Gas continues to adsorb; however, the adsorbent is no longer able to adsorb the entirety of the gas that is entering the breakthrough column

SATURATION

The adsorbent has reached saturation and can no longer adsorb the adsorbate gas, allowing it to pass through the column freely



APPLICATIONS 1

## **CARBON** DIOXIDE ADSORPTION

Single component carbon dioxide breakthrough adsorption experiments were conducted on zeolites 13X and 5A, and metal-organic frameworks MIL-53(Al) and Fe-BTC. All materials were analyzed at 30 °C while flowing an equimolar gas stream consisting of 10 sccm nitrogen and 10 sccm carbon dioxide. A 1 sccm stream of helium was also blended into the feed gas stream as a tracer gas to aid in identifying the start of the breakthrough experiment. The breakthrough curves for the four materials are plotted below on a mass normalized axis. The total quantity of CO₂ adsorbed follows the trend: molecular sieve 5A > zeolite 13X > Fe-BTC > MIL-53(Al). The table below shows the total quantity adsorbed in mmol/g.

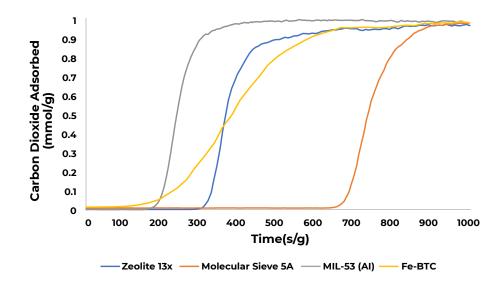
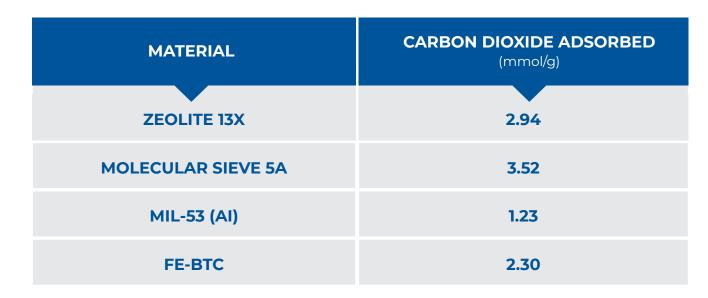
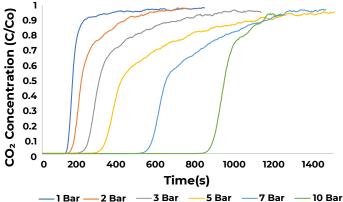


Figure: Breakthrough curves collected on two zeolites and two MOFs.



## **HIGH PRESSURE ADSORPTION**

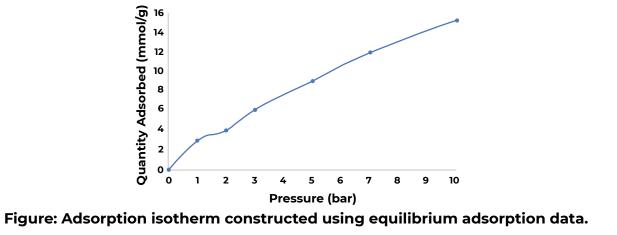
Zeolite 13X has been extensively studied for applications in catalysis and adsorption. In this study, zeolite 13X was used as an adsorbent for carbon dioxide adsorption to collect breakthrough curves from 1–10 bar pressure. These measurements were collected using equimolar flowrates of 10 sccm nitrogen and 10 sccm carbon dioxide. A 1 sccm stream of helium was used as a tracer gas to determine the start of the breakthrough experiment. All measurements were collected at an analysis temperature of 30°C. Between each measurement, the zeolite 13X sample was reactivated overnight to ensure complete desorption of carbon dioxide. The figure shows a consistent increase in breakthorugh time across successive experiments as the pressure is increased.





#### Figure: Breakthrough curves collected using an equimolar mixture of N₂ and CO₂ at 30 °C from 1 – 10 bar.

Following carbon dioxide breakthrough measurements an equilibrium adsorption guantity was calculated for each curve by solving the breakthrough equation. Next, an isotherm was constructed displaying the quantity of carbon dioxide adsorbed at 1, 2, 3, 5, 7, and 10 bar total pressure. At 10 bar, zeolite 13X adsorbed roughly 15 mmol/g carbon dioxide. While isothermal data collected via breakthrough cannot be directly correlated with static adsorption measurements, it can provide a assessment of an adsorbent in process relevant conditions.



# **CONFIGURATIONS**

Multicomponent adsorption studies often require a mass spectrometer (MS) to monitor the residual gas composition. The MS is the most common detector system used for breakthrough analysis.	<section-header>FOURIER TRANSFORM INFRARED (FTIR ANALYZERFTIR spectrometers are often selected for experimental breakthrough studies such as the separation of xylenes or other aromatic hydrocarbons.</section-header>	HUMIDITY SENSOR Allows direct tracking of H ₂ O content for low cost. Can be useful especially in production control applications.
Small quantities of active material can be mixed with an inert carrier to produce a homogeneous sample and improve analysis reproducibility.	CO₂ SENSOR Allows direct tracking of CO₂ content for low cost. Can be useful especially in production control applications.	MFC AND MIXING VALVES MAXIMUM 6 GAS INLETS Additional mass flow controllers and blending valves may be added to the BTA to increase the analytical capabilities and expand the range of experiments that may be conducted.
SAMPLE COLUMN (DIFFERENT VOLUME) The BTA may be used with a variety of column diameters to accommodate different sample morphologies included powders, pellets, and extrudates.	VAPOR SOURCE (MAXIMUM 2) Moisture or other vapors such as xylenes or other aromatics are compatible with the optional vapor sources available for the BTA.	<b>ENHANCED CHEMICAL</b> Special inert materials of construction enable simulation of process conditions – such as post-combustion CO ₂ capture – that include highly reactive gases like NOx, H ₂ S, or SO ₂ .



## **BTA** SAFETY

A safe and highly optimized device for collecting both transient and equilibrium adsorption data for multi component systems.

- Automatic shut off from Software
- Alarm in/out communication with central alarm system
- Safety system separated from PC
- Furnace temperature control alarm
- Thermostated environmental chamber temperature control alarm
- Full range of optional safety features available including automatic shut-off valves & gas detector

## **BTA** Software





The flexible, intuitive, easy-to-use software allows for the widest range of experimental conditions and automates the breakthrough from sample activation to sample analysis, including the ability to perform cyclic experiments.

Paired with industry leading MicroActive analysis software, the BTA system accurately and reproducibly characterizes adsorbents, analyzes data with comprehensive analysis methods, and solves the breakthrough equation for the most demanding samples.

## MicroActive is the most intuitive, flexible, and comprehensive analysis software for adsorption studies

MicroActive Software allows for:

- Data reduction from Mass Spectrometer
- Quantity adsorbed & selectivity





## **SERVICE** SUPPORT

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

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## **APPLICATION** SUPPORT

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.

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**APPLICATION SUPPORT 19** 

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#### **About Micromeritics Instrument Corporation**

Micromeritics is the world's leading supplier of high-performance systems to characterize particles, powders and porous materials with a focus on physical properties, chemical activity, and flow properties. Our industry-leading technology portfolio includes: pyc-nometry, adsorption, dynamic chemisorption, intrusion porosimetry, powder rheology, activity testing of catalysts, and particle size.

The company has R&D and manufacturing sites in the USA, UK, and Spain, and direct sales and service operations throughout the Americas, Europe, and Asia. Micromeritics systems are the instruments-of-choice in more than 10,000 laboratories of the world's most innovative companies, prestigious government, and academic institutions.

Our world-class scientists and responsive support teams enable customer success by applying Micromeritics technology to the most demanding applications.

#### For more information, please visit micromeritics.com

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