mixSorb Series

mixSorb

Dynamic Sorption Analyzer

- Breakthrough Curves
- Multi Component Adsorption
- Sorption Kinetics

- Material Research
- Chemical Engineering
- Energy Storage
- Industrial Adsorbents

- Gas Storage
- Gas Purification
- Separation Technologies
- Environmental

DYNAMIC SORPTION ANALYZER

3P INSTRUMENTS

Characterization of
particles · powders · pores

mixSorb
Dynamic Sorption Analyzer

Introduction

Industrial adsorbents such as active carbons, zeolites and silica gels are widely used in adsorptive separation processes on a multi-ton scale. The after-treatment of exhaust gasses, the removal of carbon dioxide in biogas plants, purification and fractionation of natural gas, air separation, respiratory protection and separation of isomers are just few examples where adsorptive separation is employed as the most efficient and economic separation technique. A complete understanding of the complex processes taking place in a fixed bed adsorber is the key to achieving the best separation performance.

Additionally to that mixed gas adsorption on novel materials, like MOF´s COF´s etc., are in focus of research in recent last years. These materials can have very high selectivities due to their extraordinary surface chemistry and have to be investigated by mixed gas sorption for a deeper understanding of their sorption behavior.

The mixSorb series provides unique capabilities to investigate complex dynamic sorption processes in a uniquely safe and easy-to-use bench-top instrument. Industrial adsorbents and small sample amounts of R&D groups can be investigated under authentic process conditions in a broad temperature and pressure range with adjustable gas flow rates and well-defined gas compositions.
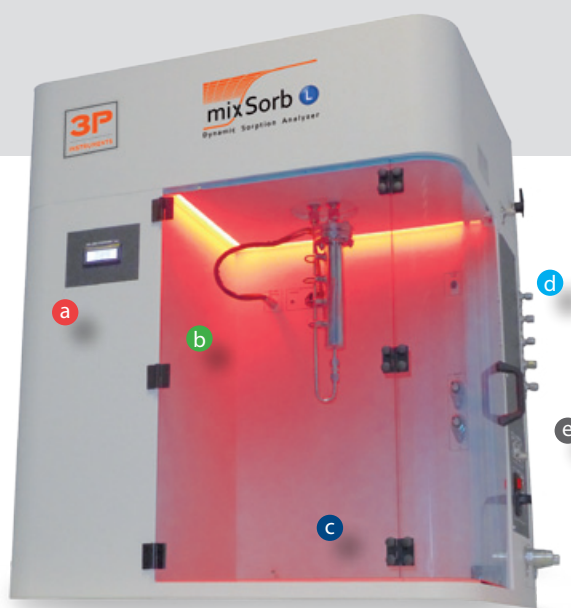
Adsorber

The different stainless-steel adsorbers can accommodate either representative sample amounts of industrial adsorbents to simulate an authentic fixed bed adsorber with realistic gas flow compositions or very representative small sample amounts of powders to investigate mixture gas equilibria on novel materials. Sample preparation can be performed in-situ using either inert gas flux or vacuum at temperatures up to 400 °C. Regulating the temperature of the inlet gas and the adsorber allows for uncompromised temperature control throughout the measurement. High precision mass flow controllers are used for quick and stable gas mixing and flow rate control. The adsorber pressure is regulated automatically and the pressure drop between adsorber inlet and outlet will be measured by the device.

The direction of gas flow through the adsorber for mixSorb L can be switched between up and down-stream to allow investigation of bed regeneration, adsorption desorption cycles, and long-term stability of the technical adsorbent.

Applications

- Determination of breakthrough curves
- Investigation of kinetic performance of adsorbents
- Investigation of co-adsorption and displacement phenomena
- Determination of sorption selectivity
- Dynamic adsorption and desorption experiments
- Determination of single- and multi-component adsorption data
- Investigation of heat profiles along the adsorber bed
- Measurement of adsorption in the presence of water or others vapors
- mixSorb L series: Reasonable downscaling of industrial separation processes
- mixSorb S series: Possibility for R&D groups to get data of dynamic adsorption and desorption experiments with low samples mass on powder (< 1 cc)

mixSorb L series with 100 ml adsorber

a Adsorber pressure

b Intelligent lighting

c Adsorber with four temperature sensors

d TCD bypass valve

e Up to four gas input and one vent output

mixSorb
Dynamic Sorption Analyzer

Safety

A robust adsorber design, protective doors, an illuminated working area, and a clearly structured PC-control interface assure a safe and convenient instrument operation. The adsorber pressure is continuously measured and displayed on the front of the instrument, even if the instrument is turned off. Work area illumination changes from white to red when the heating mantle temperature exceeds 80 °C, indicating an elevated temperature in the work area. Safety guard sensors for the detection of flammable gasses are standard in all mixSorb instruments. In the event of a gas leakage the instrument is brought to an idle state and shut off automatically.
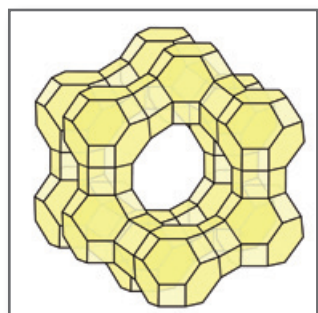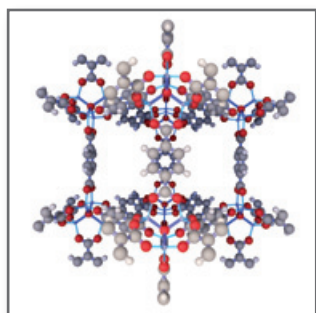
Signal Detection

The adsorber outlet gas composition is measured with a built-in Thermal Conductivity Detector (TCD) situated in a thermostatted environment for most precise and stable signal detection. The TCD is pre-calibrated by the manufacturer for 13 common gas mixtures. You are working with a different system? No problem! Due to fully automated bypass calibration, almost any binary mixture can be detected.

The additional mass spectrometer control capability allows for the quick and easy interfacing of an optional mass spectrometer.

Gas Purification

Gas Separation

Energy

Carbon

Zeolites

Metal Organic Framework

Features

- Easy and intuitive PC-control
- Automated processing of sequential adsorption and desorption experiments
- In-situ sample preparation
- Automated regulation of the adsorber pressure
- Up to 4 high precision mass flow controllers
- Automated built-in gas mixing
- Evaporator option for introduction of water and other vapors
- Measurement of inlet and outlet gas composition
- Temperature control of inlet gas and adsorber
- Determination of heat profiles within the adsorber bed with up to four temperature sensors
- Monitoring of pressure drop along the adsorber
- Built-in thermal conductivity detector (TCD)
- Optional gas analysis via interfaced Mass Spectrometer
- Safety guard sensor for flammable gases for automatic shut down
- Enhanced Safety by intelligent illuminated workspace

Built-in Sample Preparation up to 400 °C

Investigation of hydrophilic materials
(i.e., Zeolites and Silicagels).

Linear Heating Rates up to 10 K/min

Slow and controlled heating of sensitive materials.

Automated Gas Mixing

No need for multiple pre-mixed gas tanks, increasing
flexibility of measurement conditions.

PC Control and Data Acquisition

Completely programmable for operator-free analysis.

Detection Pressure on Adsorber Inlet and Outlet

Observation of pressure drop in the column.

Built-in Thermal Conductivity Detector (TCD)

Automated time-resolved measurement of outlet
gas composition.

Bypass Connection

Measurement of inlet gas composition before analysis.

Automated Pressure Regulation

Completely programmable for operator-free performing
of pressure steps.

Optional Gas Analysis via Interfaced MS

Investigation of ternary, and for analysis of trace quantities.

Battery-Backed Pressure Display

Monitoring of column pressure, even when power is off.

Safety Guard Sensor

Automatic shut down and warning in PC software after reaching 1 % C_xH_y in manifold (i.e., caused by leaks).

Switching Illumination

Light in working area switched from white to red, if the column temperature is above 80 °C (preventing of accidental contact with hot surfaces).

Large Flow Range

Investigation of mixture equilibria and kinetics, downscaling of
industrial processes.

Temperature Regulated Inlet Gas and Adsorber

For uncompromised temperature control during the measurement.

Automated Counter Current Operation

Downscaling industrial PSA-processes.

Small Blank Volume

Optimized for short dwell times.

Sample Volume

especially for R&D groups for small powder amount (< 1 cc)

Alternative Sample Cells

A small adsorber option allows separation studies with very low concentrations and vapors on high performance materials with shorter measurement times. Other adsorber sizes as well as customized membrane and filter cells are available on request.

Circulator Option

A single circulating bath can be used for the precise temperature control of the inlet gas and the adsorber between -20 °C and 90 °C.

Beaker Option

The Beaker Option gives the user the opportunity to regulate the temperature of the adsorber column.

Mass Spectrometer Option

With the Mass Spectrometer Option, a mass spectrometer can be controlled by the mixSorb Manager software. Controlling includes turning the filaments on/off, starting/stopping data acquisition and live data integration.

Customized

The number and range of mass flow controllers (MFCs) is determined by the customer's needs. Mass flow controllers are available in different flow ranges. With respect to the application, a suitable set of up to four MFCs can be configured for optimal experimentation. Flexible: The number and range of MFCs can be changed at any time in field upgrades. Several options and accessories are available for further customizing mixSorb L analyzers for individual needs and applications. This includes a Kalrez® version, with better chemical resistance.

Vacuum Option

With the Vacuum-Option, a vacuum pump can be connected to the instrument. The software allows the activation of the pump to evacuate the manifold and the adsorber column during pretreatments or analyses. In addition to a more effective sample pretreatment, this option will allow the emulation of a VPSA-process (Vacuum-Pressure Swing-Adsorption) with one adsorber column.

Mass Flowmeter Option

A Mass Flowmeter can be included in the manifold to measure the mass flow after the adsorber column. Prior to 100 % breakthrough the mass flow entering the column can be different from flow coming out, especially in high concentrations. This option is useful, when the calculation of flow rate changes is not possible.

Evaporator Option

This option injects a liquid into a flash evaporator which is then mixed with a carrier gas stream, introducing a vapor at controlled concentration. This allows the evaluation of the adsorption charateristics of an adsorbent in the presence of a vapor (e.g. water) or the separation of the vapor from a gas (i.e. VOC removal). Up to two evaporators can be included in one instrument.

Evaporator Option

A well tempered saturator with high performance is used in mixSorb S for water and other liquids to get a stable vapor feed for low flow rates by high pressure up to 68 bar. The fill level of the saturator is displayed in the mixSorb manager software.
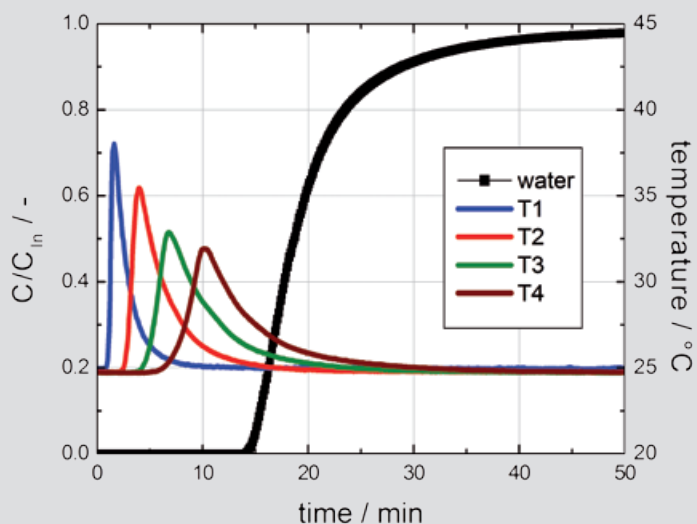
Figure: Breakthrough curve of water on 80 g activated carbon (30 % RH at 25 °C in N_2, gas flow 4000 ml (STP) /min) measured by mixSorb L.
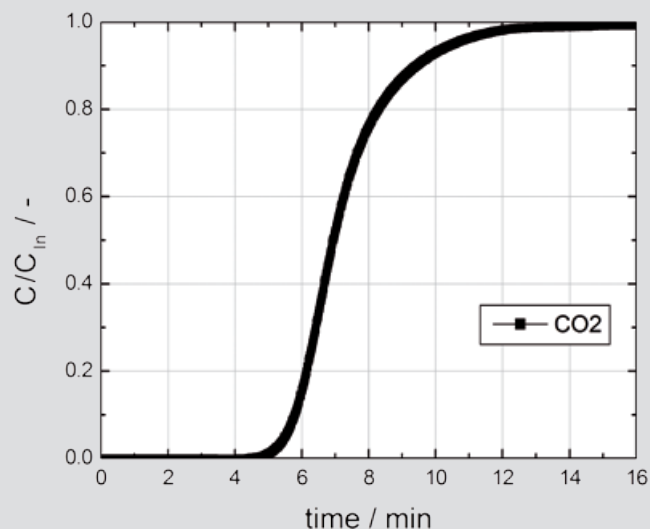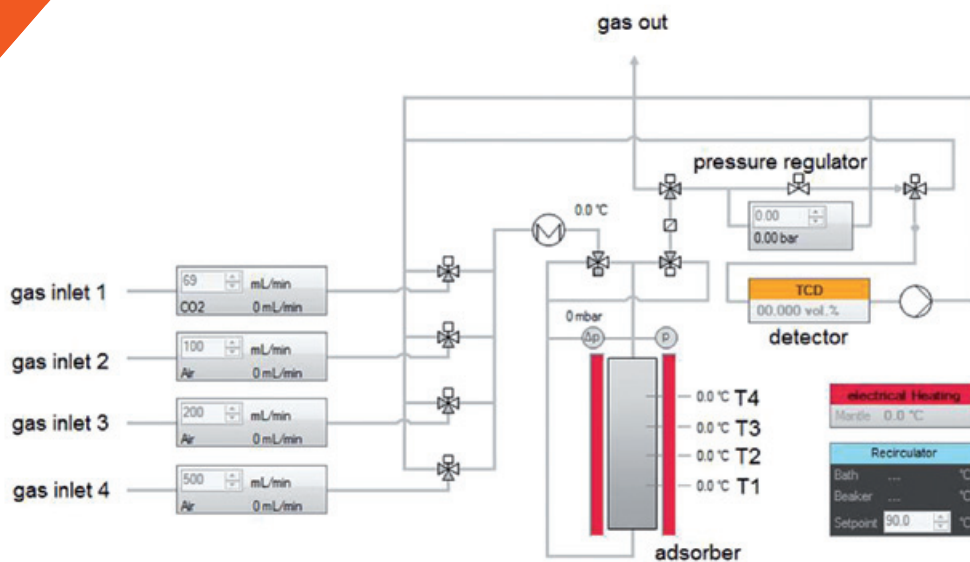

Figure: Breakthrough curve of 5 % CO_2 in N_2 on 0.5 g activated carbon at 5 bar (total flow rate 20 ml/min) measured by mixSorb SHP.

The configuration, installed options, and current status of the system are visible at a glance on the main display.

Specifications

Feature	L	S	SHP
No. of MFCs	2 – 4	2 – 4	2 – 4
MFC types	8	4	3
Pressure range	0.1 – 10 bar	0.1 – 8 bar	0.1 – 68 bar
Required inlet pressure	15 bar	10 bar	85 bar
Evaporator	Direct liquid injection	Saturator	Saturator
No. of evaporators	1 – 2	1 – 2	1 – 2
Differential pressure measurement	Direct (sensor)	Indirect	Indirect
Counterflow possible	Yes	No	No
Bypass pressurized	No	Yes (important for saturator)	Yes (important for saturator)
TCD	Feed sampling pump	Force flow through TCD	Force flow through TCD
Temperature sensors in sample bed	1 - 4	1	1
Sample amount	4 – 130 cm^3	< 1 cm^3	< 1 cm^3
Blank volume / Sample volume	1 – 20	10	10
Temperature range heating mantle	ambient to 400 °C		
Temperature range circulator bath	-20 °C to 90 °C		
Height	960 mm (38 in)		
Width	860 mm (34 in)		
Depth	640 mm (25 in)		
Weight	120 kg (265 lbs)		

mixSorb Software

mixSorb Manager

The user-friendly control software mixSorb Manager provides real-time control and programmable operation of all system functions, such as gas flow rates and gas compositions, flow paths and direction of flow, temperature ramps and dwell times, etc. The Software assists the user in the configuration of complex adsorption and desorption sequences. Analysis sequences can be processed fully automated and unattended. The definition of intelligent start/stop criteria provides unprecedented flexibility and facilitates a most efficient and economic operation (conserving gas usage). Further software features are:

- Real-time data presentation
- Manual mode control
- Demo mode for operator training and education
- Historical data trending
- System alarm settings
- User account management (hierarchy of user access levels)
- Well-structured log files
- Auto-save function

The status of all sensors and valves, the path and direction of gas flow, and all relevant system information for safe and convenient operation can be viewed at a glance on the controlling PC.
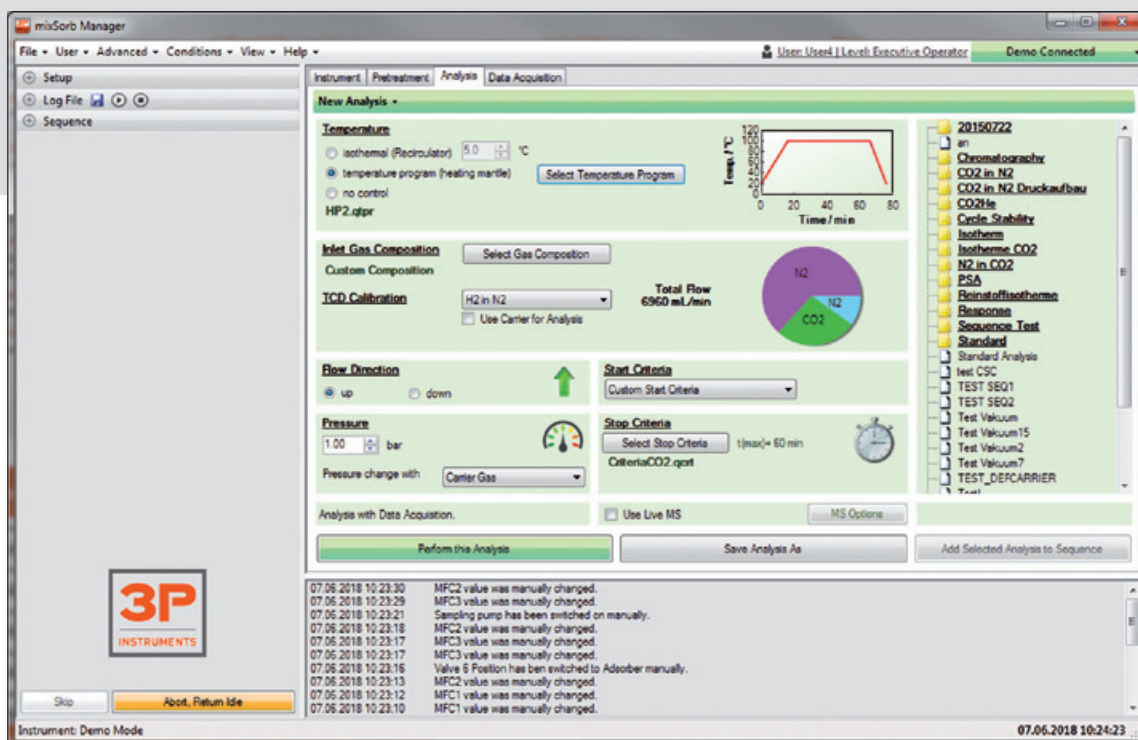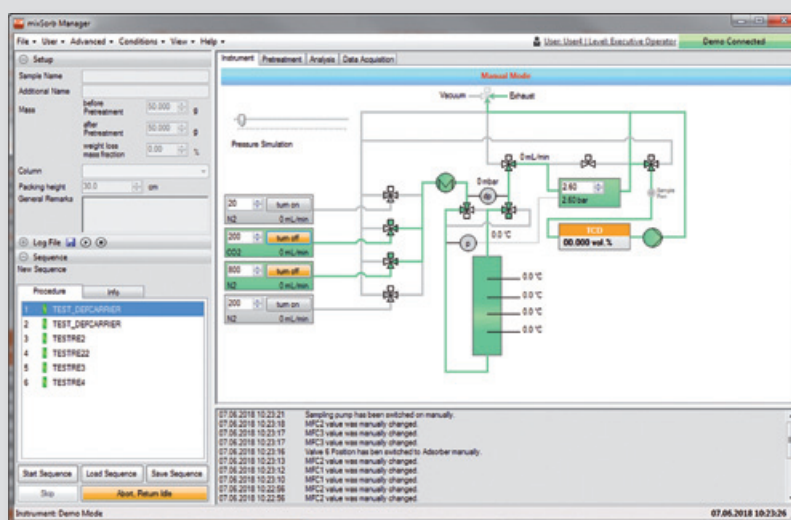
3P sim

The included simulation software 3P sim provides sophistica-ted data reduction and simulation capabilities, such as:
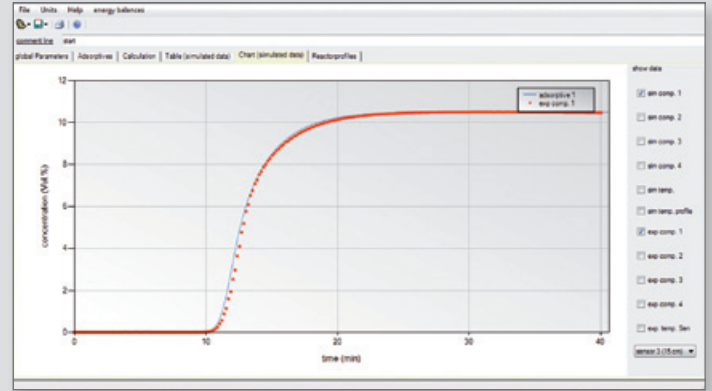
- Integration of breakthrough curves
- Comprehensive parameter studies
- Simulation and prediction of breakthrough behavior and heat profiles

- Calculation of single- and multi-component adsorption data
- Determination of selectivity, affinity, and kinetic coefficients

Complex calculations or basic research, 3P sim accommodates it all, making it a powerful tool for both industry and academia.
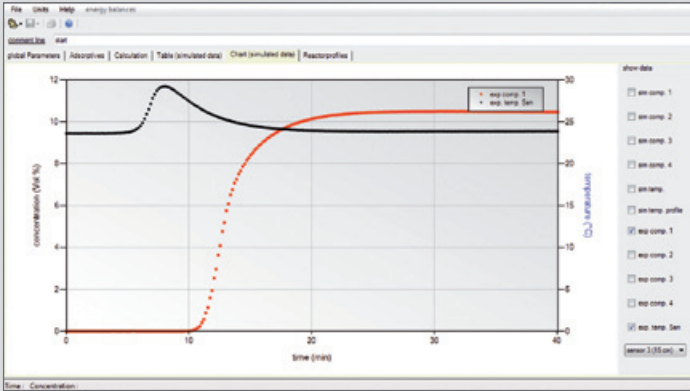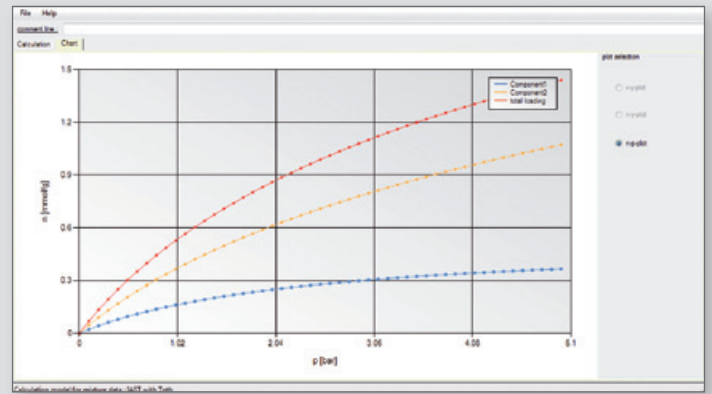

A typical breakthrough curve (red) with the corresponding temperature signal (black) measured at the bottom of the ad-sorber bed.
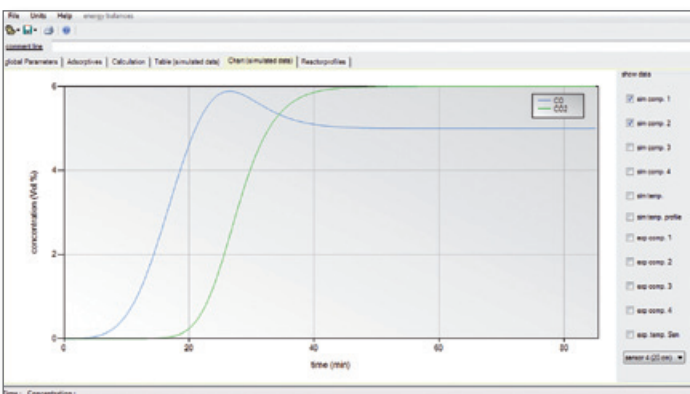

Comparison of a measured breakthrough curve (red) with the simulated breakthrough behavior (blue) computed with 3P sim.
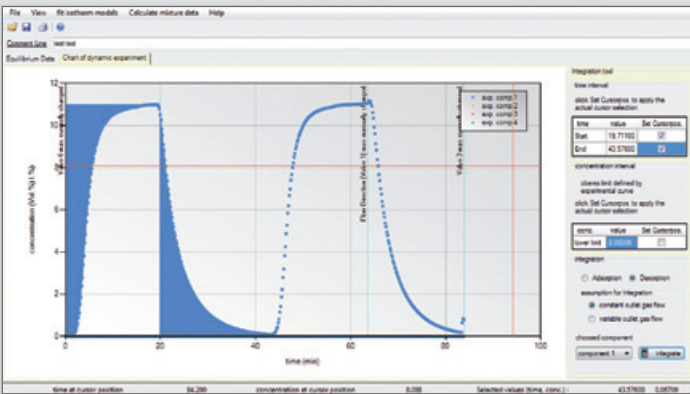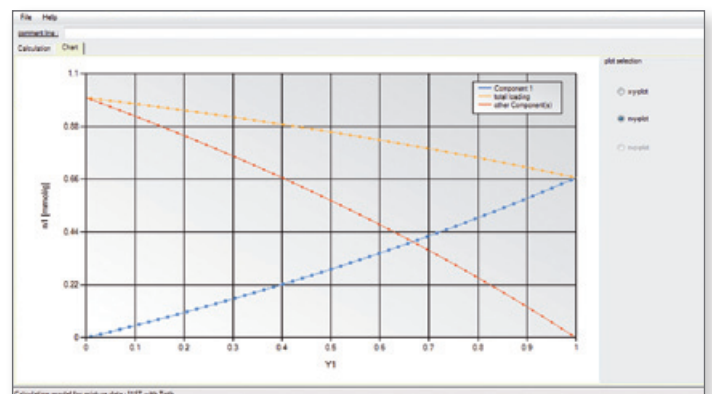

Integration of consecutive adsorption-desorption cycles.

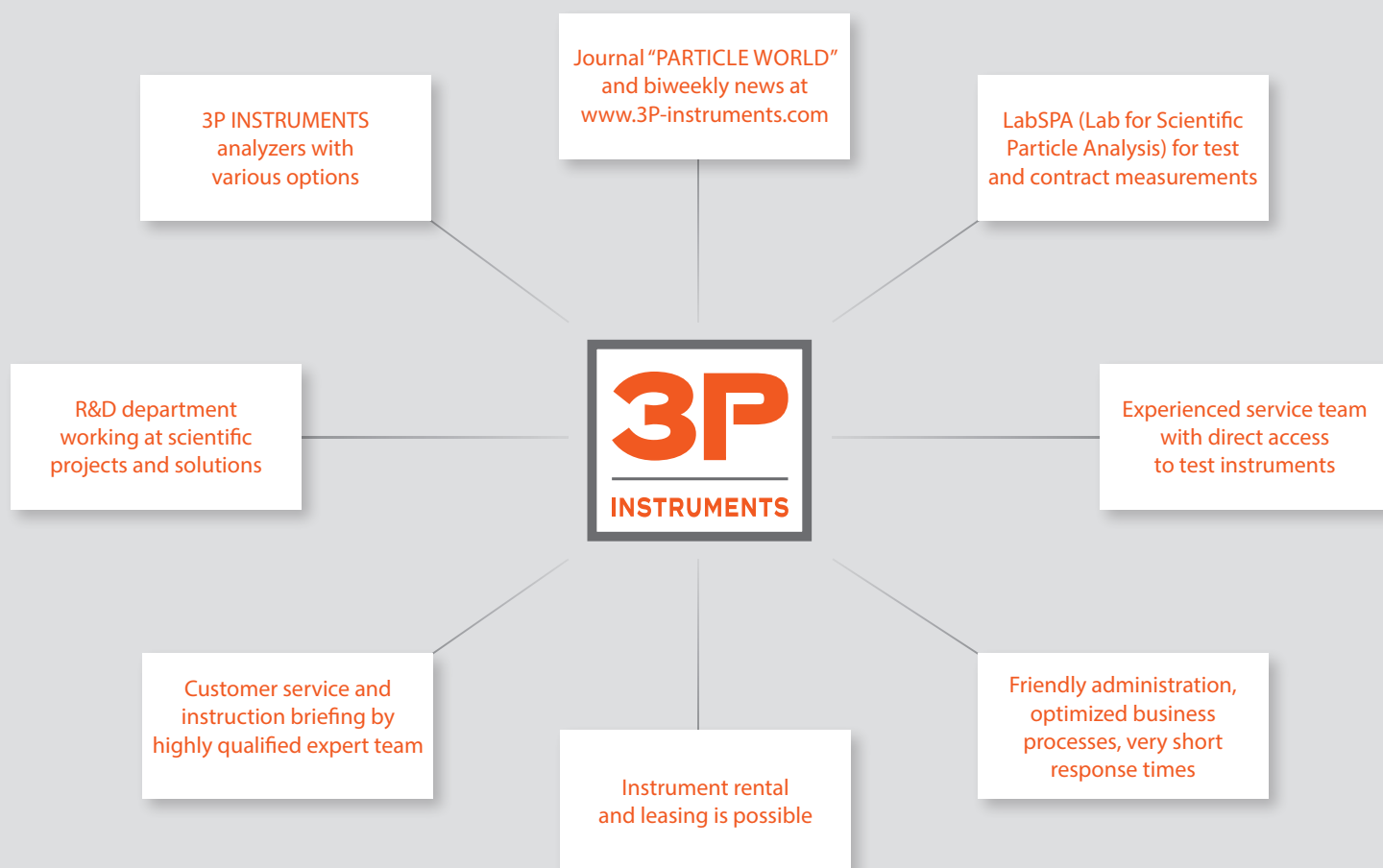

Calculated multi-component sorption equilibria.

Calculated Breakthrough curves of a binary gas mixture in helium carrier gas.

Calculated multi-component sorption equilibria showing total loading (yellow) and partial loading (red and blue).

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collects and summarizes all the features, advantages, examples
and knowledge of dynamic sorption (or flow sorption) methods.
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