Surface Measurement Systems
World Leader in Sorption Science

DVS
ADVENTURE

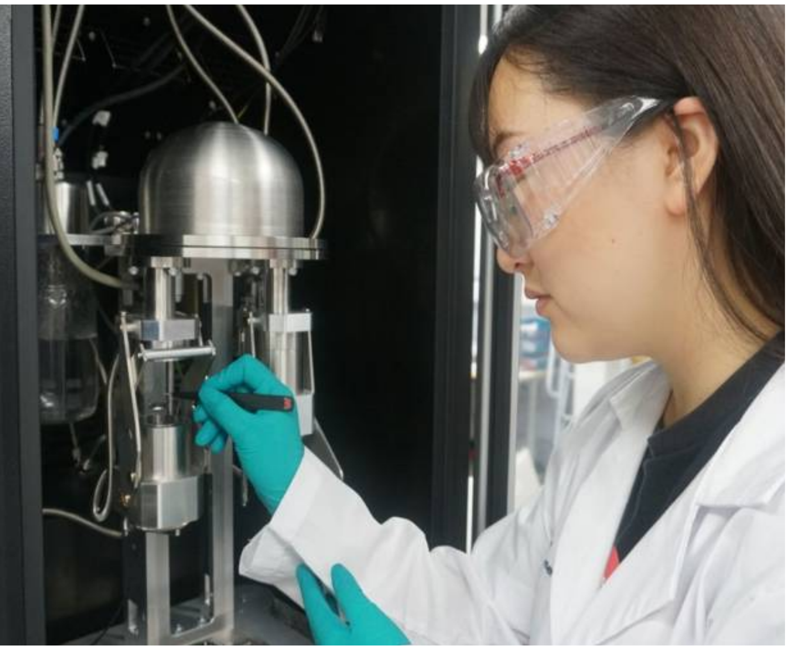
Automated Dynamic Water Vapor Sorption Analyzer

- Dynamic water vapor sorption isotherms from 5 °C to 85 °C
- Water sorption kinetics
- *In-situ* preheating/drying of samples to 200 °C
- Water diffusion and permeation measurements
- Optional Fiber Optic Raman / NIR accessory
- Optional Color Video Microscopy
- True0™ drying at 0.0% RH
- Next generation experimental control and evaluation software

www.surfacemeasurementsystems.com

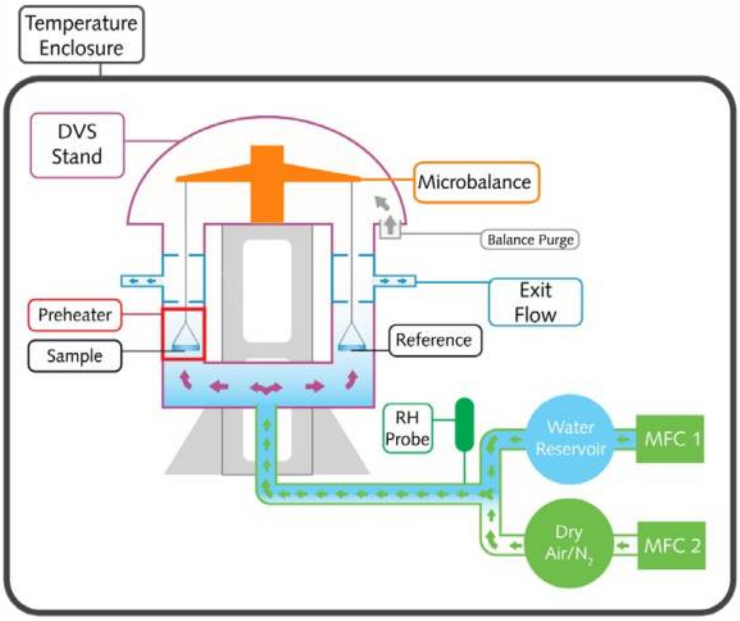
DVS Adventure Water Vapor Sorption Analyzer

Dynamic Vapor Sorption (DVS) is a gravimetric sorption technique that measures the rate and amount of solvent sorbed and released by a sample, such as a dry powder absorbing or releasing water. The DVS accomplishes this by varying the vapor concentration surrounding the sample and measuring the resultant mass changes.



Applications

- Hygroscopicity of pharmaceutical solids
- Moisture induced T_g and phase transitions
- Amorphous content determination of solids
- Diffusion and permeation in polymers
- Food, flavors and fragrances
- Sorbents
- Wood, cellulosic and natural materials
- Composites
- Hydrophillic and hydrophobic materials



Standard DVS Adventure Schematic

Hardware

- Open stainless steel stand design enabling easy access to sample pan while minimizing static electric charging
- Accurate and uniform temperature across a broad temperature range (from 5 to 85 °C)
- Optional IR, Raman and video imaging with integrated control software
- Quick and easy to change reservoir bottle
- Variable sample area for multiple sample geometries

Software

The software package provided with the DVS Adventure allows the users to create and customize experimental methods while enabling the full analysis of the kinetic data collected. Examples of the control and analysis software used in a standard water sorption experiment are outlined below.

•Control Software

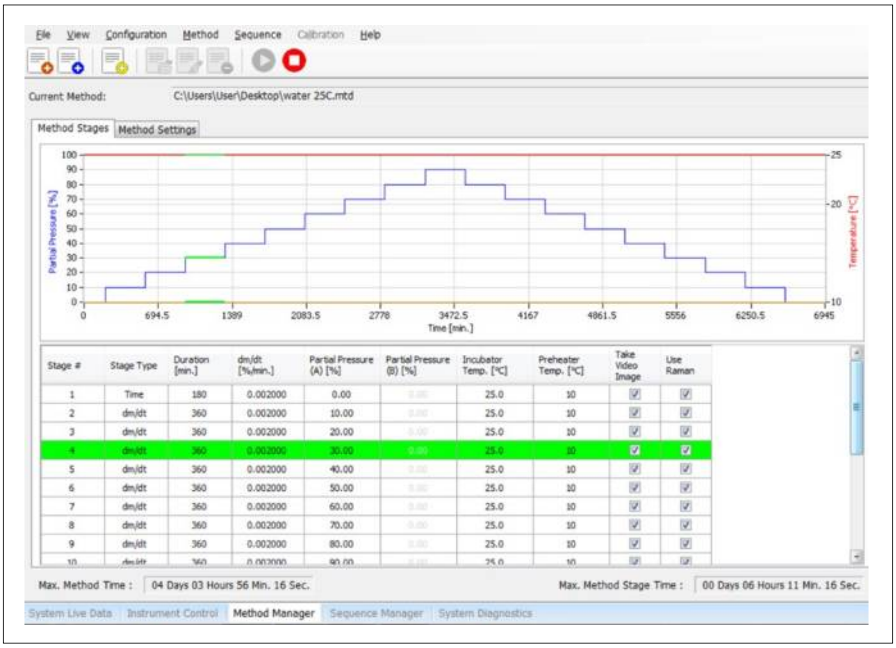


Figure 1. The above graph shows the method panel within the method manager. It displays numerically and graphically the current method for a water sorption experiment at 25 °C. The active stage of the ongoing experiment is highlighted in green. Figures 2 and 3 (below) are typical data generated by this method.

•Analysis Software

Water Sorption Data

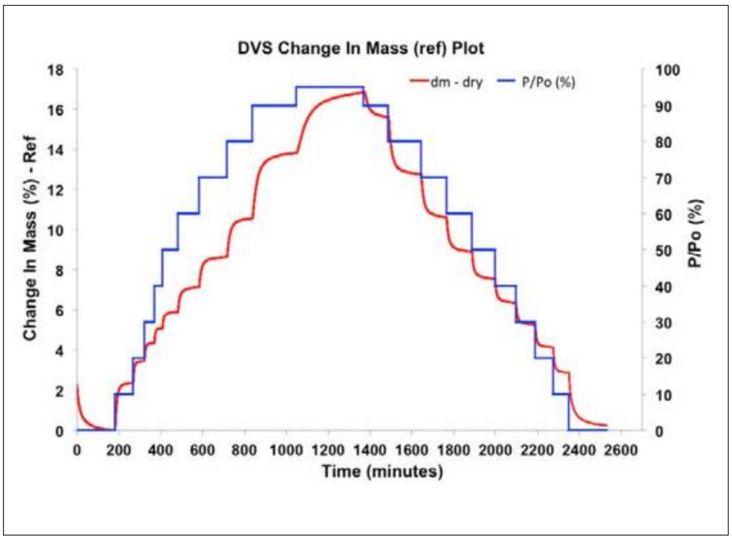


Figure 2. Water sorption kinetics of micro-crystalline cellulose membrane at 25 °C

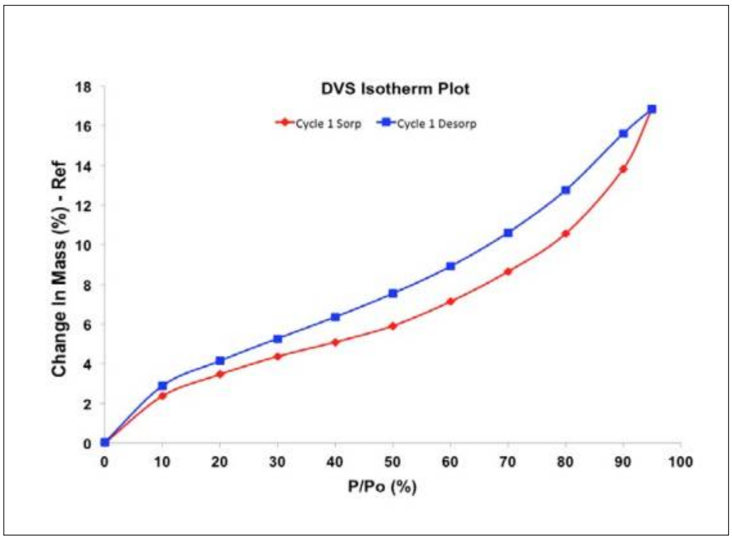


Figure 3. Water sorption isotherm of micro-crystalline cellulose membrane at 25 °C

Outstanding Performance

The DVS Adventure allows for the collection of high quality data, owing to the outstanding Ultrabalance performance (Figure 4), precise vapor generation (Figure 5) and accurate temperature control (Figures 4, 6, 7).

Mass and Temperature Measurement

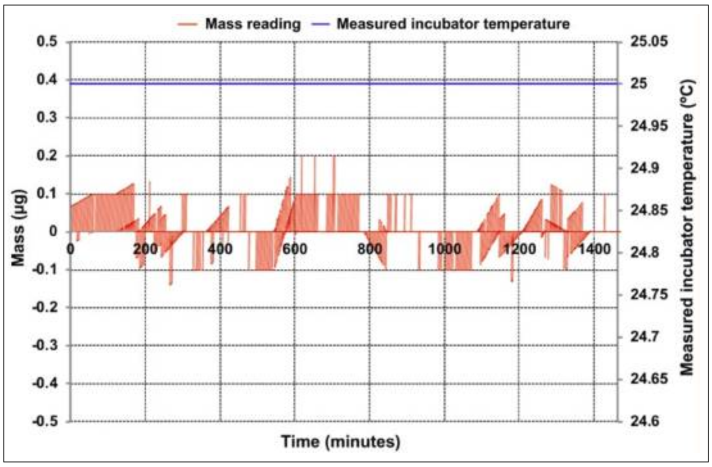


Figure 4. DVS mass baseline stability plot over 24 hours, in µg

- Mass changes at a resolution of 0.01 µg for low mass balance
- Root mean square noise of ≤ 0.3 µg for low mass balance (averaged over 24 hours)

True0™ RH

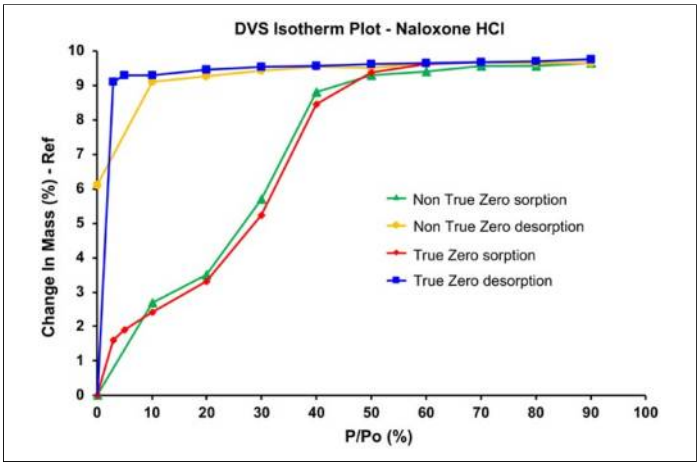


Figure 5. Comparison of Naloxone hydrochloride dihydrate water sorption isotherms

- Achieves partial pressures of water of 0.0% RH
- Hydration and dehydration kinetics below 1% RH can be readily studied

Temperature Control & Stability

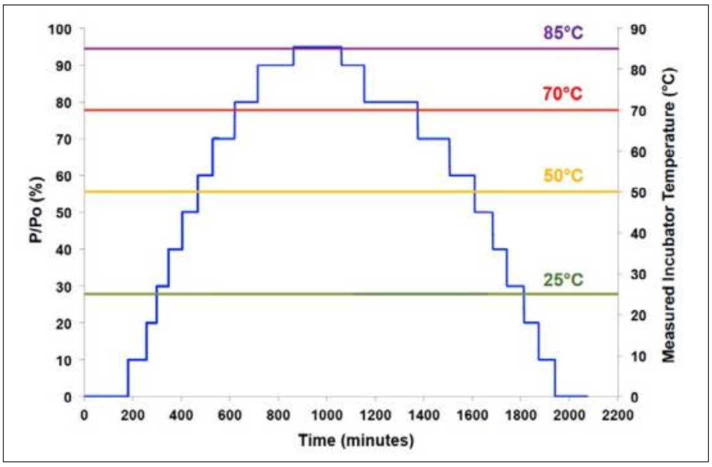


Figure 6. Multiple kinetic plots of relative humidity at different temperatures*

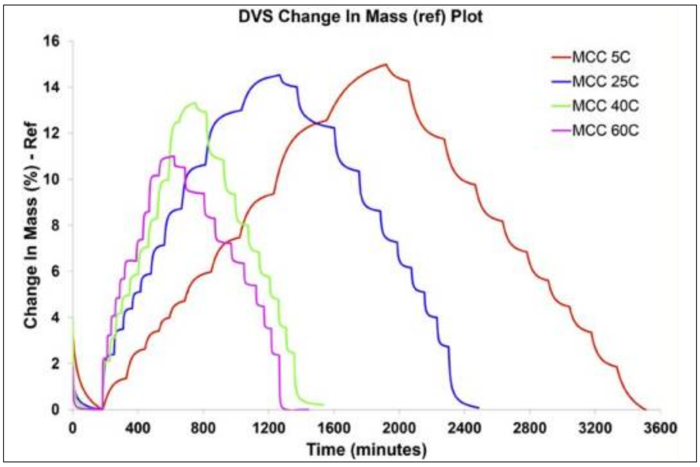


Figure 7. Microcrystalline cellulose (MCC) kinetic mass plot for different temperatures

- Stability is ± 0.05 °C at 25 °C over 6 hours
- The single temperature enclosure environment prevents condensation issues typically found in instruments with multiple temperature zones
- Accuracy of humidity generation and high temperature stability guarantee stable isotherm experiements

**For extended experimental operation at 85% RH at 85 °C an optional heated reservoir accessory is available.*

Stability study of hydrates

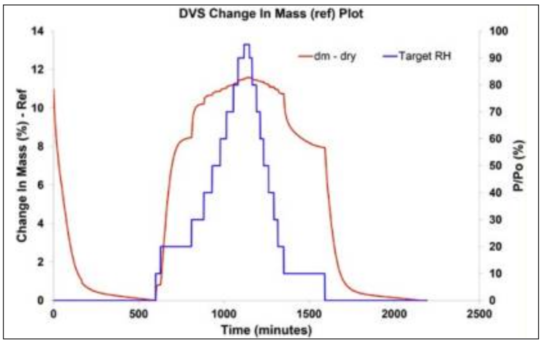


Figure 8. Water sorption kinetics of Amoxicillin Trihydrate at 40 °C

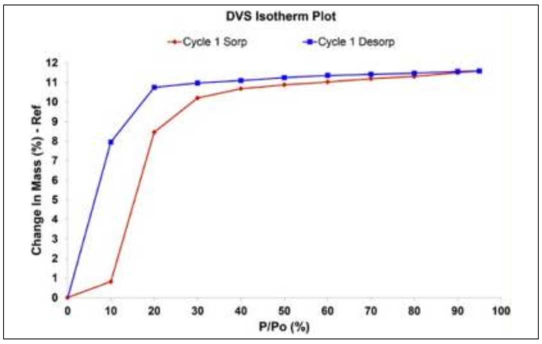


Figure 9. Water sorption isotherm of Amoxicillin Trihydrate at 40 °C

- The accurate humidity generation and excellent balance stability allows for the study of hydrate formation.
- More information about the methodology can be found in our Application Note 36 (Investigation of Hydrate Formation and Lose using the DVS).

Phase transition & recrystallization study

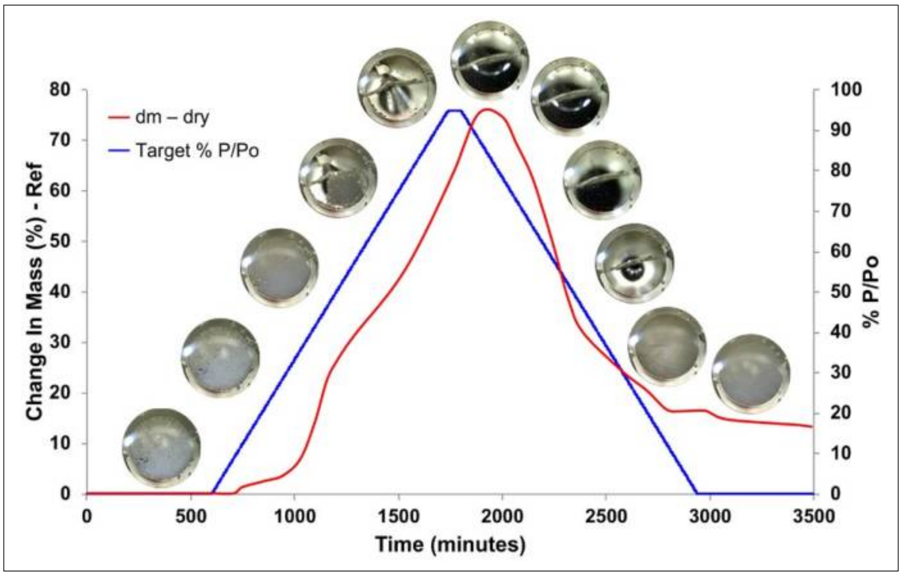


Figure 10. Moisture sorption of Amino Acid at 35 °C, collected with a ramping method & camera accessory

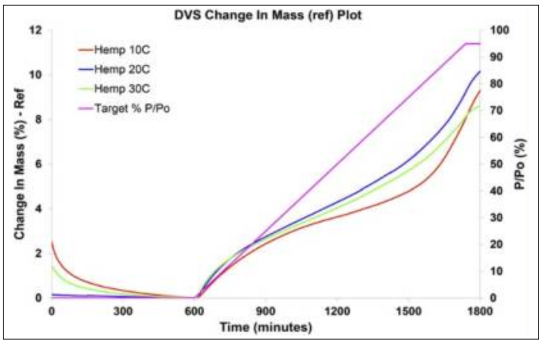


Figure 11. Water sorption of Hemp collected at 10 °C, 20 °C and 30 °C using a moisture ramping method

- Experiments performed with moisture ramping method can be used to calculate the critical % RH of the phase transition of the Hemp sample.
- More information in regard to phase transition/ recrystallization studies can be found in our Application Notes 35 and 42.

Moisture-induced phase change for Hemp

Temperature	T_g RH
10˚C	80.1%
20˚C	74.1%
30˚C	70.2%

Modular Capabilities

Raman & IR Spectroscopy

- Fully integrated hardware/software solution for triggering and capturing Raman or IR spectra during sorption experiments
- Simultaneous operation of Raman or IR and optical microscopy during the DVS experiment

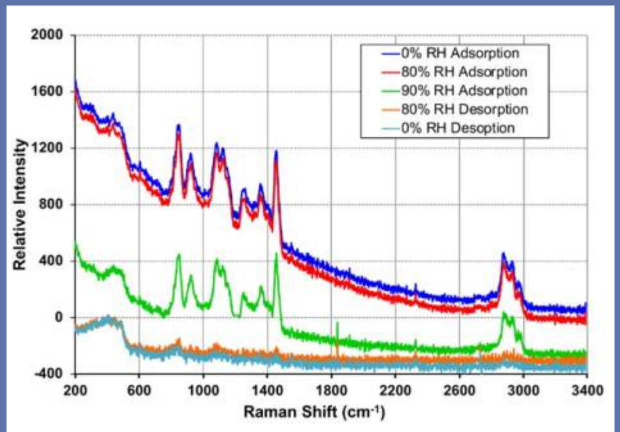


Figure 12. Raman spectra of Hydroxypropyl Methylcellulose (HPMC) at 25 °C

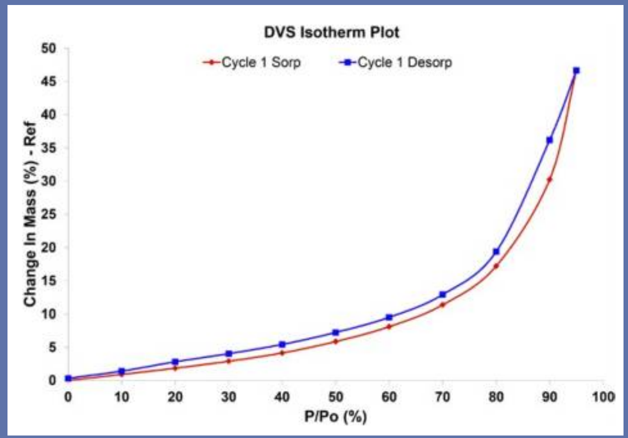
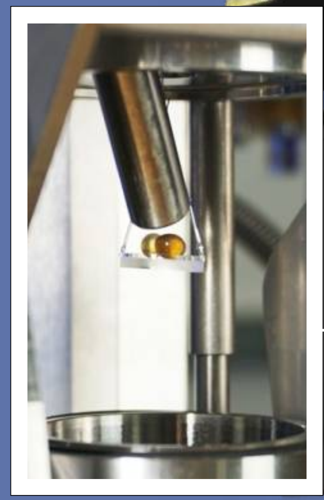
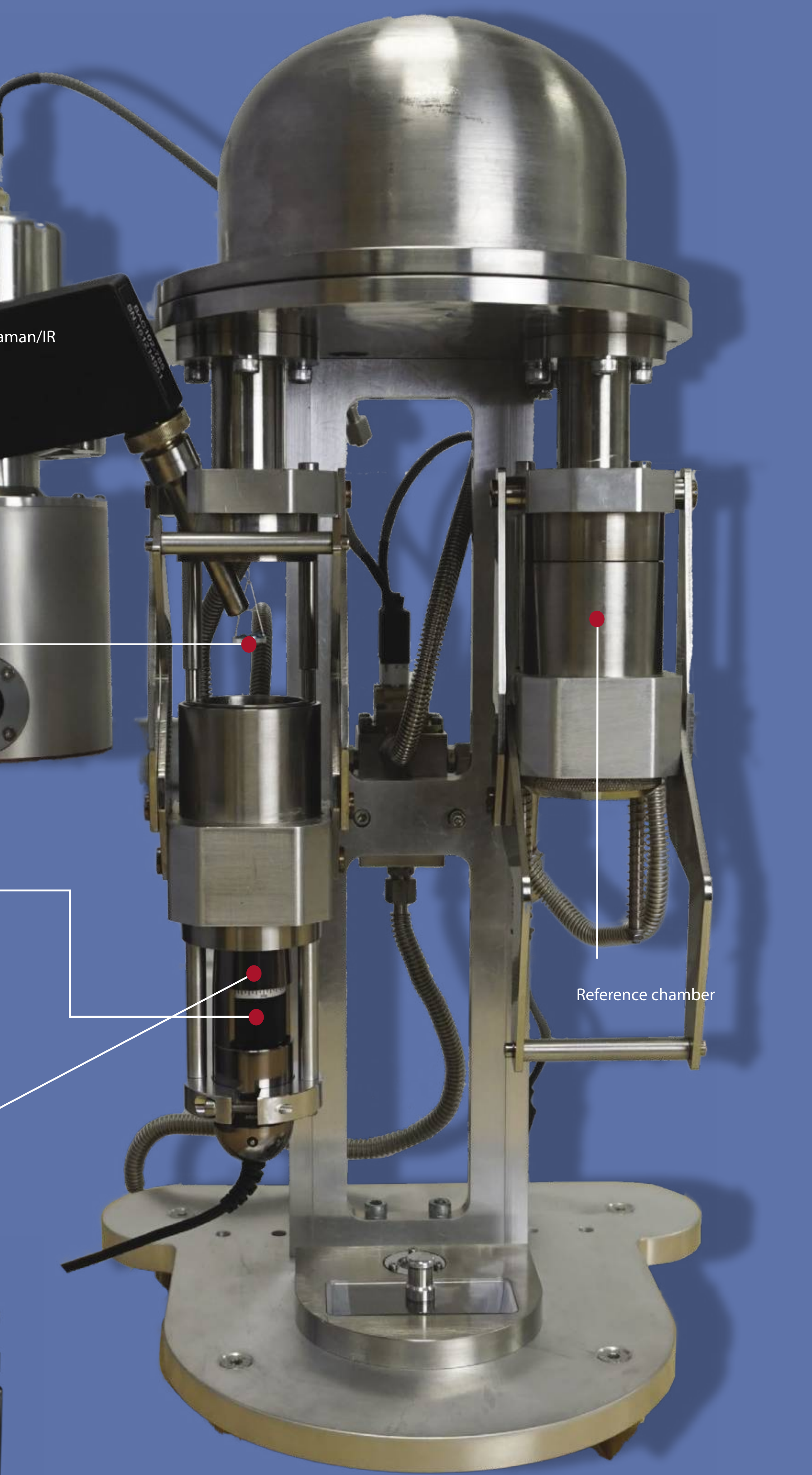


Figure 13. Moisture sorption isotherm of Hydroxypropyl Methylcallulose (HPMC) at 25 °C

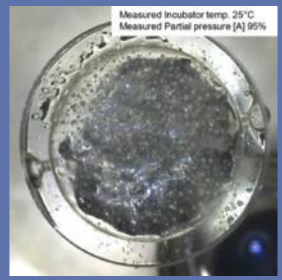




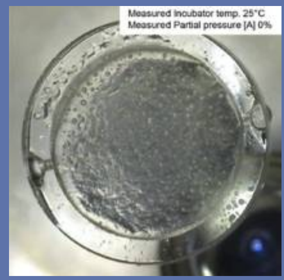
Microscopy & Video

- 1.3 megapixel color camera
- Up to 200x optical zoom
- Images have time-date-temperature-partial pressure stamps
- Grid overlay and calibration for measuring dimensional change
- The images can be composed into a timelapsed video

1) Sorption 0% RH (HPMC)



2) Sorption 95% RH (HPMC)



3) Desorption 0% RH (HPMC)

High Temperature Preheater*

(for drying and curing)

- In-situ degassing/activation of samples up to 200 °C
- The preheater temperature is controlled using a Pt-100 positioned below the sample pan
- User programmable and controlled temperature ramps or steps

NOTE: Preheater cannot be used with other modular accessories

Heated reservoir accessory*

- The heated reservoir replaces the standard glass bottle mounted on the left of the stand
- Designed for extended humidity generation 85% RH at 85°C, with fully automated temperature

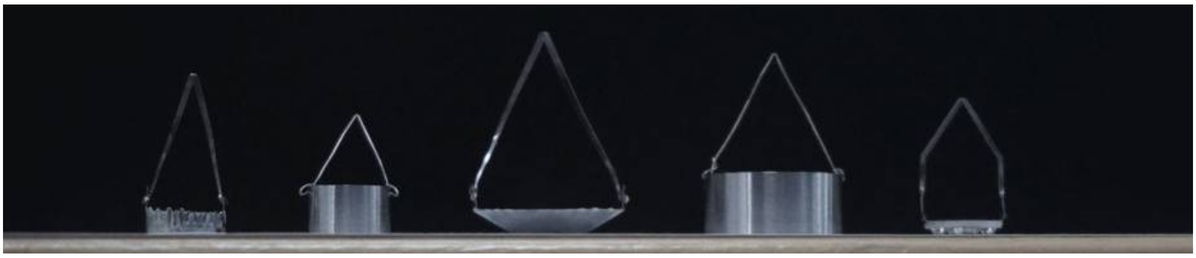
*For more information on the specification of these accessories, please contact sales@surfacemeasurementsystems.com

Accessory Compatbility

The **DVS Adventure** is compatible with a range of accessories that can greatly expand the scope and variety of your research. See the table below for a summary of the available options, and how they can work together.

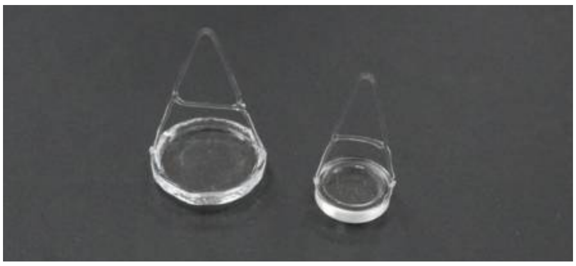
	Temperature Range (°C)	Preheater	Camera	Raman	IR	Heated Reservoir
Preheater	Up to 200 °C		✗	✗	✗	✓
Camera	Up to 50 °C	✗		✓	✓	✓
Raman	Up to 50 °C	✗	✓		✗	✓
IR	Up to 50 °C	✗	✓	✗		✓
Heated Reservoir	Up to 85 °C	✓	✓	✓	✓	

Consumables



Metal Pans: from left to right, 10mm Standard metal pans, 10mm deep pans, 17mm pans, 17mm deep pans, 10mm Aluminium Foil Holder.

Payne Diffusion Cell: Small (9mm) and Large (18mm, only for high mass balance configuration).



Quartz Video Pans: Small (10mm) and Large (19mm, only for high mass balance configuration).

For more information on consumables or accessories, or to place an order, contact our team at sales@surfacemeasurementsystems.com

Technical Specifications

Temperature

Temperature controlled enclosure

Control range: 5 °C to 85 °C

Temperature stability ± 0.05 °C over 6 hours

Temperature resolution 0.01 °C

High Temperature Pre-heater for drying samples

200 °C (maximum local temperature)

Heating ramp rates: up to 5 °C/min

Temperature sensor: Pt-100

Adventure Stand

Manifold: 316 stainless steel

Seals: Viton® and Kalrez® or equivalent

Tubing: 1/4 inch stainless steel

Water Reservoirs

1 glass reservoir as standard (500 ml, 1000 ml available)

Optional heated reservoir [1]

Flow Control

High accuracy digital mass flow controllers

Wide dynamic range - turndown ratio 1000:1

Carrier Gas - Dry air or Nitrogen

Relative Humidity

Relative humidity range from 0 to 98% for 5-60 °C [2]

Relative humidity range from 0 to 85% for 60-85 °C [1,2]

Relative humidity resolution ± 0.1%

Relative humidity stability ± 0.1% over 6 hours

RH range accuracy from 5 - 60 °C ± 0.5% [3]

RH range accuracy from 60 - 85 °C ± 1% [1,3]

Mass Measurement

Ultrabalance Low Mass

Maximum load: 1000 mg

Mass change: ±150 mg

Resolution: 0.01 µg

Balance noise: ≤ 0.3 µg [4]

Ultrabalance High Mass

Maximum load: 5000 mg

Mass change: ±1000 mg

Resolution: 0.1 µg

Balance noise: ≤ 3 µg [4]

System Information

Dimensions: 520 mm (W) x 980 mm (H) x 610 mm (D)

Weight: 80 kg (180 lb)

Electrical: 200-240 V, 50/60 Hz, 1500 VA

System Software

DVS Control Software

- Sample pre-heating
- Vapor sorption
- Temperature changes in a single experiment
- Ramp or step changes in relative humidity
- Automated video image and Raman spectra acquisition
- Complex isotherm experiments
- Experimental stages may be based on fixed-time or a user-defined dm/dt criteria
- Experiments may include half, full or multiple partial pressure or temperature cycles
- Windows™ 10

DVS Analysis Software

- Isotherms
- Permeability and diffusion
- Kinetics information
- Surface area models
- Amorphous content
- Heat of sorption
- T_g determinations

Software Options

Standard

- Control Software
- Standard Analysis

Advanced

- Advanced Analysis Suite
- Isotherm Analysis Suite

21CFR Part 11 software solution (optional)

Footnotes

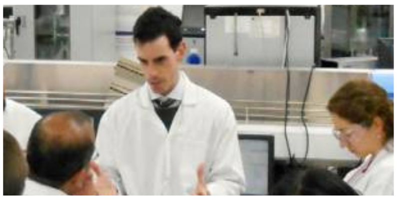
[1] Optional configuration (heated reservoir) for long term 85°C, 85% RH operation

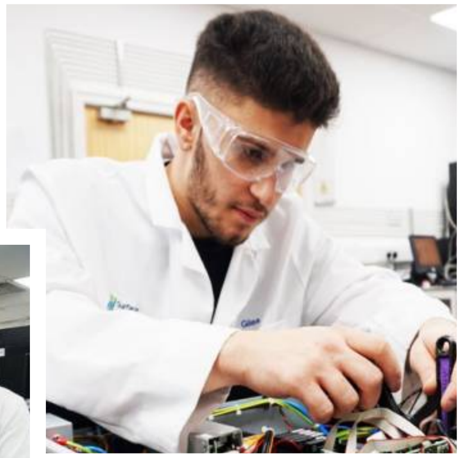
[2] System factory calibrated at 25°C. Calibrations at other temperatures upon request

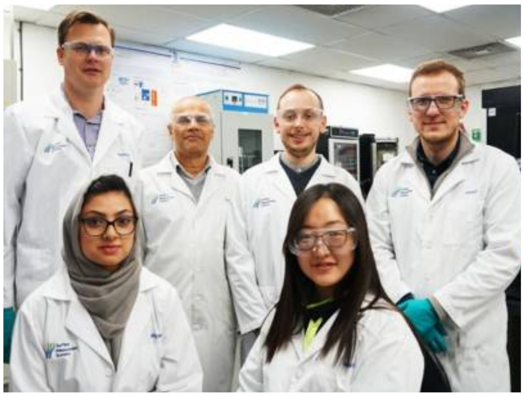
[3] 1-σ confidence level with % RH or P/P_O calibration performance based on SMS factory certified methods (Salt Calibration or equivalent method)

[4] Root mean square (averaged over 24 hours)

About Us







Surface Measurement Systems Ltd. develops and engineers innovative experimental techniques and instrumentation for physico-chemical characterization of complex solids. Our range of characterization instruments and scientific/engineering techniques has helped solve difficult problems in the pharmaceutical, biomaterial, polymer, catalyst, chemical, cosmetic and food industries, and are used by hundreds of leading laboratories and universities throughout the world.

Why us?

- Invented the DVS Technology with over 25 years of continuous innovation
- Every instrument is built upon the knowledge and experience of our industry leading sorption scientists
- Our service team provides uncompromising support to our customers and partners
- Outstanding instrument performance
- Most complete and intuitive Windows™ software for experimental control and analysis
- Winner of EEF Innovation Award 2019 and ISO 9001:2015 accredited

Surface Measurement Systems
World Leader in Sorption Science

GAS INTERNATIONAL ISO 9001:2015 REGISTERED COMPANY

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Surface Measurement Systems
World Leader in Sorption Science

DVS Carbon

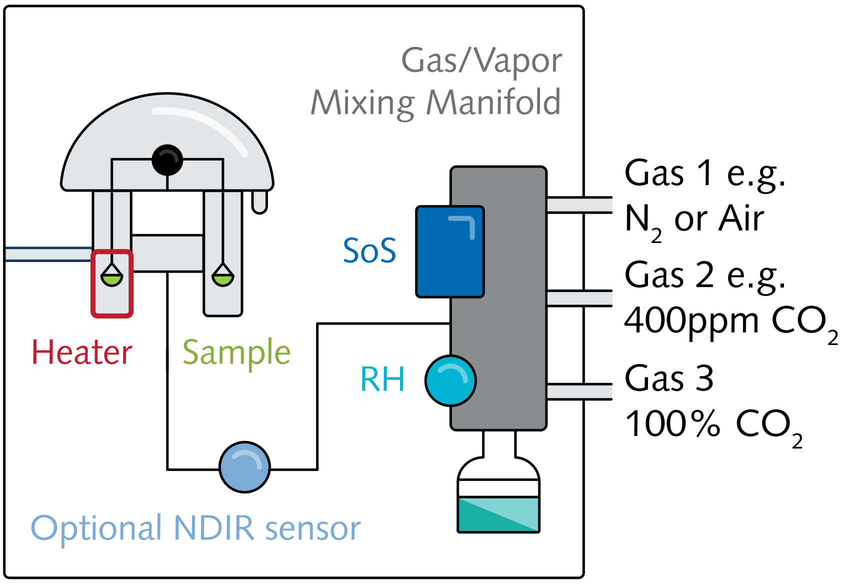
Our Range of CO_2 and H_2O Gravimetric Sorption Analyzers for Real World Conditions

Gravimetric adsorption instruments with precise CO_2 & humidity control for applications in carbon capture, utilization, & storage, with options for high throughput & low concentrations

DVS Carbon

The world's most advanced gravimetric CO_2 capture analyzer

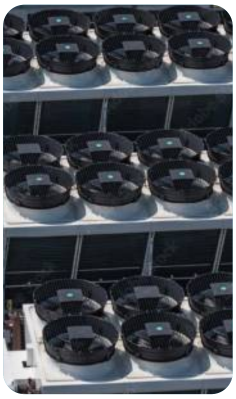
The latest addition to the DVS family, the DVS Carbon instrument range offers the first purpose-built gravimetric sorption analyzers for advanced carbon capture conditions. In most gas phase carbon capture applications, CO_2 often competes with other chemical species at adsorbent sites, especially water vapor. The new DVS Carbon range enables the measurement of CO_2 uptake in real life conditions, controlling both temperature and humidity at a broad range of CO_2 concentrations, from flue gas capture to direct air capture.



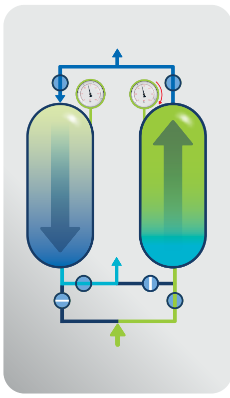
Instrument Schematic

Carbon Capture, Utilization, & Storage (CCUS) Applications:

Post-combustion capture (PCC)



Direct air capture (DAC)



Temperature/ moisture swing sorption

Carbon sequestration

Solid sorbent characterization

What is DVS?

Dynamic Vapor Sorption (DVS) is a gravimetric sorption technique that monitors sample mass change across varying concentrations to measure how quickly and how much of a gas or vapor is sorbed by a sample. Learn more on the back cover.

Key Features

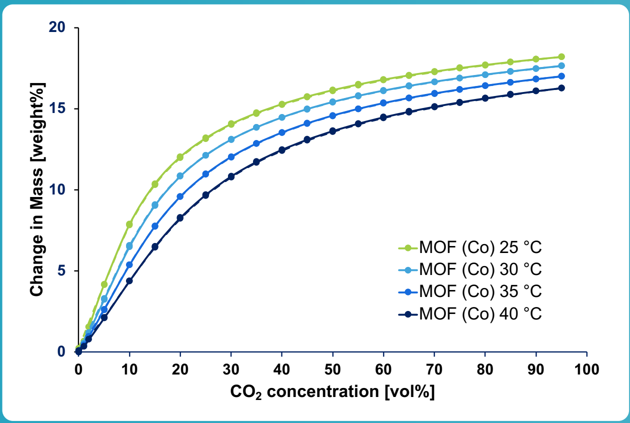


Figure 1: CO_2 uptake as a function of temperature for a Cobalt-based MOF sample. Adsorption and desorption are seen as fully reversible.

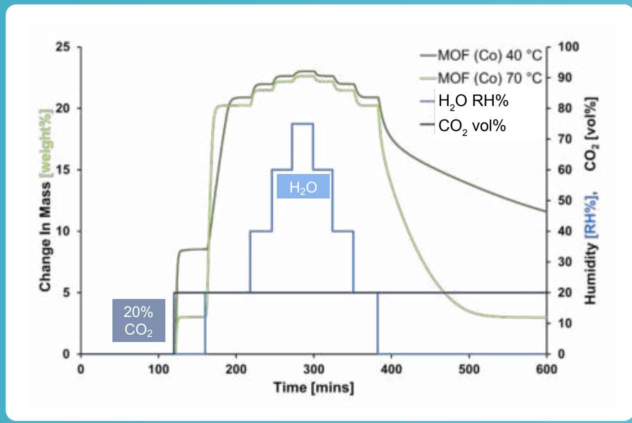


Figure 2: Co-sorption of water at 40 °C and 70 °C in the presence of 20 vol% CO_2. Total capacity is not affected by experimental temperature.

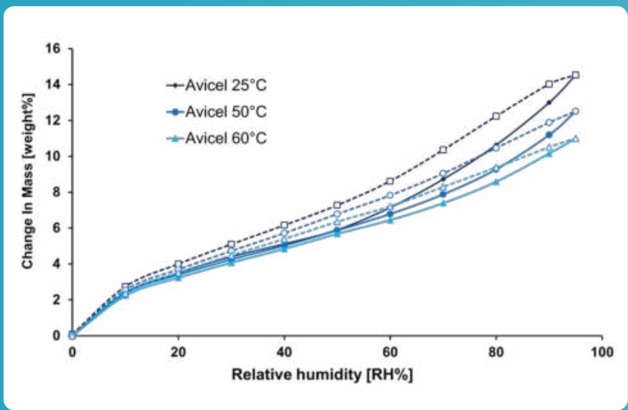


Figure 3: Isotherms of water on a hydrophobic support at increasing temperatures.

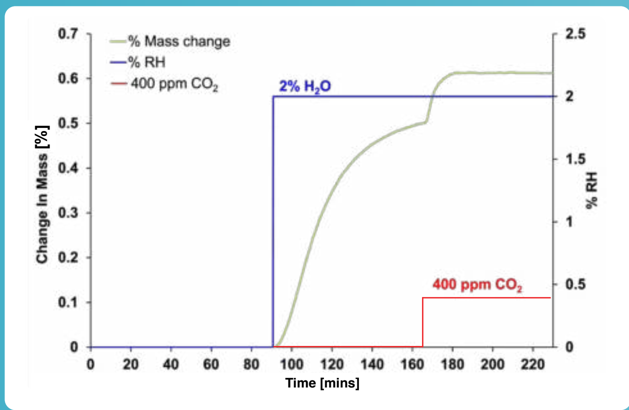


Figure 4: Kinetic plot of CO_2 uptake at atmospheric levels of an amine functionalized carbon, with 2% background of relative humidity. A noticeable increase in mass is observed when 400 ppm CO_2 is introduced.

Independent multicomponent control

- Concentration of CO_2 and H_2O can be individually controlled
- Cycling or complex sorption programs are easily created
- Concentration and temperature changed in precise steps or ramps *(Fig 2 & 4)*

Isotherms of CO_2 and H_2O

- CO_2 (Fig 1) and water isotherms (Fig 3) in real world conditions
- Kinetics for each step available by default

Multiple concentration & temperature ranges for all CCUS applications

- High CO_2 levels (Fig 2) or low ppm CO_2 range (Fig 4) can be controlled, suitable for DAC,
- PCC, or other CCUS conditions
- Lower CO_2 concentrations (e.g. 400 ppm) are generated using a pre-diluted gas cylinder

In-situ activation and regeneration

- Sample can be locally heated up to 300° C under inert or process gas
- Drying, activation, or regeneration kinetics determined directly

Optional high throughput configuration

- Accommodates five simultaneous samples with a combination low or high mass balances

Hardware

Balance, Temperature, and Humidity Stability

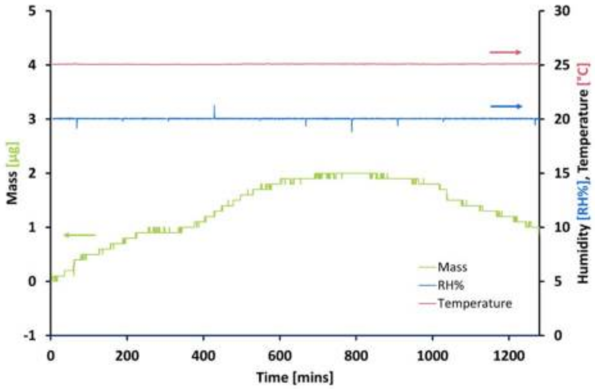


Figure 5: Baseline at 20% RH over 24 hours

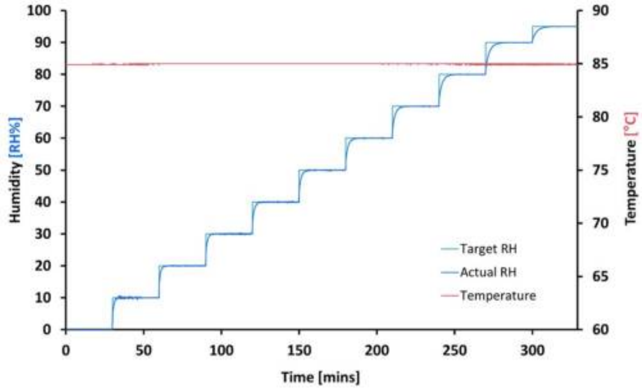


Figure 6: Stability and range of humidity generation at 85 °C

The SMS UltraBalance is a custom-built symmetric microbalance that measures microgram changes in sample weight. Temperature control ensures long-term stability, under dry and humid conditions.

- Mass changes at a resolution of 0.01 µg (for low mass balance)
- Root mean square noise of ≤ 0.3 µg for low mass balance (averaged over 24 hours)
- High throughput option available for 5x balance in parallel

An advanced mixing system and calibrated mass flow controllers enable the DVS Carbon to generate accurate concentrations of CO_2 and humidity.

- Factory-calibrated with salt at 25 °C and 70 °C
- Achieves high humidity (<85%), even at high temperatures (<85 °C), without condensation
- Accurate humidity control with 0.1% RH stability over 6 hours
- Large 500 mL heated water reservoirs

Humidity and CO_2 Range

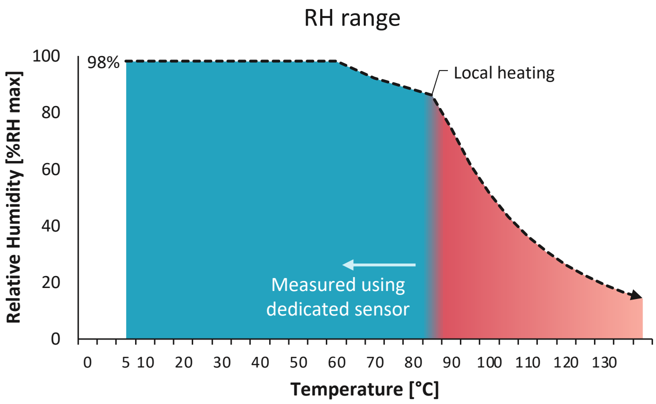


Figure 7: Accessible humidity as a function of temperature

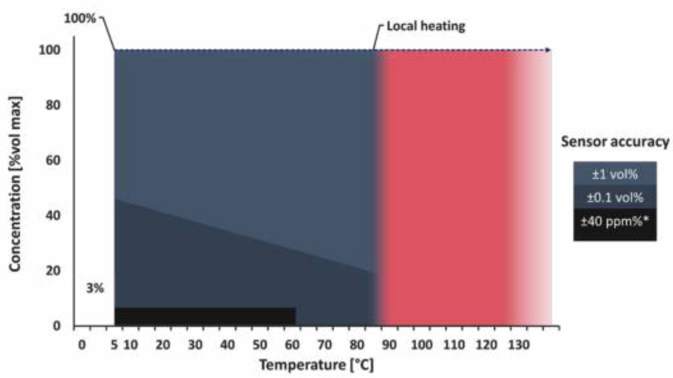


Figure 8: Accessible CO_2 concentration as a function of temperature
** Requires DVS Carbon Advanced*

Thanks to a precisely tuned temperature enclosure, heated water reservoirs, and calibrated sensors, accurate high relative humidity is achievable in the enclosure operating temperature range.

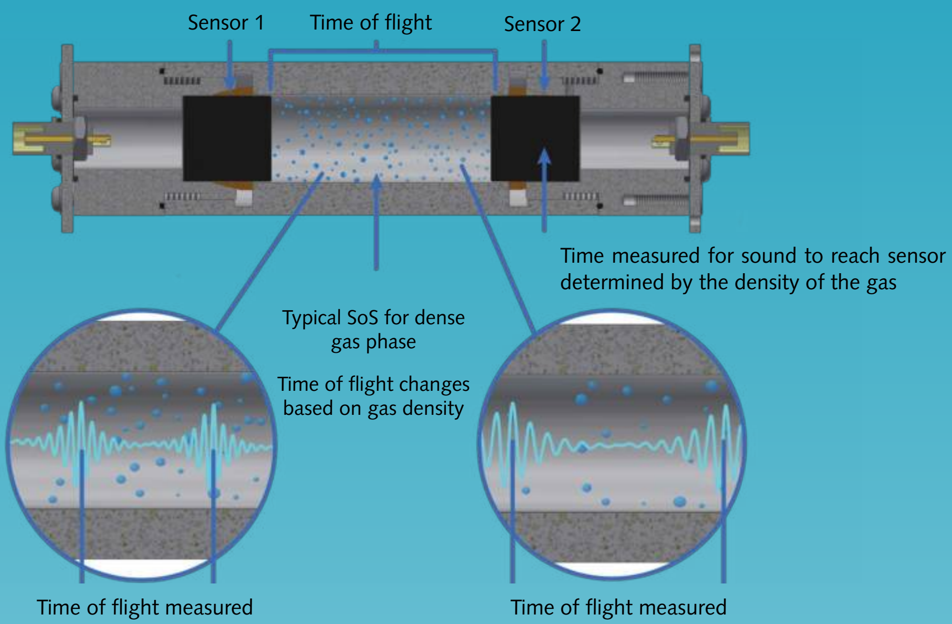
An extended range of reduced RH is accessible using local heating up to 300 °C.

Wide range of CO_2 concentrations generated by mixing pure CO_2 diluted CO_2 and an inert carrier gas.

Our patented SoS sensor affords accurate measurements with CO_2 concentrations up to 100 vol%, and an increased resolution below 50%. A non-dispersive infrared (NDIR) sensor can be installed for measuring CO_2 down to 50 ppm.

Speed of Sound Sensor

The speed of sound is an intrinsic property of gas or vapor measured in sorption experiments. It depends on gas/vapor temperature, concentration, and species. Surface Measurement Systems redesigned its patented Speed of Sound (SoS) Sensor to provide the most accurate real-time precision. Meaning this compact device enables controlled generation across a range of concentrations, without the need for large, complex machinery.



Comparing DVS Carbon Models

	DVS Carbon Standard	DVS Carbon Advanced	DVS Carbon[5]
Generate 0-100 vol% CO_2 by mixing pure CO_2 & carrier gas, measured by SMS patented Speed of Sound Sensor	✓	✓	✓
Humidity generation from 0-98 RH% across wide temperature range with heated water reservoirs	✓	✓	✓
Sample activation or high temperature sorption experiments up to 300 °C with local heater	✓	✓	✓
Seamless carrier gas switching, e.g. between nitrogen and 400 ppm of CO_2 in N_2	✓	✓	✓
Generation of CO_2 and H_2O mixtures in a carrier gas, in subject to limits of individual component concentration [vol%CO_2 + RH%] < 100	✓	✓	✓
Mixing capabilities for low concentrations of CO_2 from 50 ppm to 5000 ppm) **, including a second NDIR CO_2 sensor***		✓	
Independent dynamic control & mixing of CO_2 and H_2O to enable greatly extended CO_2/RH generation (e.g. increasing CO_2 concentration from 0 to 100% at a constant 50% RH)		✓	✓
High throughput with combination of five low or high mass Ultrabalances			✓

* Sensor accuracy is unspecified on carrier gases that contain less than 99% N_2 or air,
** Requires pre-calibrated CO_2 cylinder, *** 2nd NDIR sensor limits maximum temperature to 60°C when installed

Case Study: Post Combustion

Zeolite 13X is a sodium-exchanged aluminosilicate zeolite with a faujasite topology and an effective pore size of around 0.9-1 nm. It is often used in industrial gas separations, such as oxygen production, gas drying, or desulfurization. Due to its high uptake of CO_2, it was investigated for its capture in flue gas conditions. Unfortunately, its extremely high affinity for water means that CO_2 adsorption is inhibited in humid conditions, as H_2O competes and occupies the micropores.

The DVS Carbon was used to examine the uptakes of two components, and to evaluate the impact of humidity on CO_2 capture. An initial CO_2 isotherm shows a high uptake for carbon dioxide, of nearly 15 weight%.

To evaluate co-adsorption conditions, a second CO_2 isotherm is recorded with 5% RH of background relative humidity. The introduction of CO_2 induces a negligible increase in mass, highlighting that the sorption sites and pores are already occupied by water.

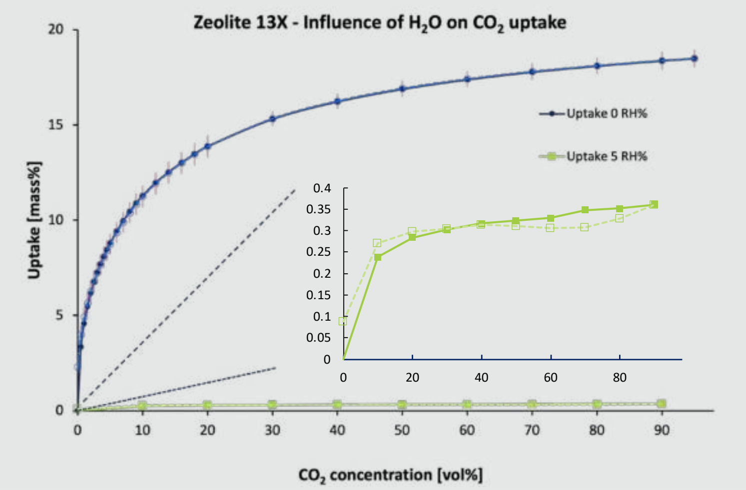


Figure 9: Impact of constant humidity (5 RH%) on CO_2 uptake at 25 °C on Zeolite 13X in a N_2 carrier flow. Inset shows zoom of isotherm on 5 RH%, assuming no displacement of water by CO_2

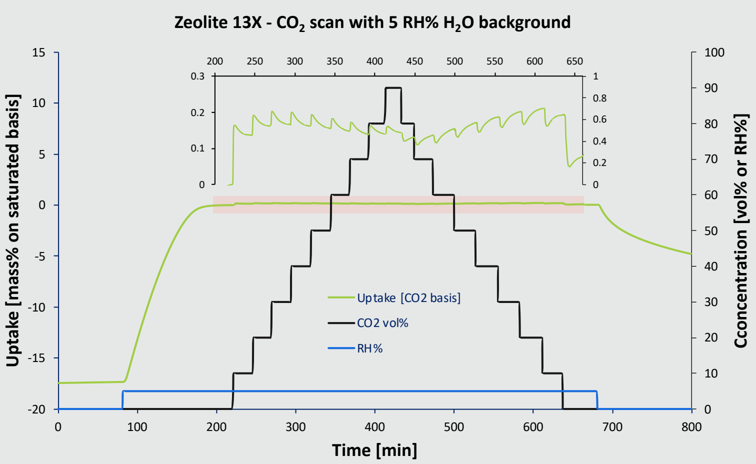


Figure 10: Kinetics of uptake when CO_2 is changed at a constant 5 RH% on Zeolite 13X at 45°C. Inset is a zoom of mass change during CO_2 scanning

Observing the kinetics in detail, a small displacement effect can be observed. Nevertheless, as has been also proven by breakthrough studies, the uptake for CO_2 decreases by a factor of 10. An interesting observation is that the kinetics of water sorption are slow compared to CO_2. This could allow kinetic-driven, rather than equilibrium-driven separation processes with Zeolite 13X.

Case Study: Direct Air Capture

Direct air capture (DAC) is an ambitious initiative to capture CO_2 from the ambient air. This endeavour is challenging due to the low concentration of carbon dioxide in the atmosphere, around 400 ppm. Amines are promising for this application due to their high affinity for carbon dioxide. Functionalizing amine groups or impregnating amines on various porous supports helps to mitigate issues such as accessibility, degradation, and vaporization. The influence of water is particularly important, as its presence can reduce or even enhance the capacity for CO_2.

Uptakes on two different types of supported amines have been evaluated. In one case, humidity was changed in the presence of low concentrations of CO_2 to study total uptakes. In another, steps of each component were introduced, to study in-depth the kinetics of adsorption in the presence of individual and combined components.

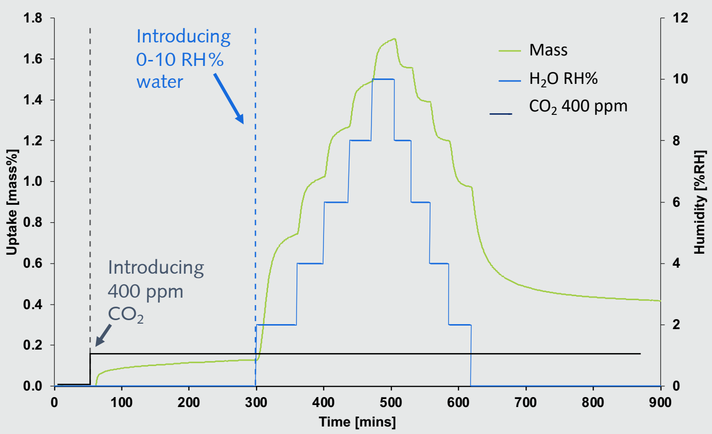


Figure 11: Evaluating total uptake in the presence of humidity and CO_2 on an amine functionalized sorbent

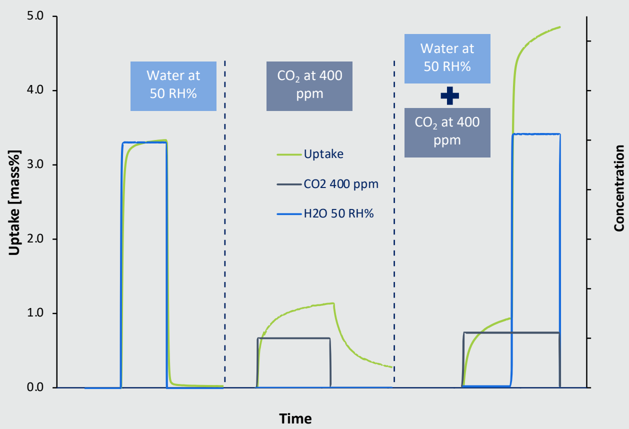
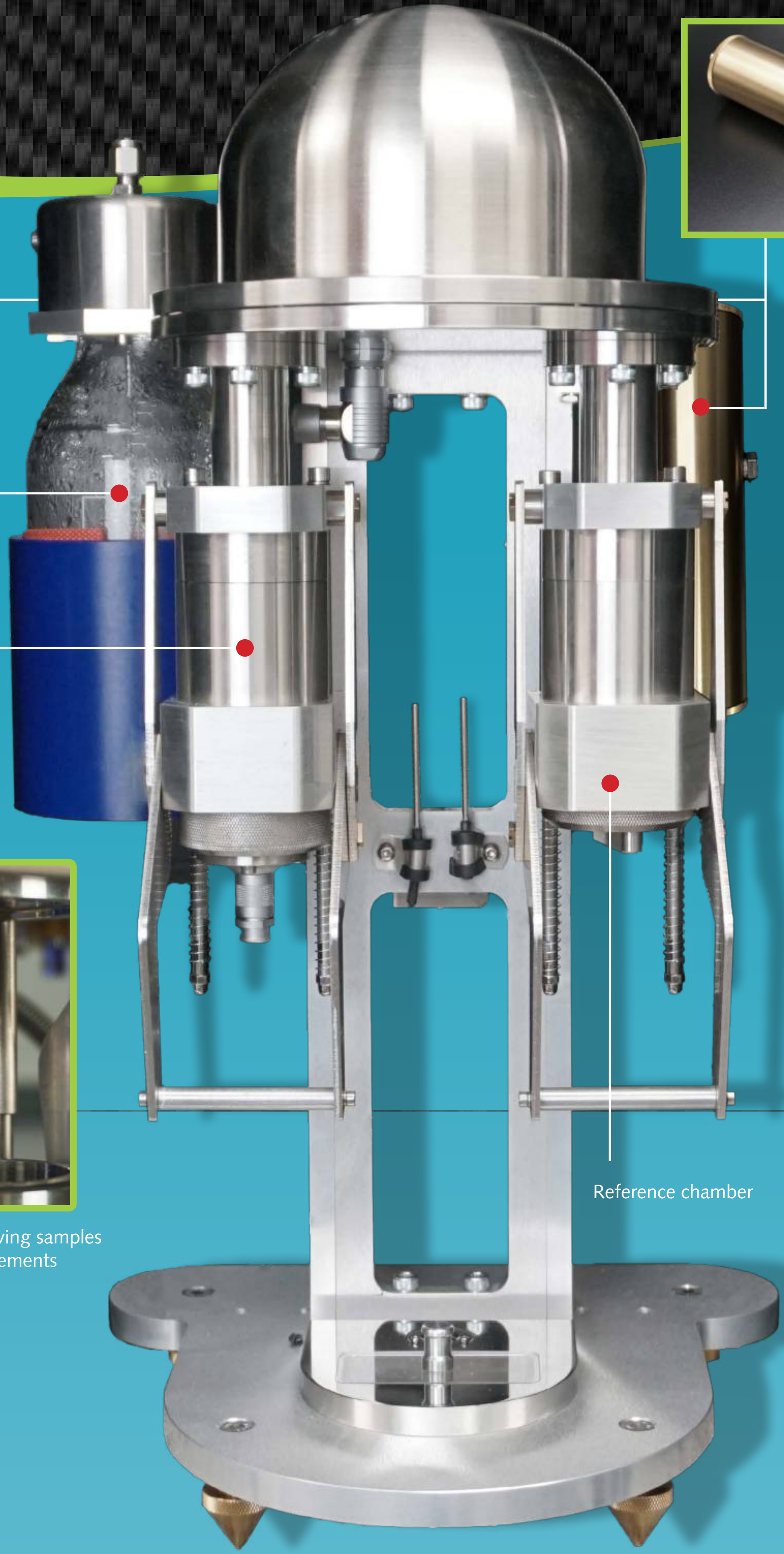


Figure 12: Sequential experiments evaluating kinetics of adsorption during step changes in humidity, 400 ppm CO_2 and both components

The DVS Carbon allows straightforward control of all important variables: CO_2 concentration, humidity, and temperature on its standard, advanced, and high throughput models.

Modular Capabilities



Speed of Sound Sensor

Speed of Sound Sensor

The latest iteration of the SoS sensor is specially developed to enable control and precision in the generation and measurement of CO_2 concentrations, from high (100%) to low (0.5%) at 25 °C.

Heated Reservoir

- The heated reservoir is located in the temperature enclosure
- Designed for extended humidity generation up to 85°C, with fully automated temperature control
- Features a heated jacket to avoid evaporative cooling

High Temperature Preheater

(For drying and regeneration)

- *In-situ* degassing/activation of samples up to 300 °C
- The temperature is measured by a Pt-100 directly below the sample pan
- User programmable and controlled temperature ramps or steps
- Up to 5 x preheater available for the DVS Carbon[5] high throughput option

Raman probe observing samples during measurements

Optional:

Raman Spectroscopy

- Fully integrated hardware/software solution for triggering and capturing Raman spectra during sorption experiments

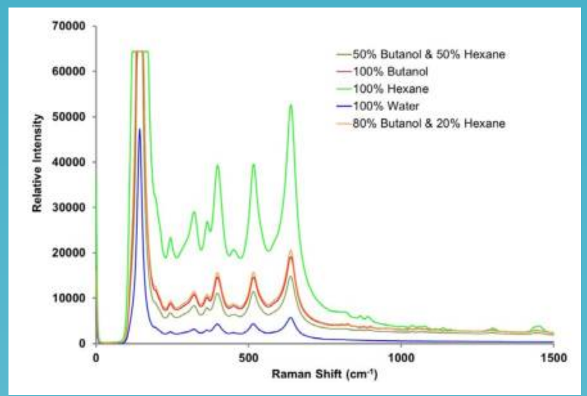


Figure 13. Raman spectra of single and dual-solvents sorption of butanol and hexane for a titanium oxide sample at 25 °C

Microscopy and Video

- 1.3 megapixel color camera and up to 200x optical zoom

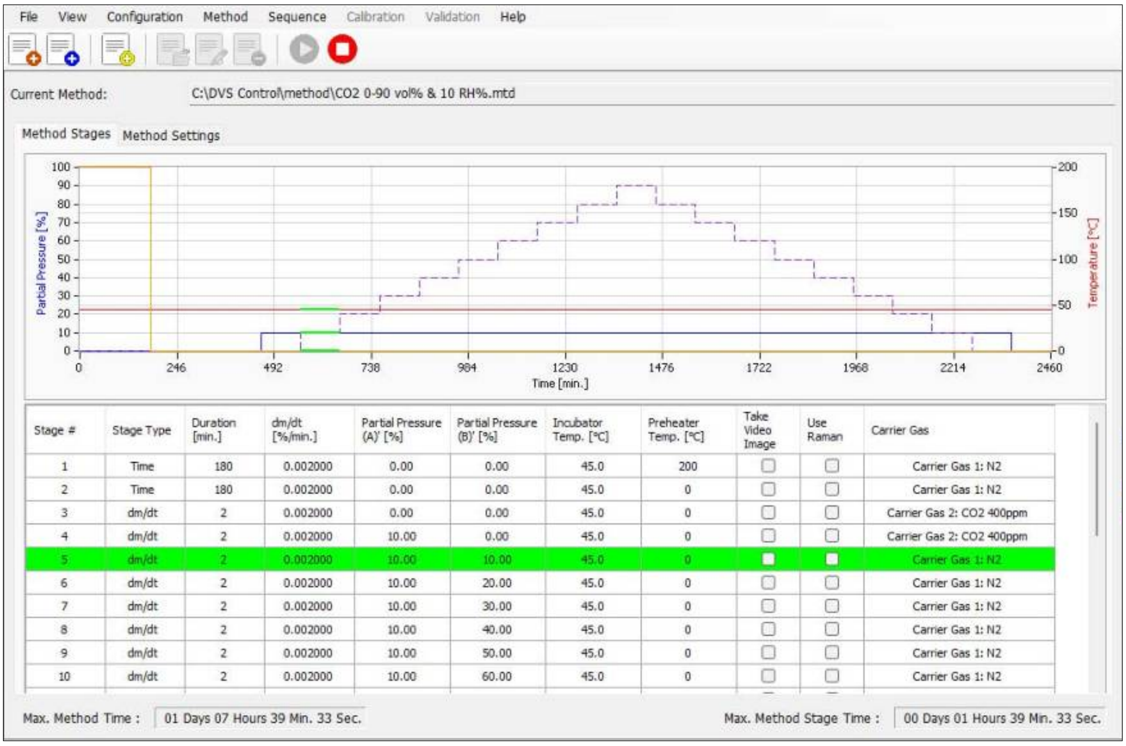
Figure 14. In-situ monolith support

Important: Camera and Raman cannot be used while the Preheater is mounted to the chamber

Purpose-built Software

The software package provided with the DVS Carbon allows the users to create and customize experimental methods while enabling the full analysis of the kinetic data collected. Examples of the control and analysis software used in two sorption experiments are outlined below.

Control Software



Stage #	Stage Type	Duration [min.]	dm/dt [%/min.]	Partial Pressure (A)' [%]	Partial Pressure (B)' [%]	Incubator Temp. [°C]	Preheater Temp. [°C]	Take Video Image	Use Raman	Carrier Gas
1	Time	180	0.002000	0.00	0.00	45.0	200			Carrier Gas 1: N2
2	Time	180	0.002000	0.00	0.00	45.0	0			Carrier Gas 1: N2
3	dm/dt	2	0.002000	0.00	0.00	45.0	0			Carrier Gas 2: CO2 400ppm
4	dm/dt	2	0.002000	10.00	0.00	45.0	0			Carrier Gas 2: CO2 400ppm
5	dm/dt	2	0.002000	10.00	10.00	45.0	0			Carrier Gas 1: N2
6	dm/dt	2	0.002000	10.00	20.00	45.0	0			Carrier Gas 1: N2
7	dm/dt	2	0.002000	10.00	30.00	45.0	0			Carrier Gas 1: N2
8	dm/dt	2	0.002000	10.00	40.00	45.0	0			Carrier Gas 1: N2
9	dm/dt	2	0.002000	10.00	50.00	45.0	0			Carrier Gas 1: N2
10	dm/dt	2	0.002000	10.00	60.00	45.0	0			Carrier Gas 1: N2

Figure 15. The above graph shows the method panel within the method manager. It displays numerically and graphically the current method for a water and CO_2 sorption experiment at 25 °C. The active stage of the ongoing experiment is highlighted in green. Figures 16 and 17 (below) are typical data generated by this method.

Analysis Software

Sorption Data

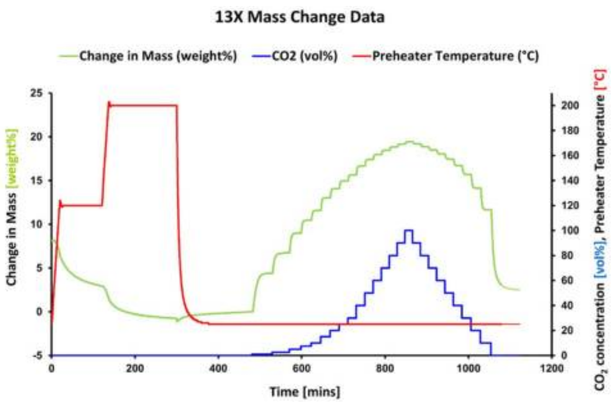


Figure 16. Sample activation and CO_2 sorption kinetics of zeolite 13X at 25 °C

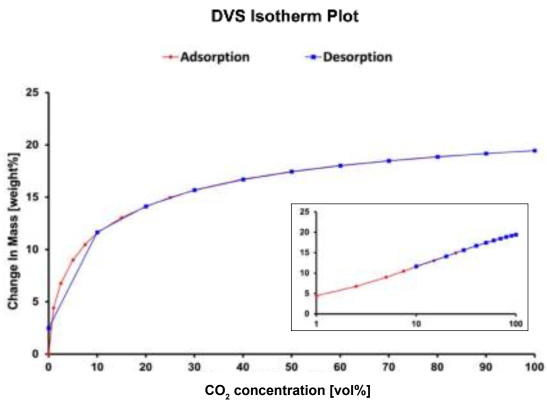


Figure 17. CO_2 sorption isotherm of zeolite 13X at 25 °C

Specifications

Construction Materials

Custom built manifold: 316 stainless steel
Seals: Viton® and Kalrez® or equivalent
Tubing: 1/4 inch 316 stainless steel

Gas Flow Control

High accuracy digital mass flow controllers
Wide dynamic range - turndown ratio 1000:1
Carrier gas: dry air, nitrogen, CO_2 (<1%) in nitrogen
Adjustable flow rates

Temperature Control

Temperature Controlled Enclosure
Contains entire generation and measurement system
Control range: 5 °C to 85 °C [10]
Temperature stability ± 0.05 °C over 6 hours
Temperature resolution 0.01 °C

Local Sample Heater
Control range: up to 300 °C
Heating ramp rates: up to 10 °C/min

Humidity Generation & Measurement

Water Reservoirs
500 mL easy-change reservoir
Heated to avoid evaporative cooling
Generation
Relative humidity range [10]
- 0 to 98% RH for 5-60 °C [1]
- 0 to 85% RH for 60-85 °C [1]

Relative humidity generation performance
- Accuracy: ±0.5% RH [2]
- Resolution: ±0.1% RH
- Stability: ±0.1% RH over 6 hours

Measurement
Relative humidity continuous measurement
- Range 0-100%RH

Relative humidity measurement accuracy
- ±0.8% RH at 5-40 °C
- ±1.5% RH at 40-85 °C

CO_2 Generation and Measurement

Generation
Concentration range 0-100%vol, atmospheric pressure
CO_2 generation performance
- Accuracy: exact [3] or 0.5%vol of inlet concentration [4]

Measurement
Patented Speed of Sound Sensor
- Range: 0-100%vol [5]
- Accuracy: 0.1%vol up to 40%vol, 1% above

In line NDIR ppm sensor [6,7]
- Range 0-3%vol
- Accuracy: 40 ppm [5]

Weight Measurement

Ultrabalance Low Mass	Ultrabalance High Mass
Maximum load: 1000 mg	Maximum load: 5000 mg
Mass change: ±150 mg	Mass change: ±1000 mg
Resolution: 0.01 µg	Resolution: 0.1 µg
Balance noise: ≤ 0.3 µg [8]	Balance noise: ≤ 3 µg [8]

Additional Note: Ability to combine five low and high mass balances exclusive to the DVS Carbon[5] high throughput mode

Hardware Configuration

Standard
Mixing of two gas streams, with controlled humidity and CO_2 concentration, respectively [9]
Switching between carrier gases e.g. Nitrogen and 400 ppm CO_2
Advanced
Two heated reservoirs for enhanced humidity generation at high CO_2 levels (up to 98% humidity in a pure CO_2 stream) [9]
Mixing and direct measurement of low CO_2 concentrations (ppm-level) through dedicated instrumentation [9]
High Throughput
Five chambers for simultaneous measurement for five samples

System Information

Dimensions: 520 mm (W) x 980 mm (H) x 610 mm (D)
Weight: 80 kg (180 lb) [10]
Electrical: 200-240 V, 50/60 Hz, 1500 VA

System Software

DVS Control Software
- Live data view and plotting
- Full control over all parameters
- Powerful custom methods and sequences
- Complex isotherm or isobar experiments
- Temperature changes in a single experiment
- Ramp or step methods available
- Intelligent (dm/dt) or time-based step equilibration criteria
- Multiple concentration or temperature cycles
- Automated video image or Raman spectra acquisition

DVS Analysis Software
- Automated isotherm calculation
- Full kinetics information during experiment
- Isotherm modeling
- Enthalpy (heat) of adsorption
- Permeability and diffusion modeling
- Surface area models

Software Options:

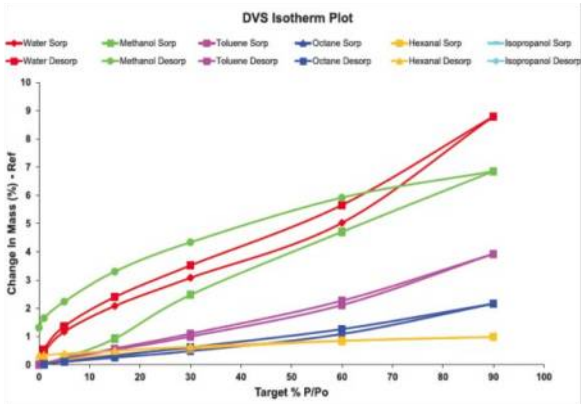
Standard (Included)
- Control software
- Standard analysis

Advanced (Optional)
- Advanced analysis suite
- Isotherm analysis suite

Footnotes
[1] Humidity factory calibrated at 25 °C and 70 °C. Calibrations at other temperatures upon request
[2] 1-σ confidence level with % RH based on SMS factory certified methods (Salt Calibration)
[3] When using pure CO_2 or a calibrated cylinder
[4] When mixing
[5] At 25 °C
[6] Only in advanced configuration
[7] Using ppm probe reduces maximum operating T to 60 °C
[8] Root mean square (averaged over 24 hours)
[9] Some measurement restrictions apply
[10] Specs vary for DVS Carbon[5] high throughput model. Contact your local rep for full specs.

What is Dynamic Vapor Sorption?

Invented by Professor Daryl Williams, Founder & MD of Surface Measurement Systems, Dynamic Vapor Sorption (DVS) is a gravimetric sorption technique that measures how quickly and how much of a vapor or gas is sorbed by a sample. It does this by varying the vapor/gas concentration around the sample and measuring the resultant change in mass. DVS is a valued tool in R&D laboratories all over the world, from pharmaceutical, food, personal care and health to energy, aerospace, agriculture, environmental, and building material industries.



About Surface Measurement Systems

Surface Measurement Systems Ltd. develops and engineers innovative experimental techniques and instrumentation for physico-chemical characterization of complex solids, solving difficult problems in materials research. With over 30 years of continuous innovation, every instrument is built with the cumulative knowledge and experience of our world-leading team of sorption scientists. This makes us the preferred sorption partner of universities, research institutes, corporate R&D, and global government organizations, who we work with and support in pioneering the future of materials research.

Surface Measurement Systems
World Leader in Sorption Science

DVS
DISCOVERY

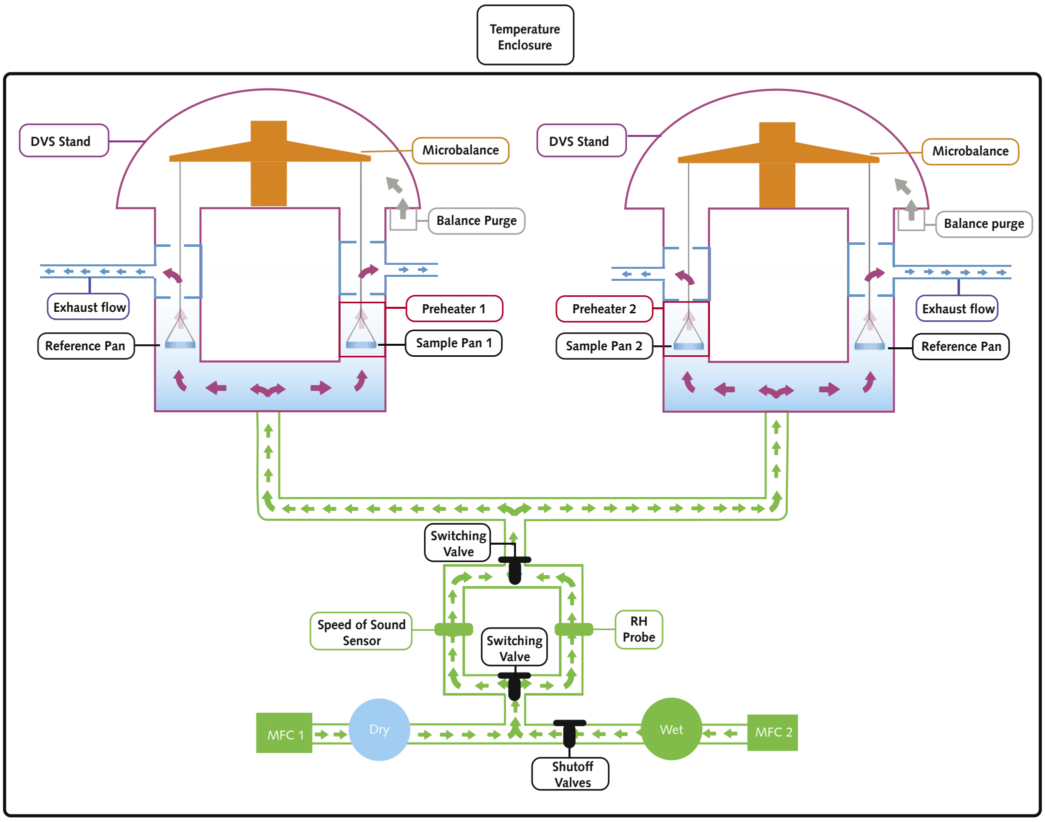
The World's Most Advanced Dual Balance Gravimetric Vapor Sorption Instrument

- Organic and water vapor sorption kinetics and isotherm from 10 to 70 °C
- Dual balance design for simultaneous mass measurements
- Real time partial pressure measurement and control
- *In-situ* sample drying/activation
- Color video microscopy/fiber optic probe spectroscopy
- True0™ drying at 0.0% RH

www.surfacemeasurementsystems.com

DVS Discovery Dual Balance Vapor Gravi

Dynamic Vapor Sorption (DVS) is a dual balance gravimetric sorption technique that measures the rate and amount of solvent sorbed and released by two samples simultaneously, such as a dry powder absorbing or releasing water. The DVS accomplishes this by varying the vapor concentration surrounding the sample and measuring the change in mass which this produces.



DVS Discovery Organic Schematic

Hardware

- The only system able to measure organic vapor partial pressure directly using the Speed of Sound Sensor (*Patent WO 2018/002612 A1*)
- Open stainless steel stand design enabling easy access to sample pan while minimizing static electric charging
- Accurate and uniform temperature across a broad temperature range (from 10 to 70 °C)
- Optional IR, Raman and video imaging with integrated control software
- Quick and easy to change bottle

Applications

- Hygroscopicity of pharmaceutical solids
- T_g and RH phase transitions in polymers
- Amorphous content determination of solids
- Diffusion and permeation in polymers
- Food, flavors and fragrances
- Sorbents
- Wood and cellulosic materials
- Composites
- Hydrophillic and hydrophobic materials

Software - the software package provided with the DVS Discovery allows the users to create and customize experimental methods while enabling the full analysis of the data collected. Examples of the control and analysis software used in a standard water sorption experiment are outlined below.

•Control Software

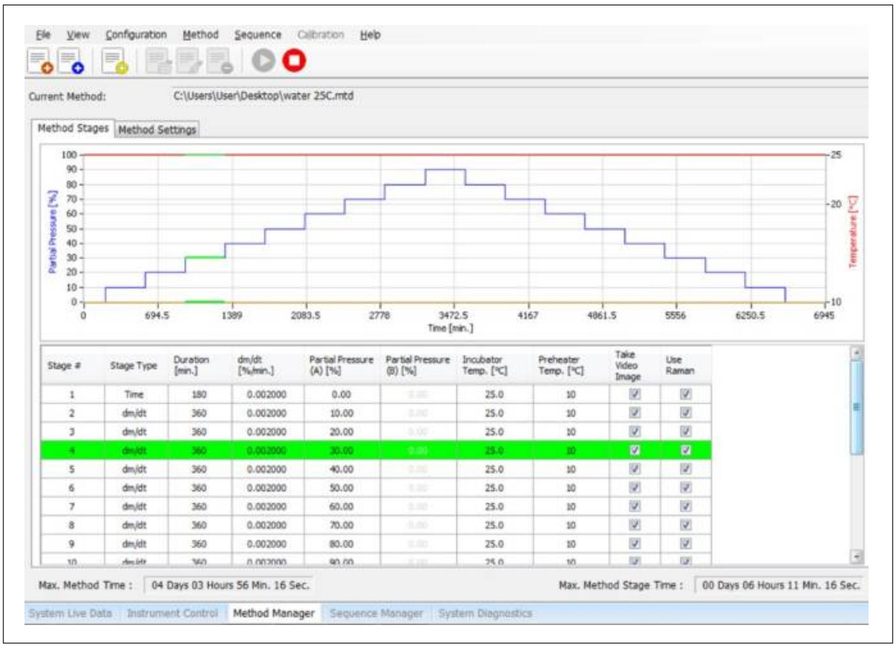


Figure 1. The above graph shows the method panel within the method manager. It displays numerically and graphically the current method for a water sorption experiment at 25 °C. The active stage of the ongoing experiment is highlighted in green. Figures 2 and 3 (below) are typical data generated by this method.

•Analysis Software

Water Sorption Data

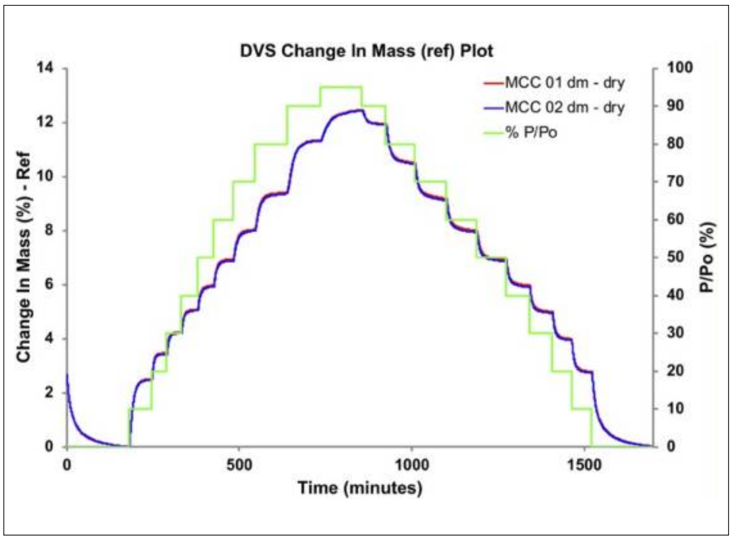


Figure 2. Water sorption kinetics of two samples of microcrystalline cellulose (MCC) at 25 °C

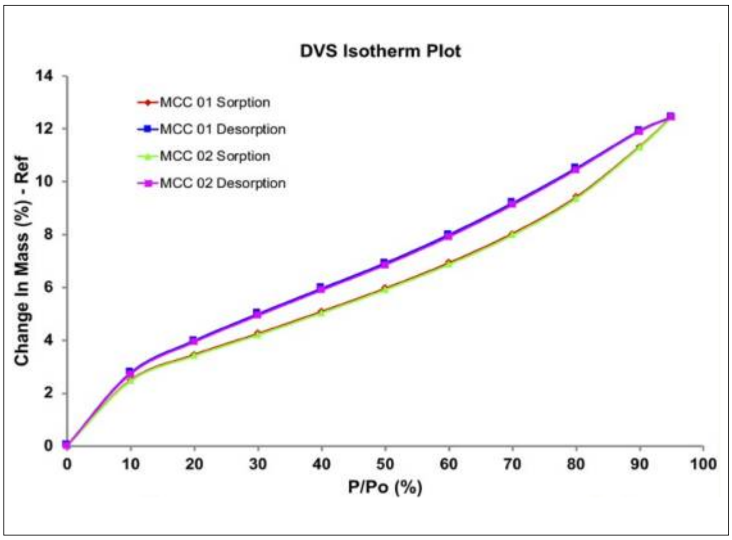


Figure 3. Water sorption isotherms of two samples of microcrystalline cellulose (MCC) at 25 °C

Outstanding Performance

The DVS Discovery allows for the collection of high quality data, owing to the outstanding Ultrabalanace performance (Figure 4), precise vapor generation (Figure 5) and accurate temperature control (Figures 4, 6, 7).

Mass and Temperature Measurement

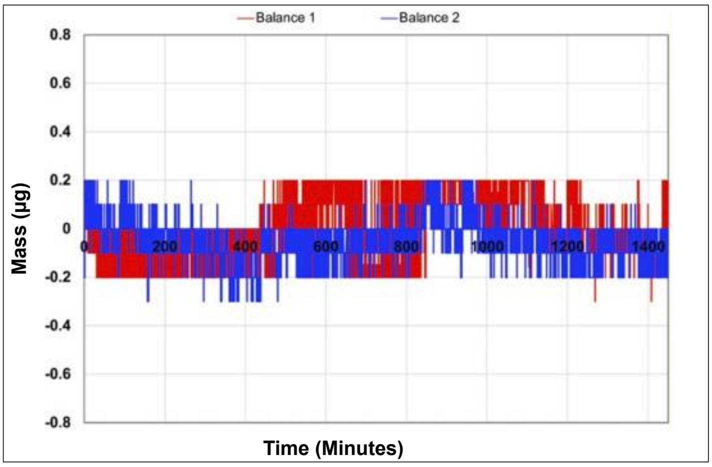


Figure 4. DVS Mass Baseline Stability Plot Over 24 Hours

- Mass changes at a resolution of 0.01 µg for low mass balance
- Root mean square noise of ≤ 0.3 µg for low mass balance (averaged over 24 hours)

True0™ RH

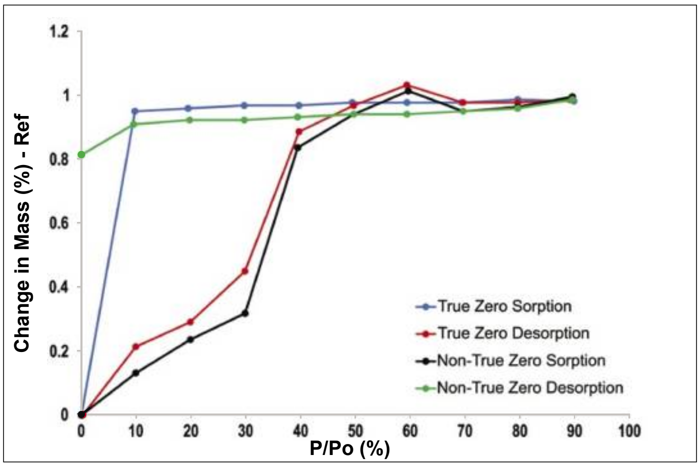


Figure 5. Comparison of Naloxone Hydrochloride Dihydrate Water Sorption Isotherms

- Only DVS instrument offering True0™ RH
- Achieves partial pressures of water as low as 0.0% RH
- Hydration and dehydration kinetics below 1% RH can be readily studied

Temperature Control & Stability

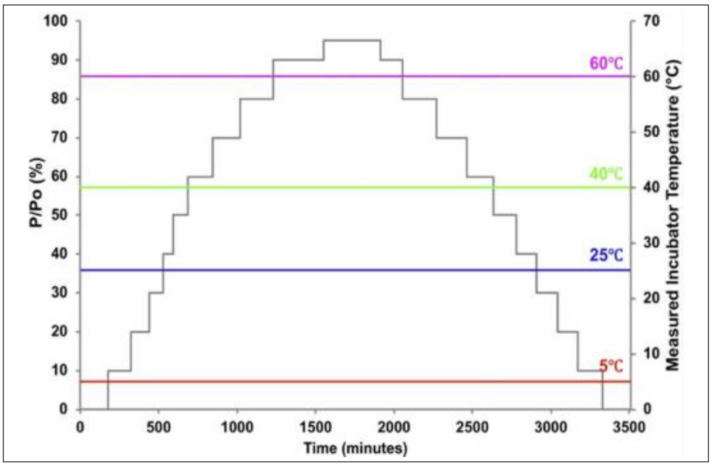


Figure 6. Partial pressure plots of Microcrystalline cellulose (MCC) at different temperatures

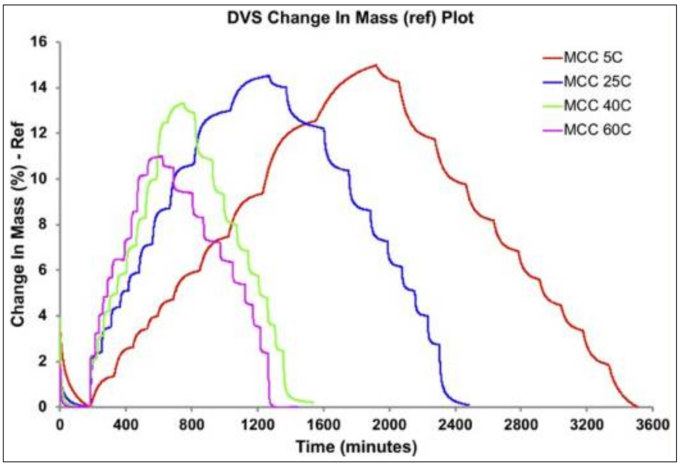


Figure 7. Sorption kinetic plots of Microcrystalline cellulose (MCC) at different temperatures

- Stability at 25 °C is ± 0.05 °C over 6 hours
- Vapor generation and delivery at sample temperature prevents condensation issues typically found in instruments with multiple temperature zones
- Allows for accurate and stable isotherm experiments

Solvent Delivery Configurations

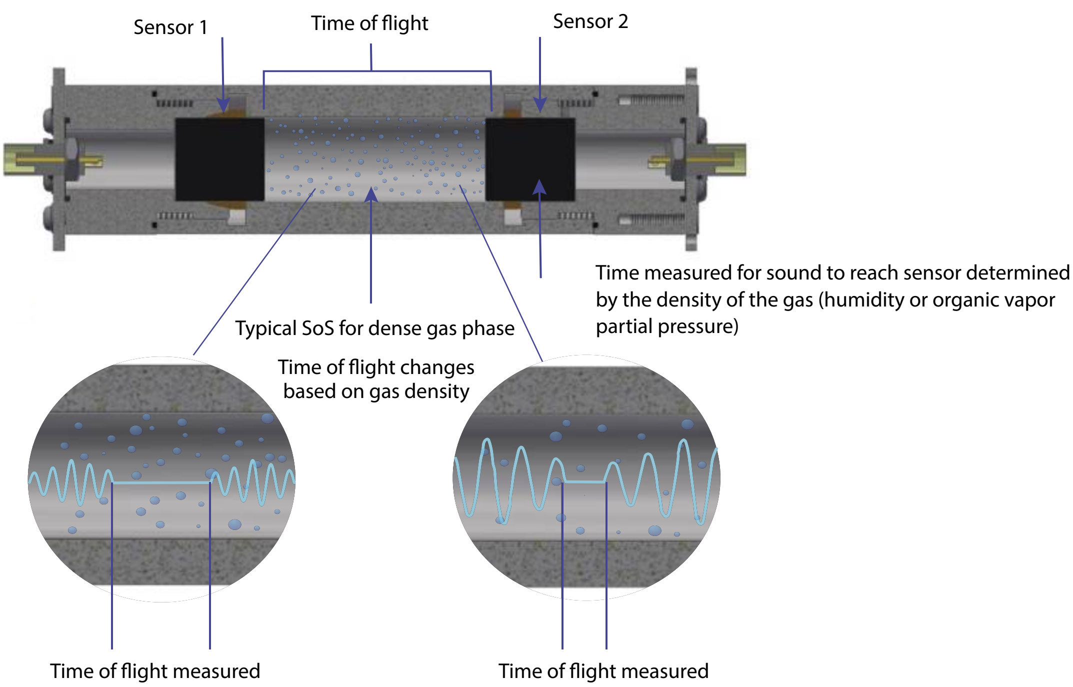
The **DVS Discovery** can precisely deliver combinations of:
(1) Humidity with controlled generation using a humidity sensor

(2) Organic vapors with controlled generation using a proprietary **Speed of Sound (SoS)** sensor.

The DVS Discovery can be provided in two different factory configurations:
(1) **AQUA**: provided with a humidity sensor to allow the controlled generation of water.
(2) **Organic**: provided with a humidity sensor and one SoS sensor, to allow the alternative controlled generation of water or organic solvent.

Speed of Sound Sensor

Speed of sound is an intrinsic property of the vapor or gas measured, and depends on the temperature, gas/vapor concentration and gas/vapor species. The **Speed of Sound Sensor*** in the **DVS Discovery** is the only method to directly measure the vapor concentration ultrasonically. The SoS sensor determines the gas/vapor concentration in real time based on the speed of sound travelling through a fixed volume of solvent gas/vapor.



Key features of SoS Sensor:

- SoS based concentration measurements enable active closed loop control, which is significantly more accurate than open loop control
- Resolution of typically ±0.1% P/P_0 of target P/P_0

Partial Pressure Control

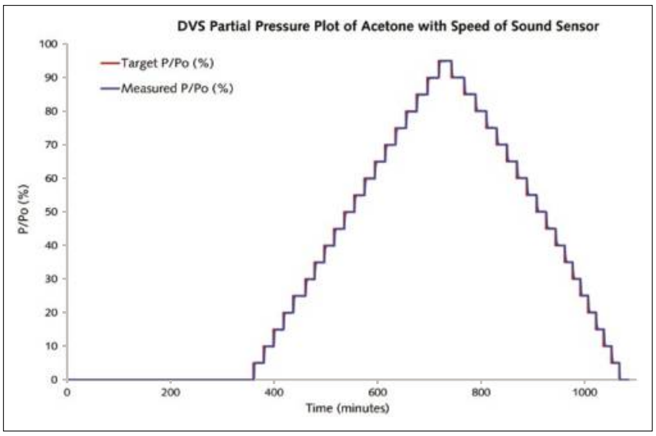


Figure 8. Acetone partial pressure Kinetic measured using the SoS Sensor during a DVS experiment

*Patent WO 2018/002612 AI

Applications

The DVS Discovery also allows the user to perform complex and advanced material analysis such as BET Surface Area (Figures 9 & 10), Amorphous Content (Figure 11), and Diffusion Measurement (Figure 12).

Typical Organic Vapor Sorption Data (Single Solvent)

Organic Solvent Sorption Experiment

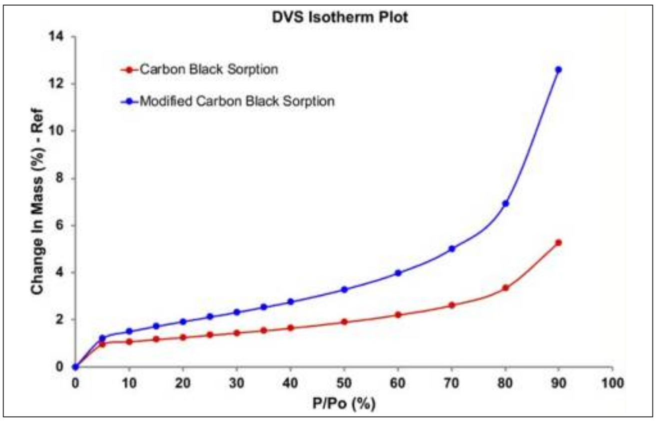


Figure 9. Octane Isotherm Plots

- Octane isotherm plots for Carbon Black (Red) and modified Carbon Black (Blue) at 25 °C

Specific Surface Area Calculations

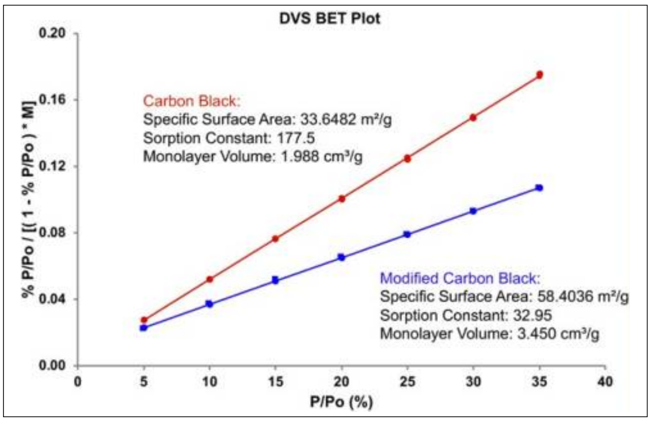


Figure 10. BET Linear Plots

- Octane BET linearized plot for surface area determination at 25 °C for as-received (Red) and modified (Blue) Carbon Black samples

Amorphous Content

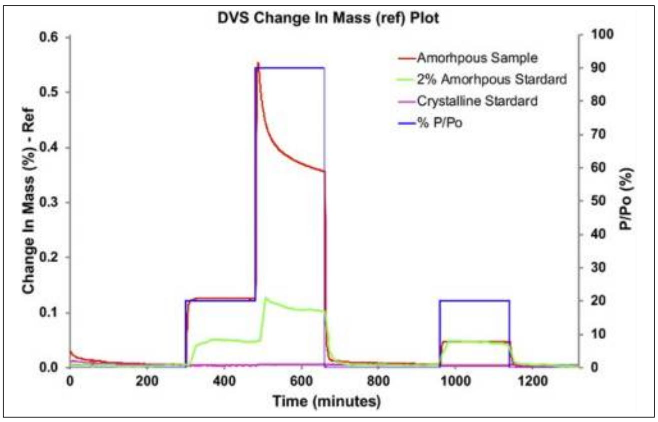


Figure 11. Amorphous content of Fluticasone Propionate

- Pre and post crystallization uptake comparison between Amorphous Sample (Red), 2% Amorphous Standard (Green) and Crystalline Standard (Pink).

Diffusion Measurement

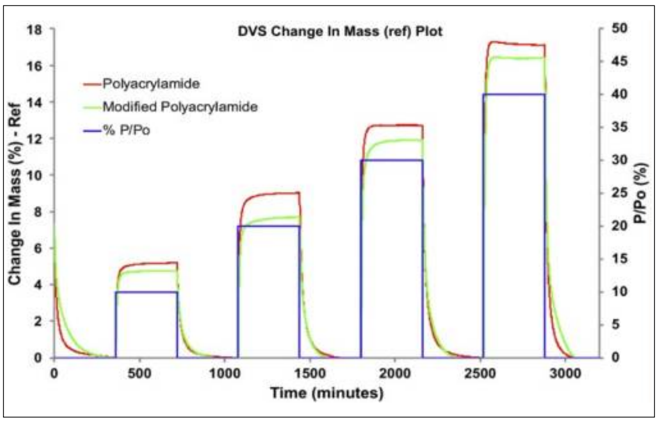


Figure 12. Kinetic plots of as-received and modified polyacrylamide samples at 25 °C

- Comparative water sorption kinetics of as-received (Red) and modified (Green) Polyacrylamide for diffusion calculation.

Modular Capabilities

Speed of Sound Sensor

Raman Spectroscopy

- Fully integrated hardware/software solution for triggering and capturing Raman spectra during sorption experiments
- Simultaneous operation of Raman and optical microscopy during the DVS experiment
- Allows for a more complete understanding of vapor-solid interactions for materials

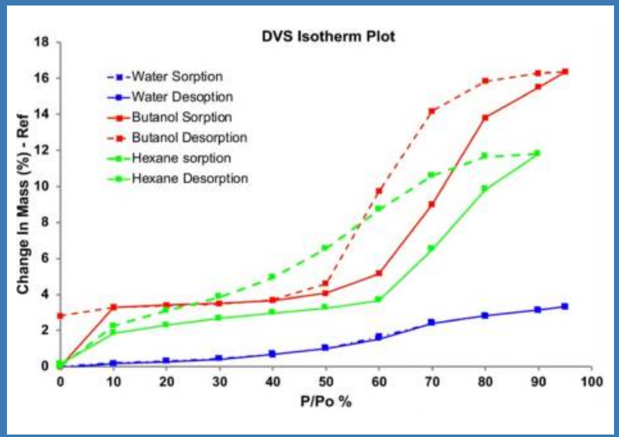


Figure 13. Isotherm plots of water, butanol and hexane sorption for titanuim oxide at 25 °C

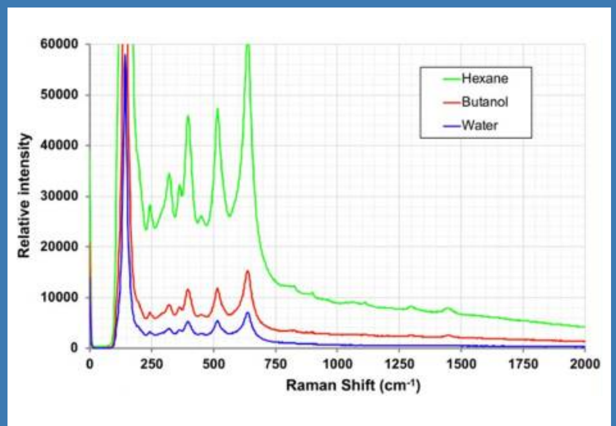


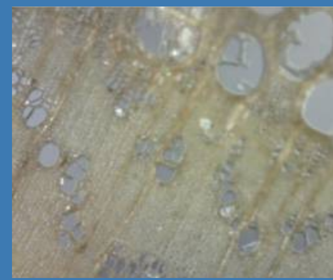
Figure 14. Raman Spectra of water, butanol and hexane sorption for titanuim oxide at 25 °C

Heated Reservoir Accessory*

- The heated reservoir replaces the standard glass bottle mounted on the left of the stand
- Designated for extended humidity generation 85% RH at 70 °C, with fully automated temperature control

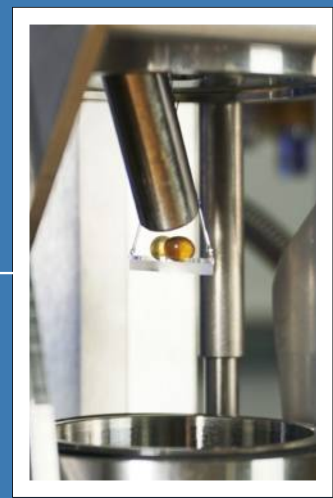
Microscopy and Video

- 1.3 megapixel color camera
- Up to 200x optical zoom
- Images have time-date-temperature-partial pressure stamps
- Grid overlay and calibration for measuring dimensional change
- The images can be composed into a timelapsed video



Cedar wood

Cellulose



Agricultural seeds

High Temperature Preheaters*

(for drying and curing)

- *In-situ* degassing/activation of samples up to 200 °C
- The temperature is measured by a Pt-100 directly below the sample pan
- User programmable and controlled temperature ramps or steps

Important: Camera and Raman cannot be used while the Preheater is mounted to the same chamber

Surface Measurement Systems
World Leader in Sorption Science

*For more information on the specification of these accessories, please contact sales@surfacemeasurementsystems.com

Technical Specifications

Temperature

Temperature controlled enclosure

Control range: 10 °C to 70 °C

Temperature stability ± 0.05 °C over 6 hours

Temperature resolution 0.01 °C

High Temperature Pre-heater for drying up to 2 samples

200 °C (maximum local temperature)

Heating ramp rates: up to 5 °C/min

Temperature sensor: Pt-100

Discovery Stand

Manifold: 316 stainless steel

Seals: Viton® and Kalrez® or equivalent

Tubing: 1/4 inch stainless steel

Solvent Reservoir

1 glass reservoir as standard

Optional heated reservoir

Flow Control

High accuracy digital mass flow controllers

Wide dynamic range - turndown ratio 1000:1

Carrier Gas - Dry air or Nitrogen

Relative Humidity

Relative humidity range from 0 to 98% for 10 - 45 °C [2]

Relative humidity range from 0 to 85% for 45 - 70 °C [1,2]

Relative humidity resolution ± 0.1%

Relative humidity stability ± 0.1% over 6 hours

RH range accuracy from 10 - 45 °C ± 0.5%[3]

RH range accuracy from 45 - 70 °C ± 1%[3]

Organic Vapor Generation and Measurement using Speed of Sound Sensor [4]

Partial pressure range from 0 to 90% P/P_o [5]

P/P_o resolution ± 0.1%

P/P_o accuracy from 10 - 70 °C ±1% P/P_o [3,6]

For full details of the Organic Vapors Generation capability, please contact us directly.

Mass Measurement

Ultrabalance Low Mass

Maximum load: 1000 mg

Mass change: ±150 mg

Resolution: 0.01 µg

Balance noise: ≤ 0.3 µg [7]

Ultrabalance High Mass

Maximum load: 5000 mg

Mass change: ±1000 mg

Resolution: 0.1 µg

Balance noise: ≤ 3 µg [7]

System Information

Dimensions: 520 mm (W) x 980 mm (H) x 610 mm (D)

20.4" (W) x 38.58" (H) x 24.01" (D)

Weight: 80 kg (180 lb)

Electrical: 200-240 V, 50/60 Hz, 1500 VA

Hardware Configuration

AQUA: 1 humidity sensor

Organic: 1 humidity sensor plus 1 SoS sensor, (1 reservoir)

System Software

DVS Control Software

- Sample pre-heating
- Vapor sorption
- Temperature changes in a single experiment
- Ramp or step changes in relative humidity
- Automated video image and IR/Raman spectra acquisition
- Complex isotherm experiments
- Organic vapor sorption partial pressure
- Experimental stages may be based on fixed-time or a user-defined dm/dt criteria
- Experiments may include half, full or multiple partial pressure or temperature cycles
- Windows™ 10

DVS Analysis Software

- Isotherms
- Permeability and diffusion
- Kinetics information
- Surface area models
- Amorphous content
- Heat of sorption
- T_g determinations

Software Options

Standard

- Control Software
- Standard Analysis

Advanced

- Advanced Analysis Suite
- Isotherm Analysis Suite

21CFR Part 11 software solution (optional)

Footnotes

[1] Optional configuration (heated reservoir) for long term high temperature generation. SMS recommends to use heated reservoir at all experimental analysis over 45 °C.

[2] System factory calibrated at 25 °C. Calibrations at other temperatures upon request.

[3] SD (Standard Deviation) with % RH or P/P_o calibration performance based on SMS factory certified methods (Salt Calibration or others)

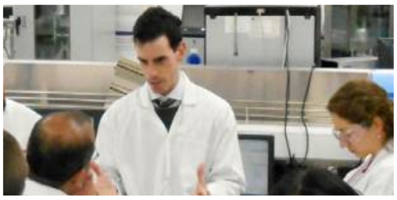
[4] For an update of the list of solvent offered, please contact us directly

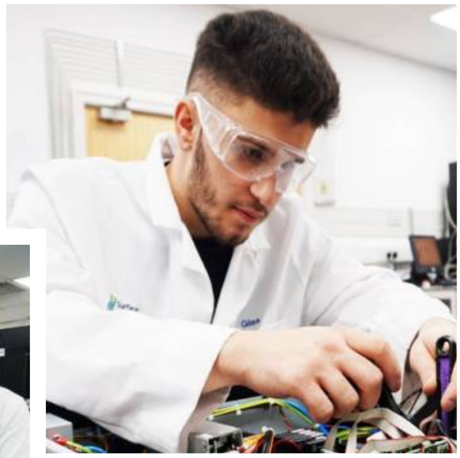
[5] Depending on the solvent selected and experimental temperature

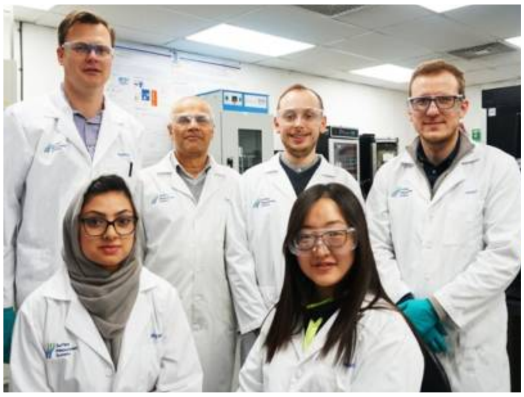
[6] Depending on the solvent selected

[7] Root mean square (averaged over 24 hours)

About Us







Surface Measurement Systems Ltd. develops and engineers innovative experimental techniques and instrumentation for physico-chemical characterization of complex solids. Our range of characterization instruments and scientific/engineering techniques has helped solve difficult problems in the pharmaceutical, biomaterial, polymer, catalyst, chemical, cosmetic and food industries, and are used by hundreds of leading laboratories and universities throughout the world.

Why us?

- Invented the DVS Technology with over 25 years of continuous innovation
- Every instrument is built upon the knowledge and experience of our industry leading sorption scientists
- Our service team provides uncompromising support to our customers and partners
- Outstanding instrument performance
- Most complete and intuitive Windows™ software for experimental control and analysis
- Winner of EEF Innovation Award 2019 and ISO 9001:2015 accredited

Surface Measurement Systems
World Leader in Sorption Science

ISO 9001:2015 REGISTERED COMPANY

WINNER Innovation Award FUTURE awards

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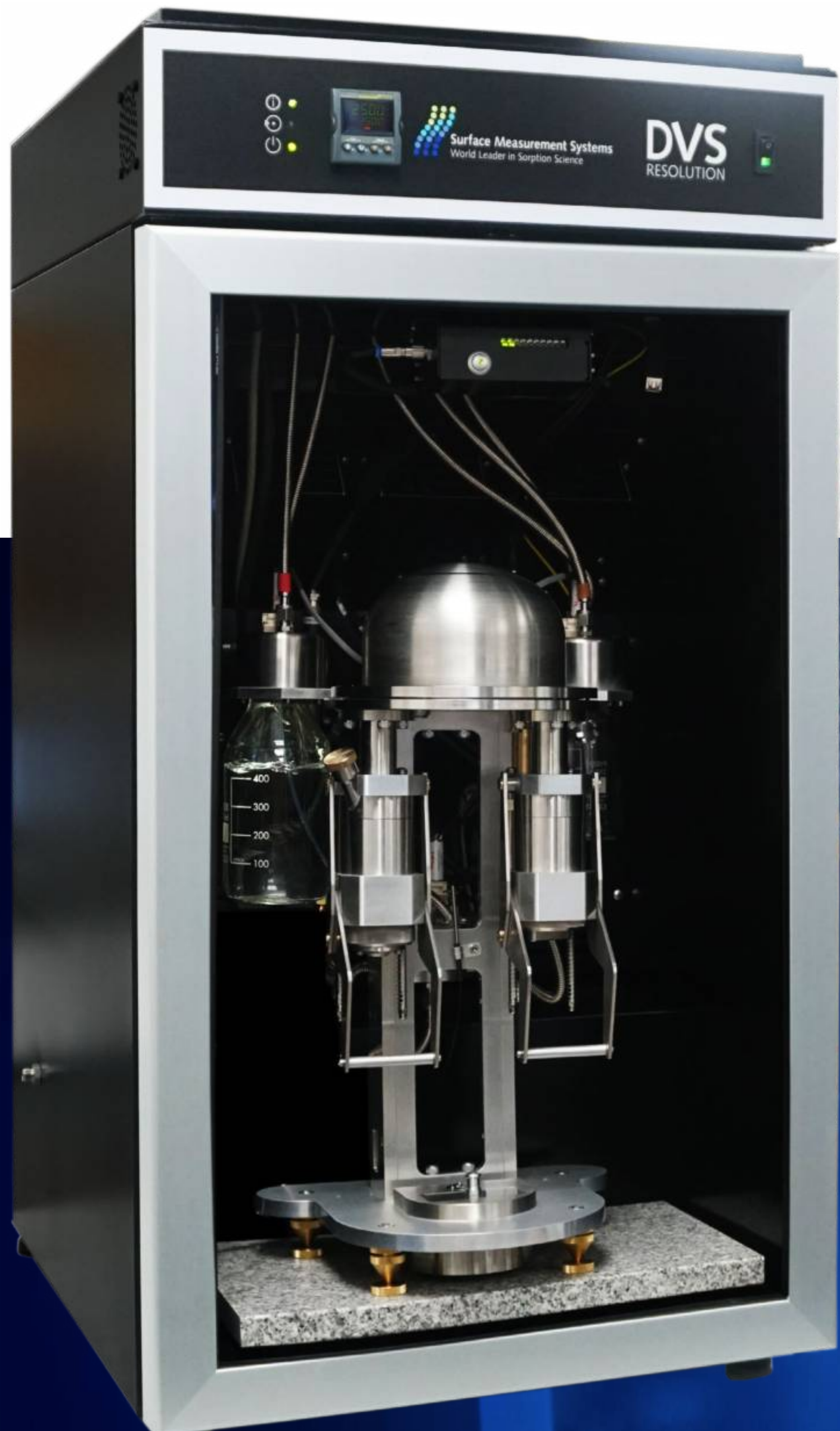
DVS
RESOLUTION



The Most Advanced Gravimetric Vapor Sorption Instrument

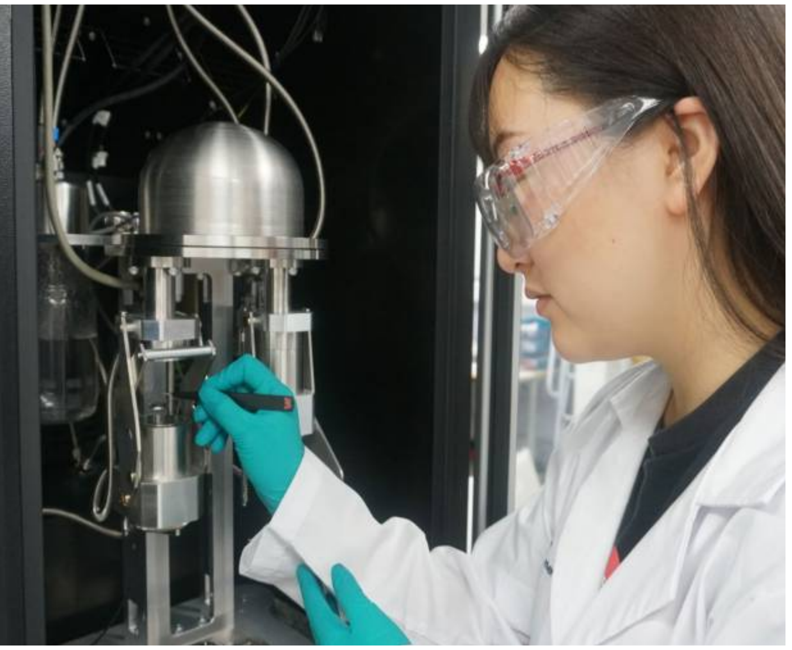
- Organic and water vapor sorption kinetics from 5 to 85 °C
- Organic and water vapor sorption isotherms
- Co-adsorption with two vapors
- Real time partial pressure measurement and control
- *In-situ* sample drying/activation
- Color video microscopy/fiber optic probe spectroscopy
- True0 drying at 0.0% RH
- Upgradable and modular system

www.surfacemeasurementsystems.com

DVS Resolution Dual Vapor Gravimetric Sorption Analysis

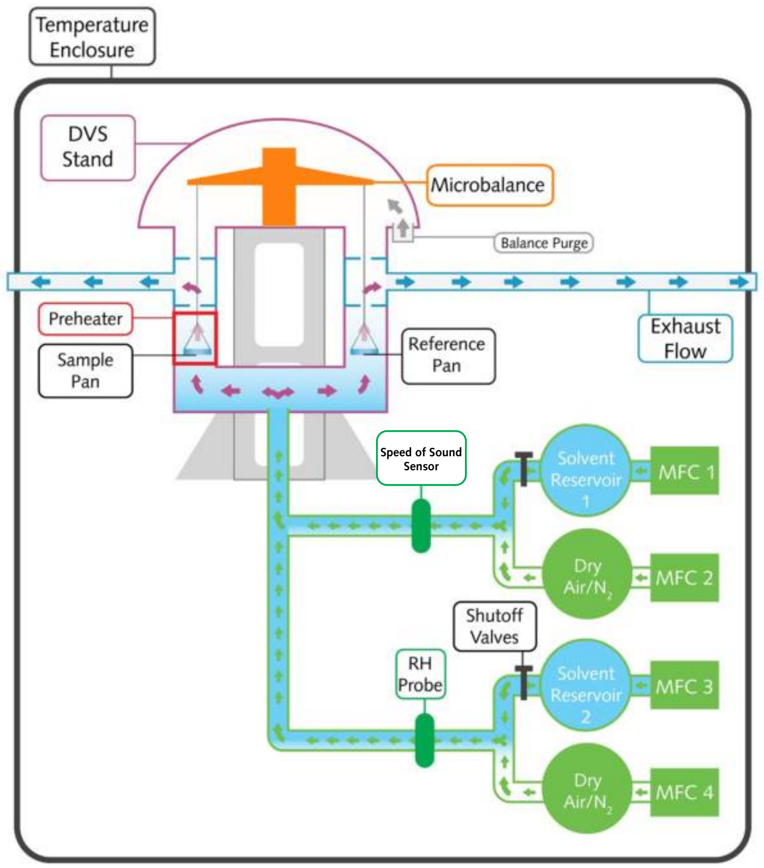
Key Benefits

Dynamic Vapor Sorption (DVS) is a gravimetric sorption technique that measures the rate and amount of solvent sorbed and released by a sample, such as a dry powder absorbing or releasing water. The DVS accomplishes this by varying the vapor concentration surrounding the sample and measuring the change in mass which this produces.



Applications

- Hygroscopicity of pharmaceutical solids
- T_g and RH phase transitions in polymers
- Amorphous content determination of solids
- Diffusion and permeation in polymers
- Food, flavors and fragrances
- Sorbents
- Wood and cellulosic materials
- Composites
- Hydrophillic and hydrophobic materials



Standard DVS Resolution Schematic

Hardware

- The only system able to measure organic vapor partial pressure directly using the Speed of Sound Sensor (patent pending)
- Open stainless steel stand design enabling easy access to sample pan while minimizing static electric charging
- Accurate and uniform temperature across a broad temperature range (from 5 to 85 °C)
- Optional IR, Raman and video imaging with integrated control software
- Quick and easy to change reservoir bottles

Software

Software - the software package provided with the DVS Resolution allows the users to create and customize experimental methods while enabling the full analysis of the data collected. Examples of the control and analysis software used in a standard water sorption experiment are outlined below.

- Control Software

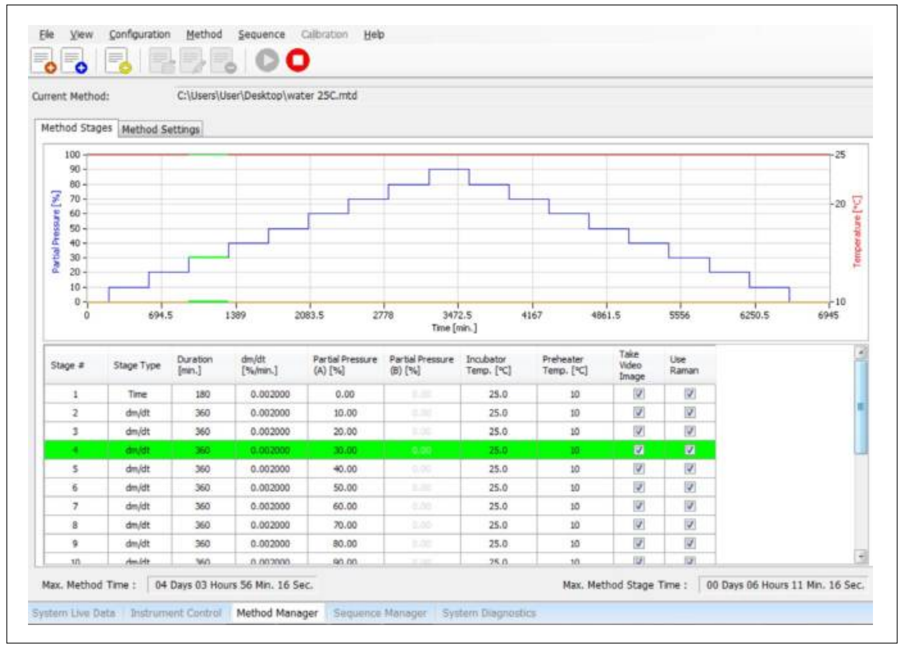


Figure 1. The above graph shows the method panel within the method manager. It displays numerically and graphically the current method for a water sorption experiment at 25 °C. The active stage of the ongoing experiment is highlighted in green. Figures 2 and 3 (below) are typical data generated by this method.

- Analysis Software

Water Sorption Data

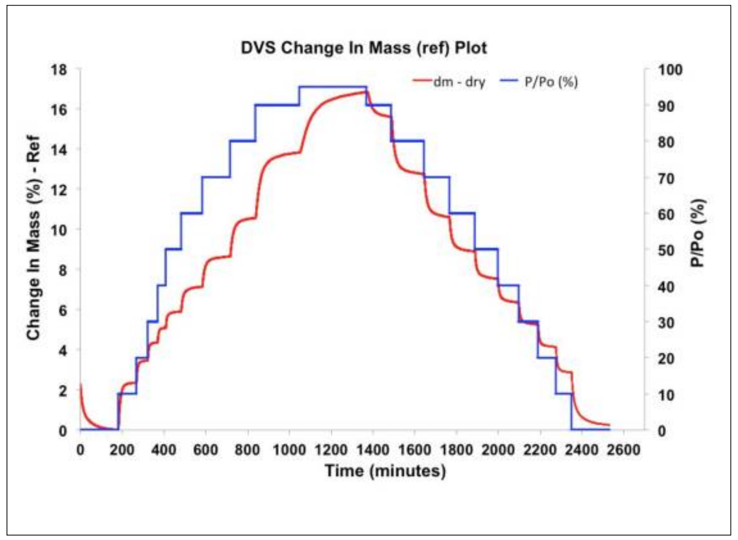


Figure 2. Water sorption kinetics of cellulose membrane at 25 °C

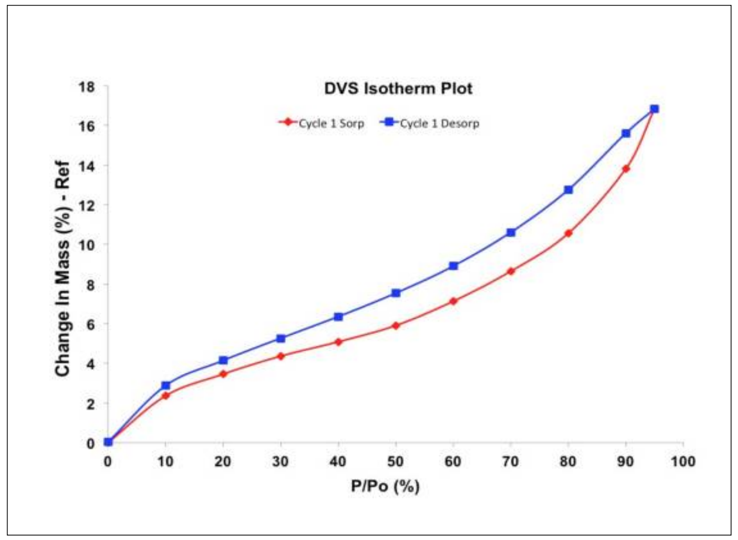


Figure 3. Water sorption isotherm of cellulose membrane at 25 °C

Outstanding Performance

The DVS Resolution allows for the collection of high quality data, owing to the outstanding Ultrabalanace performance (Figure 4), precise vapor generation (Figure 5) and accurate temperature control (Figures 4, 6, 7).

Mass and Temperature Measurement

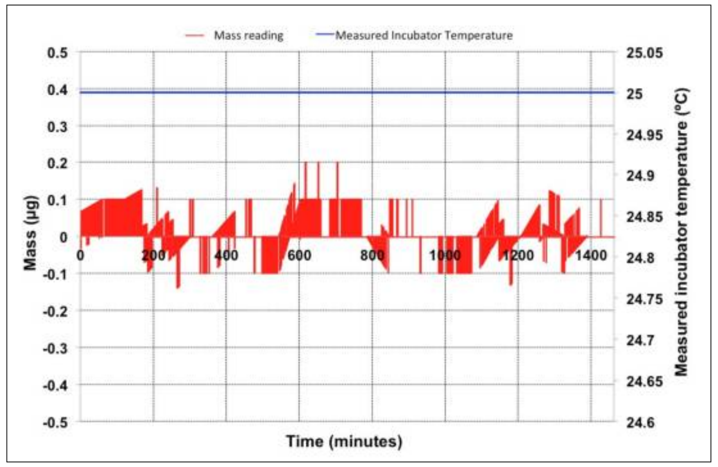


Figure 4. DVS Mass Baseline Stability Plot Over 24 Hours

- Mass changes at a resolution of 0.01 µg for low mass balance
- Root mean square noise of ≤ 0.3 µg for low mass balance (averaged over 24 hours)

True0™ RH

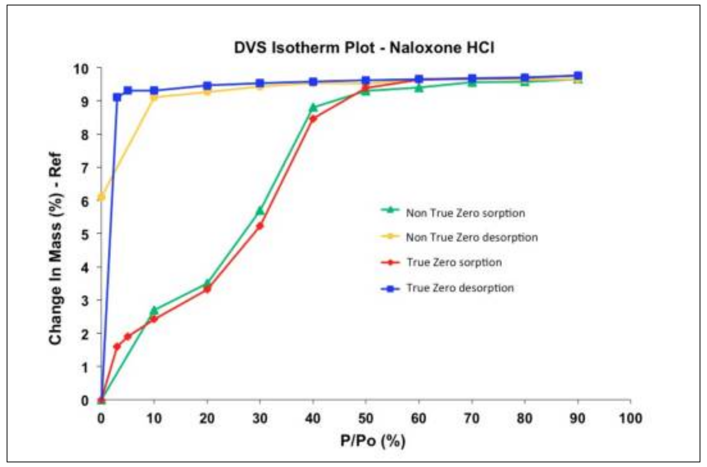


Figure 5. Comparison of Naloxene Hydrochloride Dihydrate Water Sorption Isotherms

- Only DVS instrument offering True0™ RH
- Achieves partial pressures of water as low as 0.0% RH
- Hydration and dehydration kinetics below 1% RH can be readily studied

Temperature Control & Stability

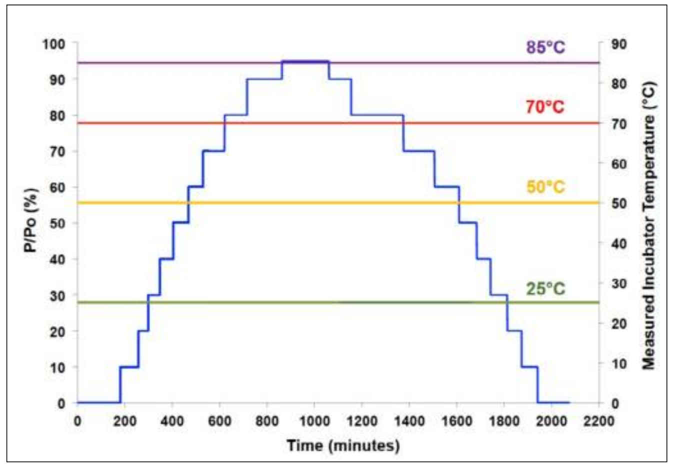


Figure 6. Kinetic plot of partial pressure (or relative humidity) at different temperatures*

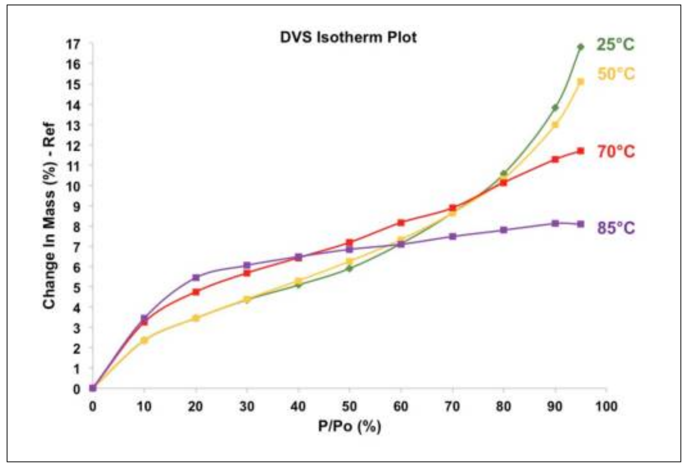


Figure 7. Water sorption isotherm of cellulose membrane at different temperatures

- Stability at 25 °C is ± 0.05 °C over 6 hours
- Vapor generation and delivery at sample temperature prevents condensation issues typically found in instruments with multiple temperature zones
- Allows for accurate and stable isotherm experiments

For extended experimental operation at 85% RH at 85 °C an optional heated reservoir accessory can be supplied.

Solvent Delivery Configurations

The **DVS Resolution** can precisely deliver combinations of:

(1) Humidity with controlled generation using a humidity sensor (2) Organic vapors with controlled generation using a proprietary **Speed of Sound** sensor.

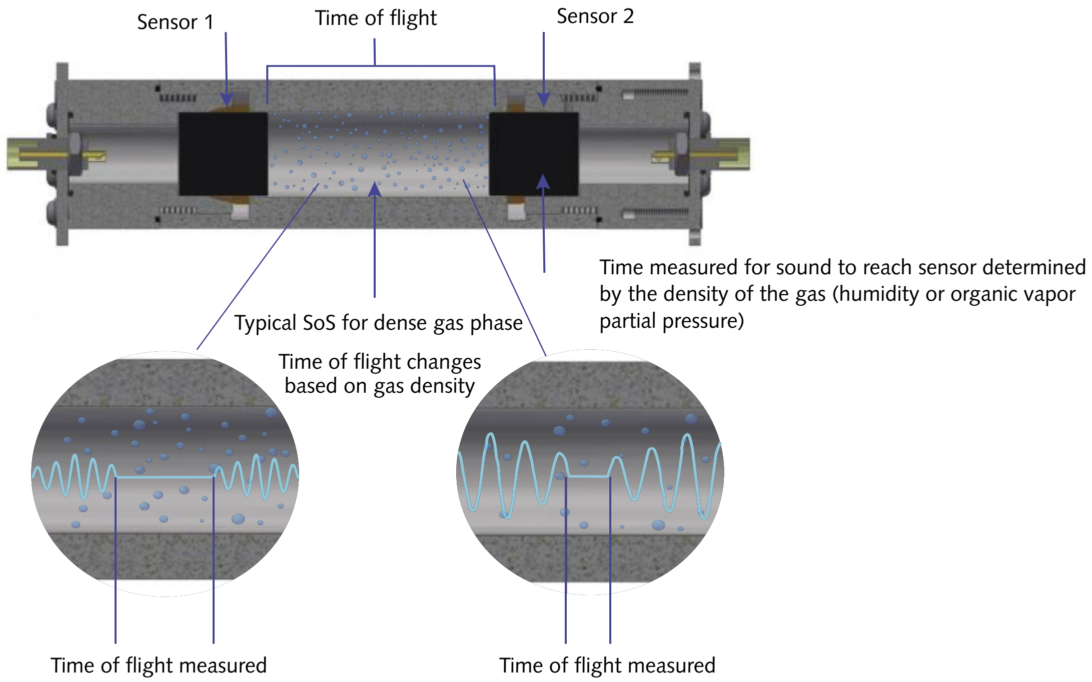
The DVS Resolution can be provided in two different factory configurations:

a. **Standard**: provided with a humidity sensor and a single SoS sensor, to allow the simultaneous generation of water and an organic solvent.

b. **Advanced**: provided with a humidity sensor and a two SoS sensors, to allow the simulataneous generation of water and an organic solvent or alternatively, two different organic solvents.

Speed of Sound Sensor

Speed of sound is an intrinsic property of the vapor or gas measured, and depends on the temperature, gas/vapor concentration and gas/vapor species. The **Speed of Sound (SoS) Sensor*** in the **DVS Resolution** is the only method to directly measure the vapor concentration ultrasonically. The SoS sensor determines the gas/vapor concentration in real time based on the speed of sound travelling through a fixed volume of solvent gas/vapor.



Key features of SoS Sensor:

• SoS based concentration measurements enable active closed loop control, which is significantly more accurate than open loop control

• Complex simultaneous generation of 2 different organic solvents can be achieved through the use of dual SoS sensors technology (DVS Resolution advanced)

• Resolution of ±0.1% P/P_0 of target P/P_0

Partial Pressure Control

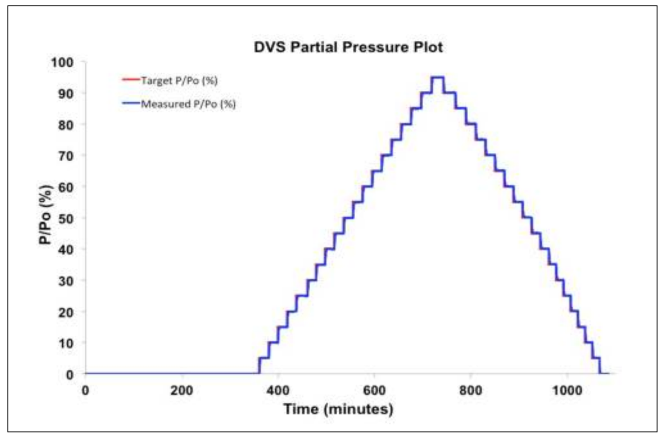


Figure 8. Kinetic of the partial pressure of acetone measured using the SoS Sensor during a DVS experiment

patent pending

Applications

The DVS Resolution also allows the user to perform complex and advanced material analysis such as BET surface area (Figures 9 & 10), co-adsorption experiments (Figure 11) and co-diffusion experiments (Figure 12).

Typical Organic Vapor Sorption Data (Single Solvent)

Organic Solvent Sorption Experiment

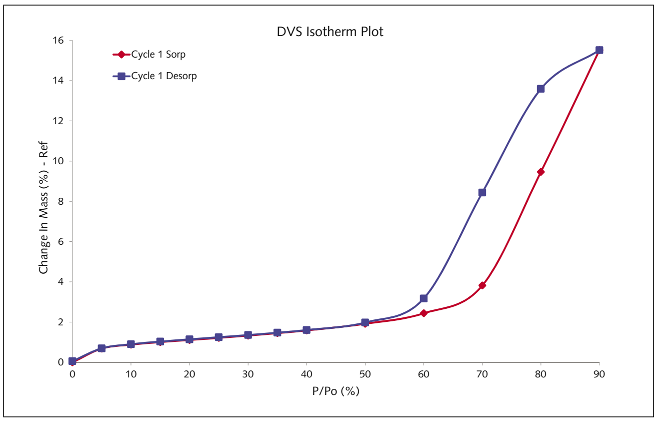


Figure 9. DVS Isotherm Plot

- Octane isotherm plot for ceramic powder at 25 °C

Surface Area Calculations

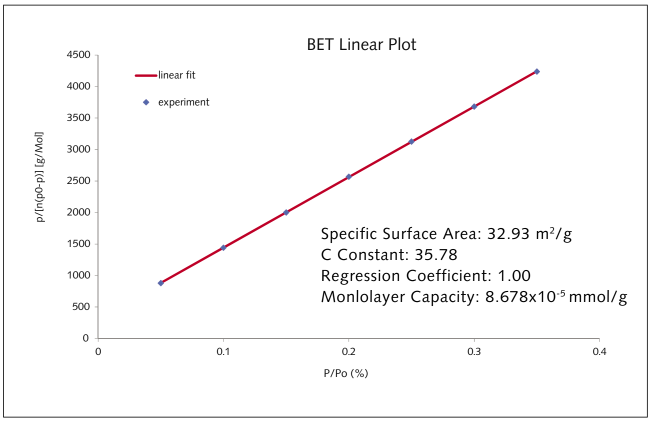


Figure 10. BET Linear Plot

- Octane BET linearized plot for surface area determination at 25 °C

Dual Solvent Experiments

Spray Dried Polymer Co-Adsorptions Isotherms

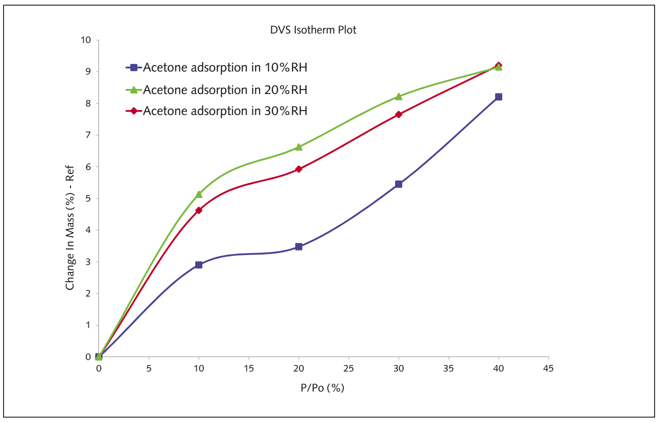


Figure 11. Acetone sorption in background humidity

- Acetone isotherm in blue/10%RH background
- Acetone isotherm in green/20%RH background
- Acetone isotherm in red/30%RH background

Thin Film Co-Diffusion

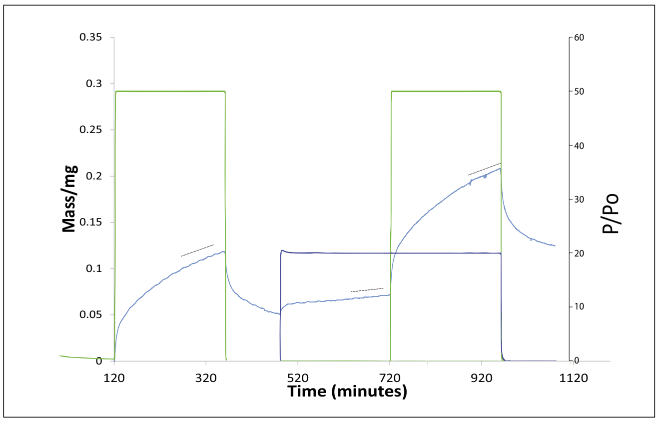


Figure 12. Kinetic plot of multi-component diffusion experiment performed on Kapton tape

- Co-diffusion of two solvents into a thin film
- Independent control of individual moisture and organic vapor flows for single component and co-adsorption experiments

Modular Capabilities

Raman Spectroscopy

- Fully integrated hardware/software solution for triggering and capturing Raman spectra during sorption experiments
- Simultaneous operation of Raman and optical microscopy during the DVS experiment
- Allows for a more complete understanding of vapor-solid interactions for materials

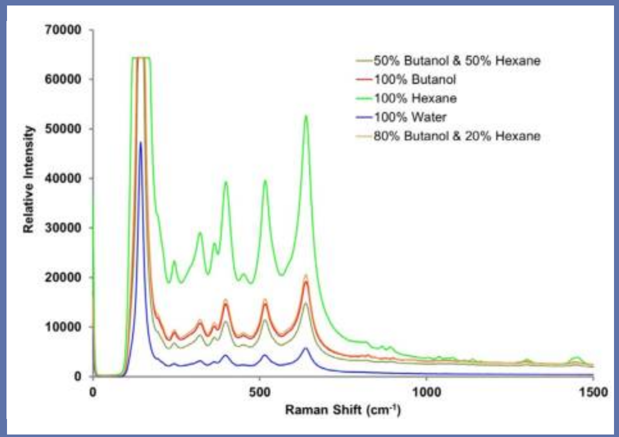


Figure 13. Raman spectra of single and dual-solvents sorption of Butanol and hexane for a titanuim oxide sample at 25 °C

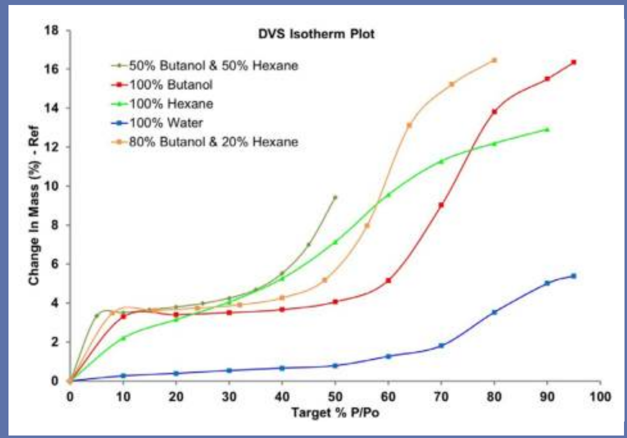
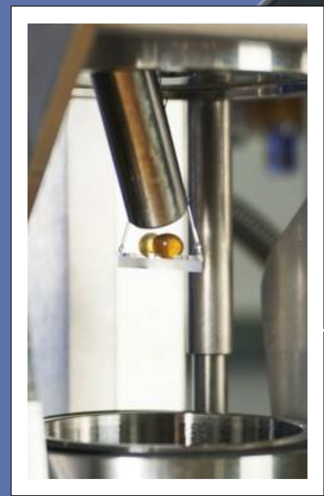
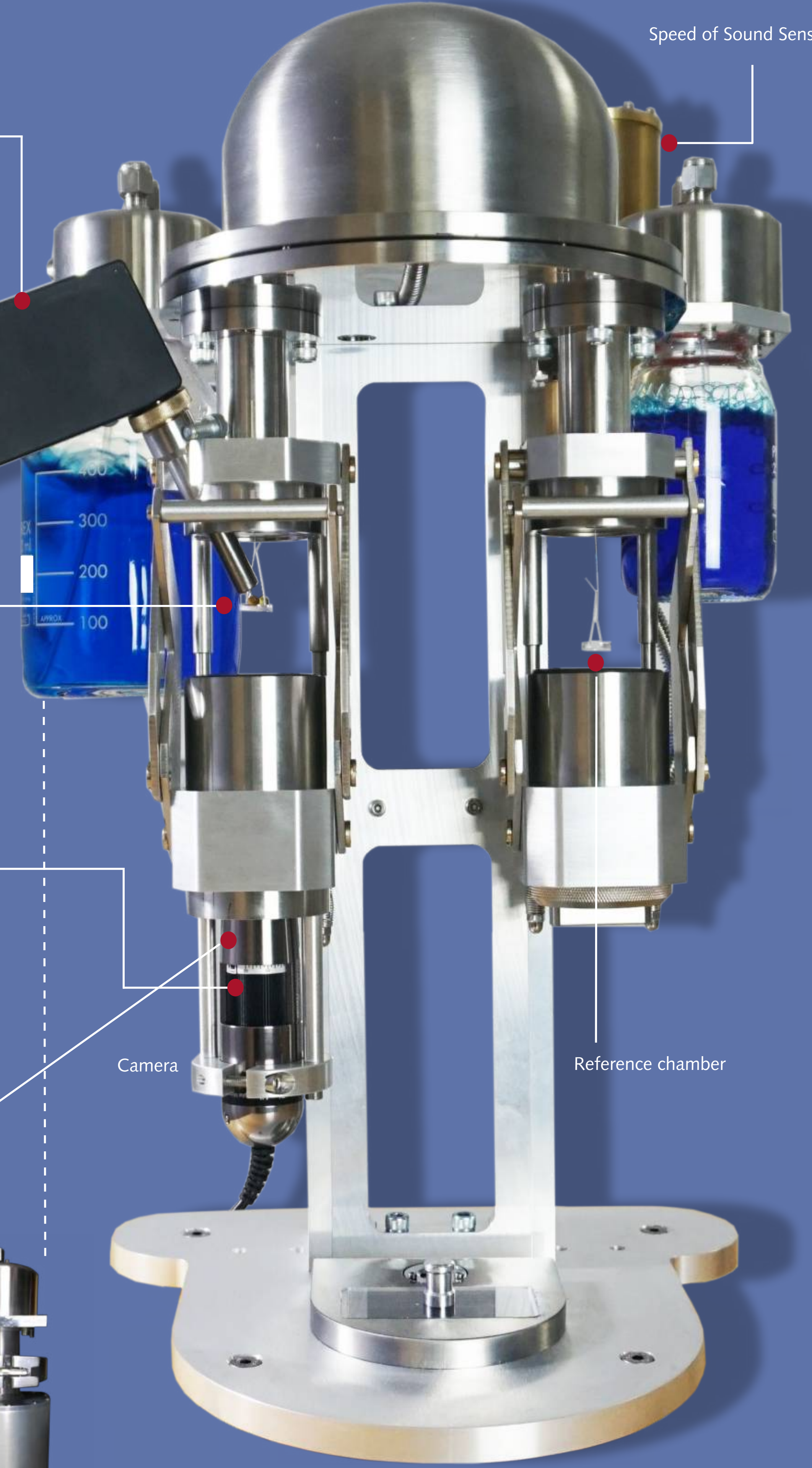


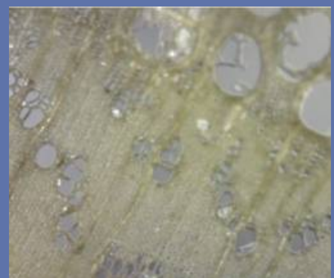
Figure 14. Single and dual-solvents isotherm of Butanol and hexane for a titanium oxide sample at 25 °C





Microscopy and Video

- 1.3 megapixel color camera
- Up to 200x optical zoom
- Images have time-date-temperature-partial pressure stamps
- Grid overlay and calibration for measuring dimensional change
- The images can be composed into a timelapsed video



Cedar wood

Cellulose

Agricultural seeds

High Temperature Preheater*

(for drying and curing)

- *In-situ* degassing/activation of samples up to 200 °C
- The temperature is measured by a Pt-100 directly below the sample pan
- User programmable and controlled temperature ramps or steps

Important: Camera and Raman cannot be used while the Preheater is mounted to the chamber

Heated reservoir accessory*

- The heated reservoir replaces the standard glass bottle mounted on the left of the stand
- Designated for extended humidity generation 85% RH at 85°C, with fully automated temperature control

*For more information on the specification of these accessories, please contact sales@surfacemeasurementsystems.com

Surface Measurement Systems
World Leader in Sorption Science

Technical Specifications

Temperature

Temperature controlled enclosure

Control range: 5 °C to 85 °C

Temperature stability ± 0.05 °C over 6 hours

Temperature resolution 0.01 °C

High Temperature Pre-heater for drying samples

200 °C (maximum local temperature)

Heating ramp rates: up to 5 °C/min

Temperature sensor: Pt-100

Resolution Stand

Manifold: 316 stainless steel

Seals: Viton® and Kalrez® or equivalent

Tubing: 1/4 inch stainless steel

Solvent Reservoirs

2 glass reservoirs as standard

Heated reservoir [1] (option for extended 85 °C 85% RH operation)

Flow Control

High accuracy digital mass flow controllers

Wide dynamic range - turndown ratio 1000:1

Carrier Gas - Dry air or Nitrogen

Relative Humidity

Relative humidity range from 0 to 98% for 5-60 °C [2]

Relative humidity range from 0 to 85% for 60-85 °C [1,2]

Relative humidity resolution ± 0.1%

Relative humidity stability ± 0.1% over 6 hours

RH range accuracy from 5 - 60 °C ± 0.5%[3]

RH range accuracy from 60 - 85 °C ± 1%[3]

Organic Vapor Generation and Measurement using Speed of Sound Sensor [4]

Partial pressure range from 0 to 90% P/P_o [5]

P/P_o resolution ± 0.1%

P/P_o accuracy from 5 - 85 °C ±1% P/P_o [3,6]

Footnotes

[1] Optional configuration (heated reservoir) for long term 85 °C, 85% RH operation

[2] System factory calibrated at 25°C. Calibrations at other temperatures upon request.

[3] 1-σ confidence level with % RH or P/P_O calibration performance based on SMS factory certified methods (Salt Calibration)

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- Surface area models
- Amorphous content
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- T_g determinations

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Advanced

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- Isotherm Analysis Suite

21CFR Part 11 software solution (optional)

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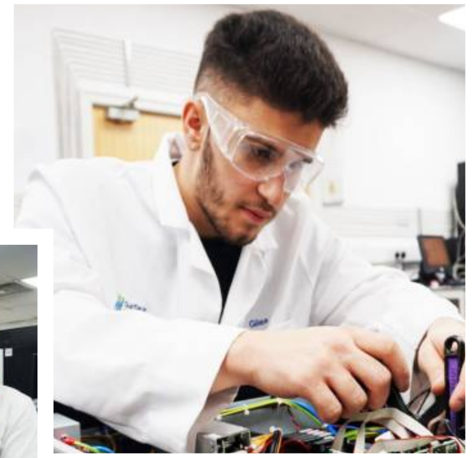
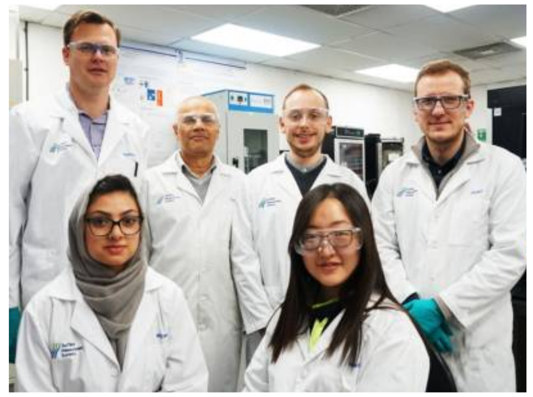
[5] Depending on the solvent selected and experimental temperature

[6] Depending on the solvent selected

[7] Root mean square (averaged over 24 hour)

About Us





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- Outstanding instrument performance
- Most complete and intuitive Windows™ software for experimental control and analysis
- Winner of Innovation Award 2018 and ISO 9001:2015 Compliance

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ISO 9001:2015

WINNER Innovation Award FUTURE awards

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version 1.0
15042020

SCAN ME

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DVS
VACUUM

Surface Measurement Systems
World Leader in Sorption Science

Dynamic Dual Vapor/Gas Gravimetric Sorption Analyzer

Capabilities:

- Dynamic/Static isotherms and isobars
- Competitive Adsorption
- *In-situ* degassing
- Real time sorption kinetics

D V S   V a c u u m

Dynamic Gravimetric Vapor Sorption System

The DVS Vacuum is the only gravimetric vapor sorption system which offers static and dynamic sorption experiments. It accurately and precisely measures adsorption-desorption isotherms and adsorption-desorption isobars over a broad range of temperatures. It provides insights about the interactions and total adsorption capacities of studied materials with water vapor, organic vapors, gases and their mixtures. More importantly, it provides data on the fundamental understanding of adsorption processes which can be used in theoretical models. The DVS vacuum is versatile due to its unique upstream and downstream sorbate control for real-time adsorption and desorption kinetics. It can be used to mimic industrial conditions, and thus assess the performance of materials prior to their deployment in adsorption based technologies.

This is particularly attractive for chemical engineers and researchers pursuing novel energy-related applications through systematic tailoring of sorption properties to specific needs such as thermally driven heat pumps, adsorption based thermal storage systems, drying, purification and separation of gases and CO_2 capture from power plant flue gas. Additional DVS Vacuum applications are found in fundamental drying and sorption studies of nanoporous materials, composites, membranes, porous ceramics, activated carbons and pharmaceutical actives and processes. The DVS Vacuum's experimental flexibility enables studying adsorption and desorption under dynamic isothermal or isobaric conditions. It performs multi-component experiments using vapor and/or gas sorbate molecules with *in-situ* sample degassing up to 400°C and high vacuum. Multiple adsorption/desorption and sample regeneration cycles can be performed with real-time sorption kinetics and thermodynamics for sample masses from 5 to 1000 mg. This greatly expands the physisorption characterization capabilities available for such materials.

Zeolites, porous polymers, composites, Aluminophosphates (AlPOs) and Silica aluminophosphates (SAPOs), silica gels, activated carbons and Metal Organic Frameworks (MOFs) are important classes of materials used in various sorption based technologies. The combination of hierarchical pore structure control and selection of appropriate adsorbents enables entry and adsorption of small molecules on internal surfaces. Such processes are typically controlled by physisorption mechanisms governed by molecular size, polarity and chemical nature of the sorbent surfaces. In some cases specific chemical interactions can give rise to more strongly bound chemisorbed species, which are an essential part in the design of heterogeneous catalysts.

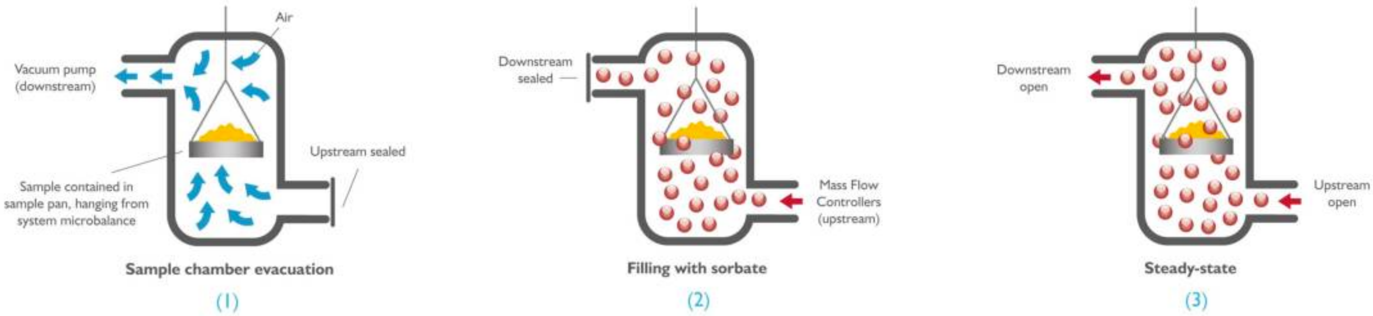
Key Measuring Capabilities

- Adsorption and desorption under dynamic/static, isothermal, or isobaric conditions.
- Competitive adsorption of two probe molecules (i.e. CO_2–H_2O, Toluene–H_2O, MeOH–H_2O).
- Perform multi-component experiments using vapor and/or gas sorbate molecules.
- Unique vapor temperature and delivery control.
- *In-situ* sample degassing up to 400°C and high vacuum ($2x10^{-6}$ Torr).
- Multiple adsorption/desorption and sample regeneration cycles.
- Real-time sorption kinetics and thermodynamics.
- Experimental background pressures as low as $1x10^{-6}$ Torr.
- Adsorption measurements at relative pressures as low as 0.005 Torr.
- Minimum deliverable relative water vapor pressure at 25°C of 0.05%.
- Sample masses from 5-1000 mg.

The principle of DVS Vacuum

A unique principle of the DVS Vacuum is the ability to control and measure sorbate entry and exit flows simultaneously while recording changes in sample mass. The benefits include: broad range of sorption experimental partial pressures, real time sorption kinetics, sorbate molecule residence time control and thermal cycling. The DVS Vacuum also covers a wide range of sample and vapor temperatures allowing for sample thermodynamic properties determination. This includes capability for *in-situ* sample degassing at elevated temperatures and high vacuum. A wide range of molecules, both gas and vapor phase, are controlled through a sophisticated flow control system. Additionally, as the entire DVS Vacuum vapor generation delivery system and sample chamber are at thermal equilibrium in the temperature controlled enclosure, variable sorbate molecules and temperature can be easily changed without condensation and decontamination concerns. Tailoring the sorbate molecule gives access to many physico-chemical parameters.

The principle of dynamic adsorption

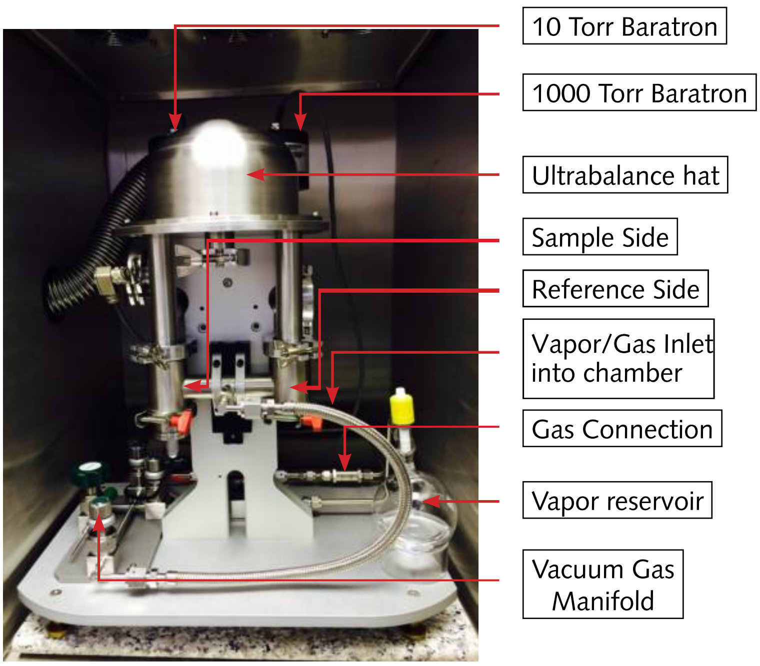


1. *In-situ* degassing of sample under high vacuum and high temperature. Downstream consisting of butterfly valve controlling pumping speed is fully open, while upstream controlled by mass flow controllers is closed.

2. Filling the chamber with sorbate molecules. Upstream is open, while downstream is closed.

3. Dynamic state whereby the amount of sorbate molecules entering and leaving the chamber is controlled simultaneously.

DVS Vacuum layout and capabilities



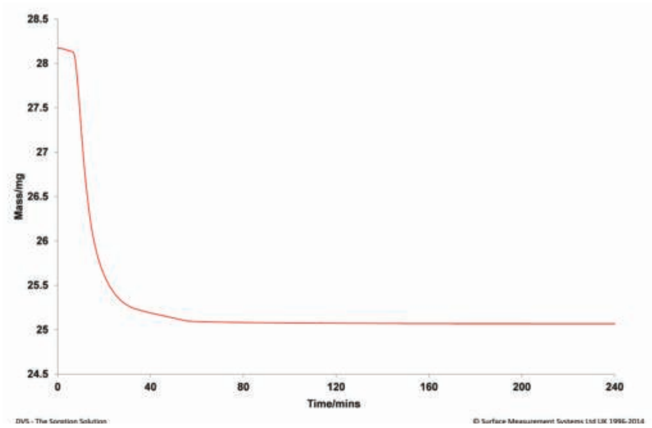
- Dynamic (or static) operational mode
- Upstream and downstream vapor/gas flow controls
- Lowest measurable pressure 0.005 Torr
- Temperature controlled enclosure operating in the range from 10 to 70°C
- *In-situ* sample degassing up to 400°C (using pre-heater) and high vacuum
- Multi vapor and/or gas injection system for sorbates (2 gases or 2 vapors or 1 gas and 1 vapor)
- Water vapor adsorption in the temperature range 10 to 70°C up to 90%P/Po and above 70°C in a limited P/Po range.
- Highly accurate Baratrons for pressure measurements
- Transducer for vacuum pressure measurement

Applications of DVS Vacuum

Drying kinetics and *in-situ* degassing of samples

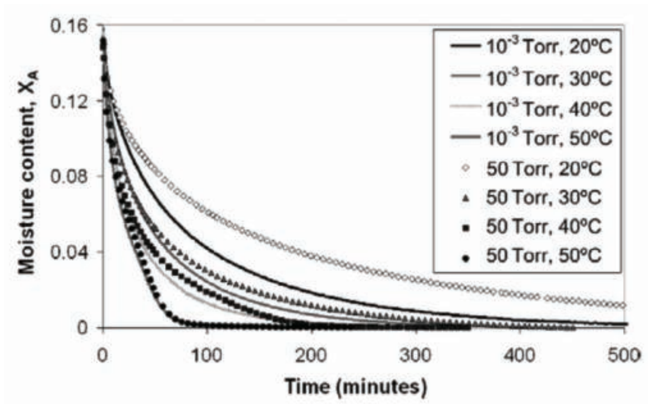
DVS Vacuum extends the capability of sample degassing to drying kinetics and mass equilibrium prior to sorption measurements. Samples can be *in-situ* degassed at temperatures of up to 400°C using various heating rates and vacuum pressures down to 10^{-7} Torr. This is immediately followed by sorption experiment at desired sorption temperature upon cooling the sample to experimental temperature. Sample transfer is not required for degassing.

Degassing of 13X Zeolite



Drying curve of 13X zeolite, which was degassed *in-situ* at 400°C and high vacuum.

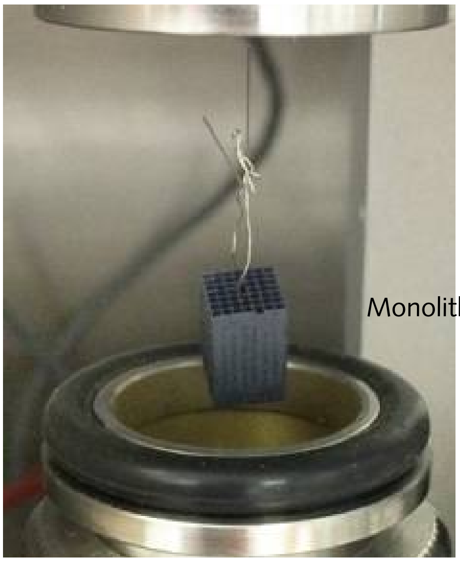
Drying kinetics of a pharmaceutical powder



The effect of temperature and vacuum pressure is shown to provide information on dehydration kinetics of carbamazepine dihydrate.

High temperature vacuum preheater and housing for variable sample geometries

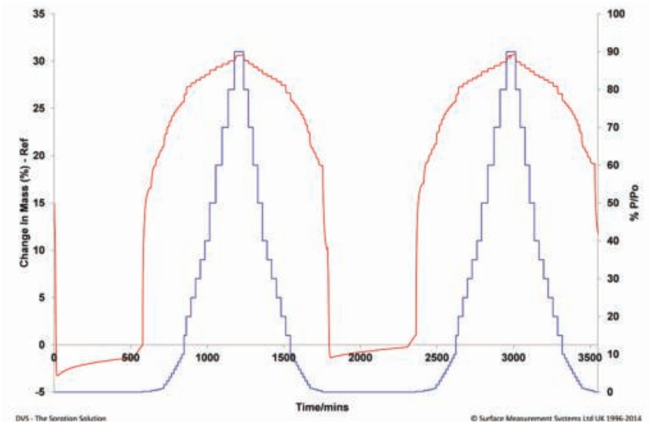
High temperature pre-heater for *in-situ* degassing/regeneration of samples up to 400°C. Temperature is measured by Pt-100 thermocouple, which is placed below the stainless steel sample pan.

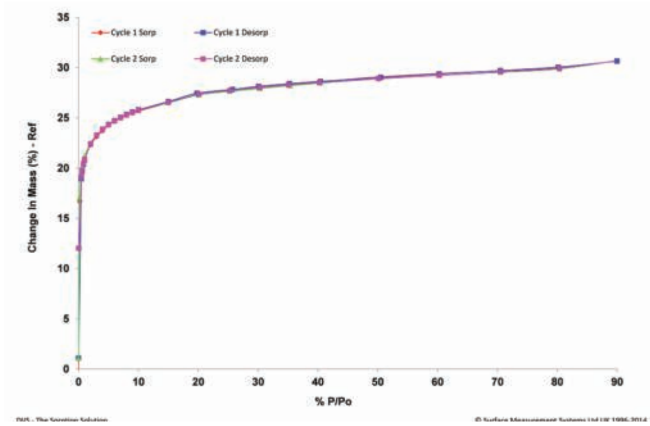


Variable samples such as catalytic converters, thin films and porous membranes can be measured in the system. The sample is connected directly on the hangdown wire. In typical experiments, where powders, pellets or beads are used, samples are loaded in stainless steel pans. Other experimental cells are available for a wide range of sample types and applications such as diffusion/permeation and Knudsen vapor pressure.

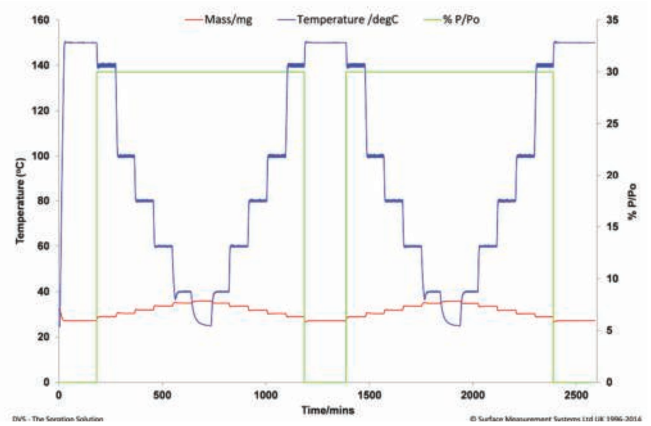
Water vapor adsorption in zeolites and MOFs for thermal adsorption storage and heat transformation

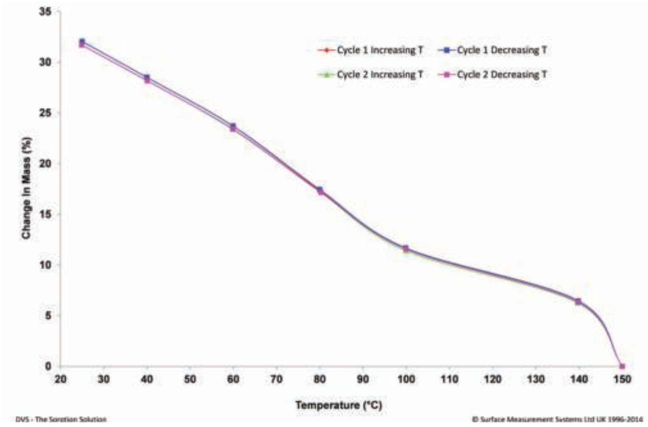
Micro- and meso-porous materials such as zeolites 4A, A10 Sylosiv, 13X, MCM-41 respectively, aluminophosphates including SAPO-34, and MOFs have gained much interest recently due to their promising applications in novel heating and cooling technologies, exploiting thermo-adsorptive effects. These materials show interesting sorption properties; high or low uptakes in the low partial pressure range in which water can be considered as an adsorbate for cooling applications. Therefore, adsorption by these materials in the presence of water vapor is extensively studied in order to understand water vapor sorption kinetics at low partial pressures, the total adsorption capacity, stability of material after several adsorption/desorption cycles over wide range of temperatures and energy requirements for the regeneration of adsorbents. In addition, the selection of various probe molecules and their mixtures (water- alcohol) give access to physico-chemical parameters that are critical for understanding the material performance in extreme conditions. Examples of isobars and isotherms including sorption kinetics of promising adsorbents are shown below.



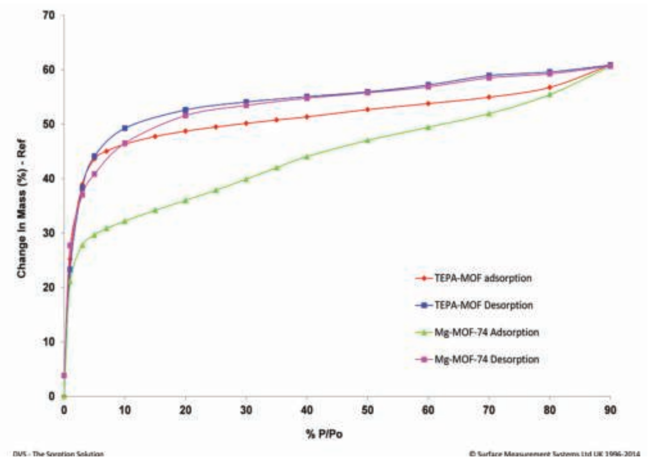


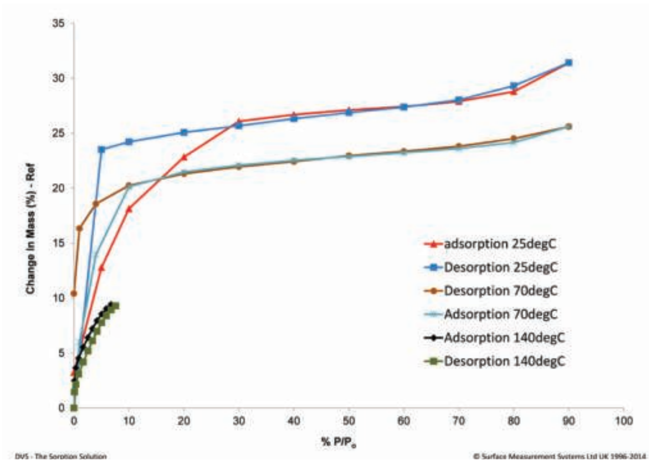
A10 (powder, Grace Chemicals) water vapor sorption at 40°C. Prior to sorption measurements A10 was *in-situ* degassed at 180°C under high vacuum of 10^{-5} Torr for 480min and subsequently cooled down to sorption temperature of 40°C. Sample was regenerated between cycles using the same degassing conditions. Resulting water adsorption-desorption isotherms of A10 is shown on the right.





A10 water vapor isobars at 7.14 Torr (Figure left). Two cycle of A10 showing change in the mass (in red) during stepping down (adsorption) and stepping up (desorption) temperature (in blue) at pressure (in green) of 7.14Torr. A10 water vapor isobar at 7.14 Torr (Figure right).

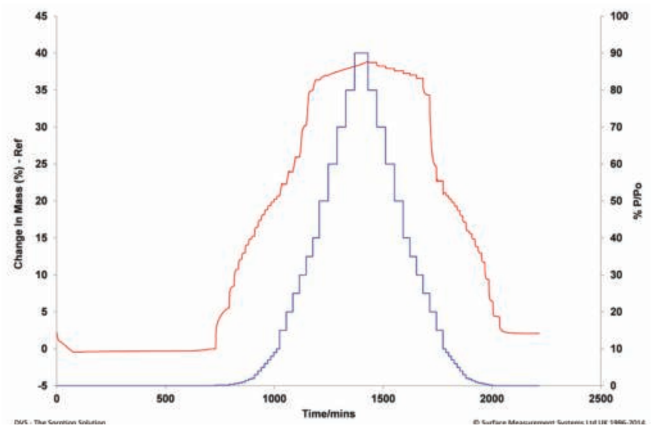


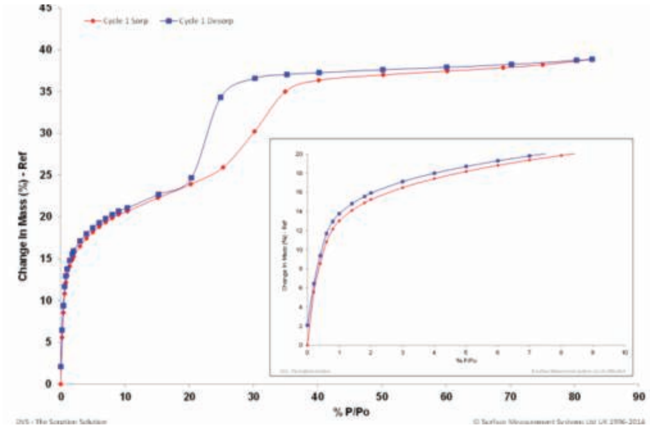


Comparison of 25°C water vapor adsorption-desorption isotherms for Mg-MOF-74 and TEPA-MOF-74. Mg-MOF and TEPA-MOF were prepared by Xiao Su & Lev Bromberg from MIT, Chemical Engineering. (Figure left). Comparison of 4A (Sigma Aldrich, 1/16inch diameter, 1-2mm pellets) water adsorption- desorption isotherms measured at 25, 70 and 140°C (Figure right). The large hysteresis at 25°C is due the fact that mass equilibrium at low P/Po was not reached.

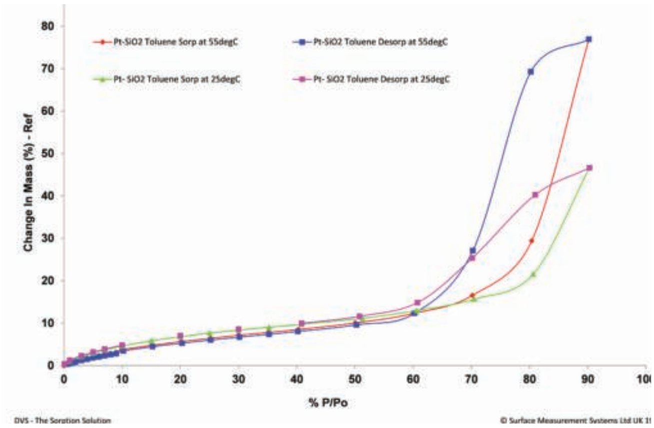
Molecular separations of catalysts and zeolites

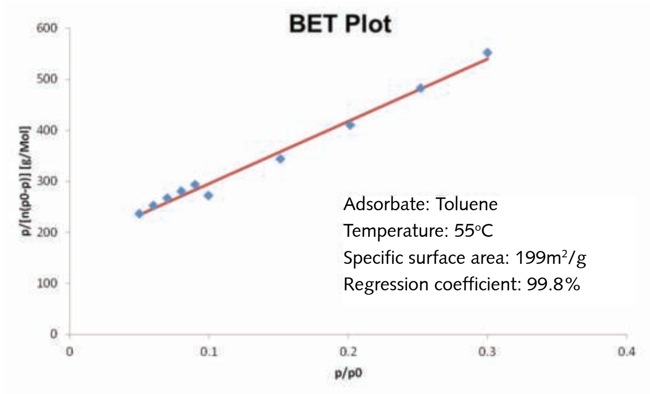
A wide range of sorbate molecules including water, organic vapors (i.e. alcohols, toluene, benzene, cyclohexane, xylene, acetaldehyde, etc.) and gases are controlled through a sophisticated flow manifold. The selection of sorbate molecules for specific adsorption based applications gives access to many physico-chemical parameters, such as size selectivity and or chemical selectivity of the sample.





Zeolite Y benzene sorption at 25°C. Adsorption-desorption cycle showing benzene sorption kinetics (left) and corresponding isotherm (right) at 25°C. The inset shows the isotherm in the Henry region where pressure was increased by 0.2 % P/Po.

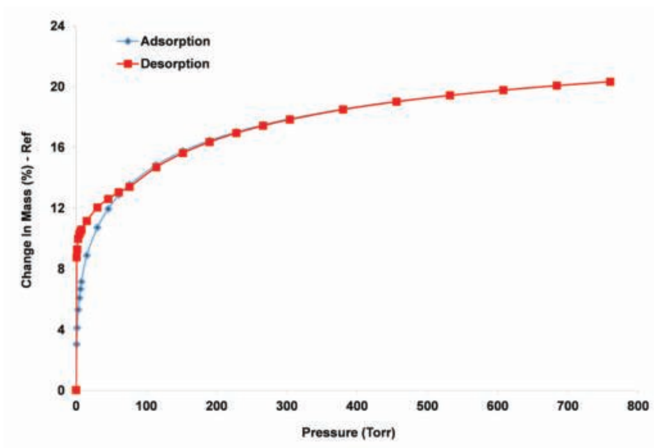




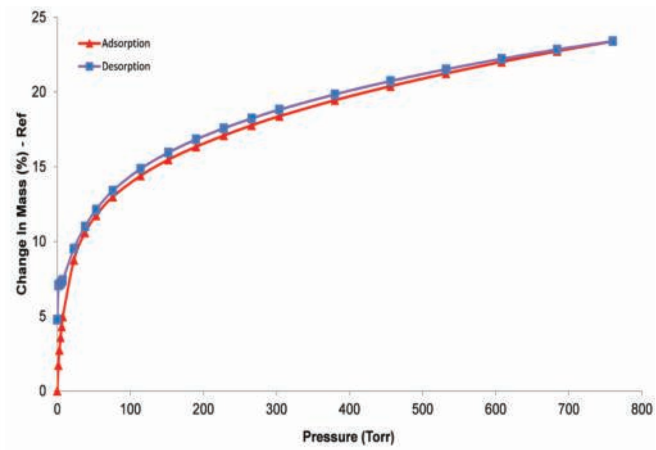
Comparison of Pt-SiO_2 toluene adsorption-desorption isotherms at 25 and 55°C (left). Determination of specific surface area using BET equation for toluene sorption at 55°C (right).

MOFs and zeolites as adsorbents for CO_2 capture and storage

MOFs and zeolites are promising porous crystalline materials for CO_2 capture. Both materials are strong candidates due to high adsorption capacities, the economic feasibility of its reagents as well as ease of synthesis.



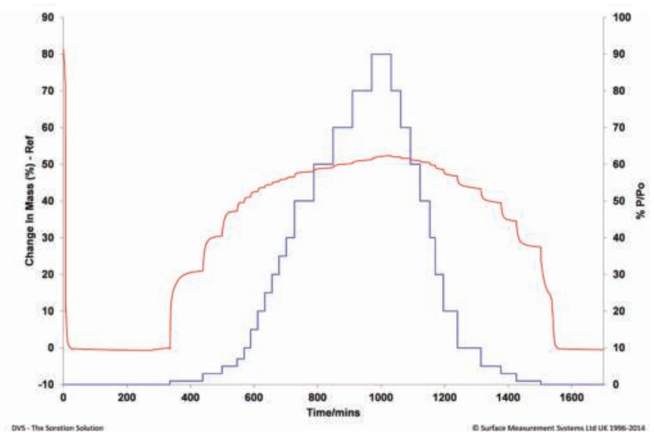
13X adsorption-desorption of CO_2 in the pressure range from 0 to 760 Torr at 25°C. 13X was in the form of beads having 1-2mm diameter.

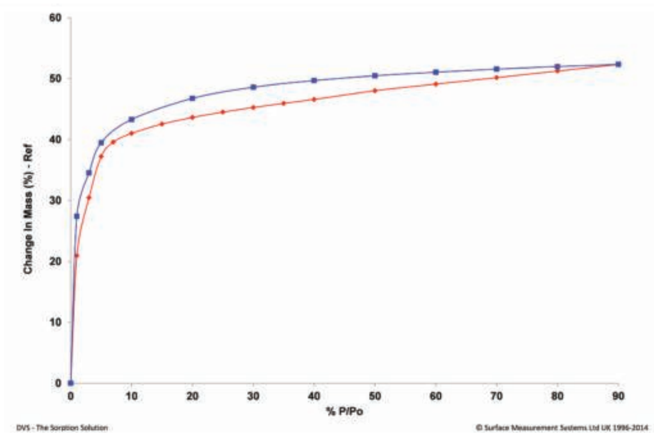


Mg-MOF-74 adsorption-desorption of CO_2 in the pressure range from 0 to 760 Torr 25°C.

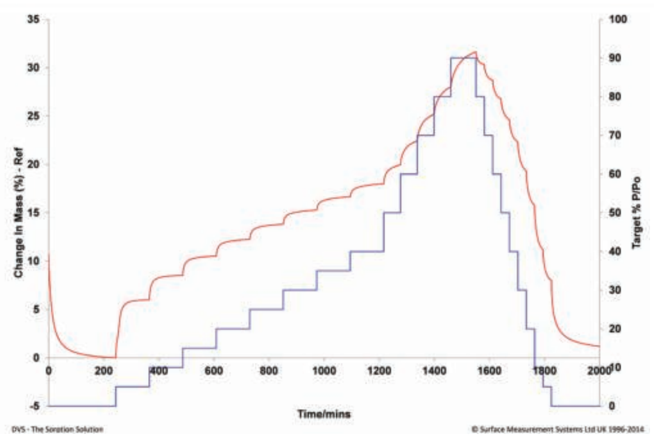
Co-Adsorption on MOFs and fibers

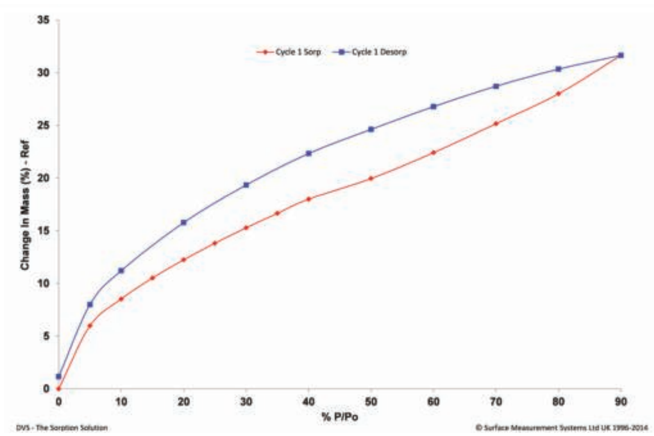
Many industrial processes, such as gas purification, drying, filtration and carbon capture, use MOFs, porous solids or porous polymers to adsorb gases or organic molecules. Water vapor often co-exists with gases or organic molecules and its concentration can vary from very low to saturated. Therefore, it is important to understand the effects of the presence of water vapor on adsorption of gases or organic molecules. DVS Vacuum can be used to study co-adsorption of two adsorbates over broad temperature and relative pressure ranges.





50/50 CO_2/H_2O Mg-MOF-74 sorption at 25°C. Sorption kinetics shown on left and corresponding isotherms on right with adsorption in red and desorption in blue.





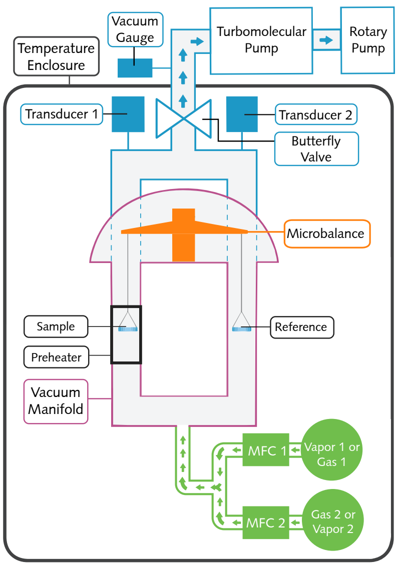
Natural fiber 50/50 Toluene/Water sorption at 25°C. Prior to sorption measurements the fiber was dried at 25°C and under high vacuum for 240min. Adsorption/desorption cycles showing water-toluene kinetics (left) and resulting isotherms (right).

High temperature preheater for *in-situ* degassing/ activation
Maximum temperature: 400°C
Temperature sensors: Pt-100

Gas /vapor injection system (Upstream control)
Injection system consists of mass flow controllers (upstream) which can deliver vapors and gases of desired flow rates.
Number of Mass flow controllers: 2
Full scale: 200 sccm
Adsorptive species: organic solvents i.e. water, toluene, methanol, ethanol, benzene, acetaldehyde
Gases: CO_2, N_2, 2000 ppm NH_4, H_2S

Butterfly valve (downstream control)
The butterfly valve regulates the amount of vapors or gases in the system by opening and closing depending on the pressure inside the chamber, while total pressure is kept constant.

DVS Vacuum Schematic



Water vapor and organic vapors
Water vapor is generated up to 90% P/Po in the temperature range between 10 to 70°C. Water vapor can be generated in a limited P/Po range above 70°C up to 150°C. Organic vapor generation is limited to solvents' boiling point temperatures.

Gases
Gas adsorption up to atmospheric pressure can be performed using pure or mixtures of gases.

Vacuum system
Roughing pump: rotary vane pump or dry pump
Ultimate vacuum $1x10^{-3}$ Torr
High vacuum pump: Turbomolecular pump
Ultimate vacuum: $4x10^{-8}$ Torr

Vacuum stand
Manifold: 316 stainless steel
Diaphragm valves orbital welded
Seals: Viton, Kalrez (MFCs), VCR gaskets
Tubing: 1/4 inch stainless steel

Specifications

Temperature
Temperature controlled enclosure
Control range: 10˚C to 70˚C
Temperature accuracy: ± 0.2˚C
Enclosure also provides anti-condensation protection.

High Temperature Pre-heater for Outgasssing
400˚C (maximum local temperature)
Temperature sensor: Pt-100

Vacuum Stand
Material: 316 stainless steel
Seals: Viton® and Kalrez®
Tubing: 1/4 inch stainless steel

Solvent Reservoir
250ml side arm flask as standard
100ml side arm flask optional

Flow Control
High accuracy mass flow controllers
Flow rate: 0 to 200sccm
Resolution: 0.1sccm

Mass Measurement
Ultrabalance Low Mass
Sample mass: between 1 and 1000mg
Mass change: ±150mg
Resolution: 0.01 µg
Root mean square balance peak noise: ≤ 0.3 µg[1]

Ultrabalance High Mass
Sample mass: between 10 and 5000mg
Mass change: ±1000mg
Resolution: 0.1 µg
Root mean square balance peak noise: ≤ 3 µg[1]

Pressure measurement
1000 Torr Baratron full scale from 0.1 up to 1000 Torr (standard)
10 Torr Baratron: full scale from 0.005 up to 10 Torr (standard)
Other ranges available upon request (100 and 500 Torr)
Accuracy: ±0.5 of Reading
Resolution: 0.01% of full scale

Vacuum pressure measurement
Vacuum pressure transducer: full scale from $1x10^8$ Torr up to 760 Torr

System Information

Dimensions:	120cm(H) x 58.5cm (W) x 65.5cm (D) 47.2" (H) x 23" (W) x 25.8" (D)
Weight:	150kg (330.7 lb)
Electrical:	200-240 V, 50/60 Hz, 3000 VA

System Software
DVS Vacuum Software

- In-situ sample outgassing/ drying
- Real-time plot of experimental data
- Balance tare and calibration wizard
- Multiple-method protocols incorporating sample outgassing, vapor sorption/desorption cycles
- Dual vapor/gas or vapor-gas co-adsorption
- Ramp or step changes in relative pressures
- Mass equilibrium points at each relative pressure step can be reached by using a fixed time or user defined dm/dt criterion
- Gas adsorption/desorption up to 1bar
- Isotherm and isobar experiments
- Experiments can be performed in half, full or multiple cycles

Analysis Software

- Isotherm calculation
- Permeability and diffusion
- Kinetics information
- Specific Surface Area
- Plotting of experimental data in real time
- Vapor pressure Analysis module
- Heat of Adsorption

Software Options

Standard

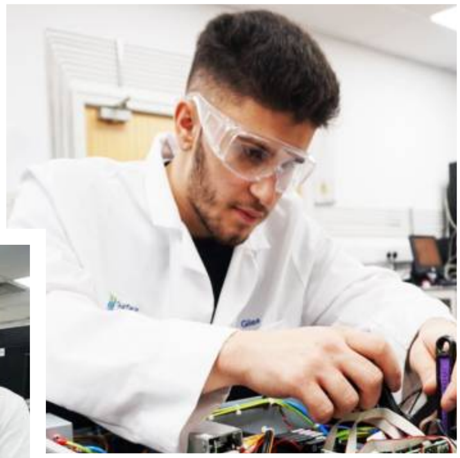
- Control Software
- Standard Analysis

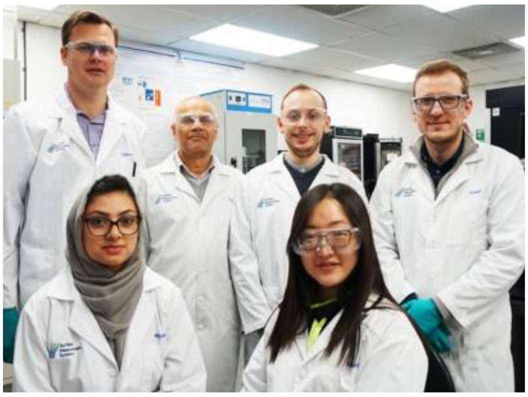
Advanced

- Advanced Analysis Suite
- Isotherm Analysis Suite

Footnotes
[1] Root mean square (averaged over 24 hours)

About Us





Surface Measurement Systems Ltd. develops and engineers innovative experimental techniques and instrumentation for physico-chemical characterization of complex solids. Our range of characterization instruments and scientific/engineering techniques has helped solve difficult problems in the pharmaceutical, biomaterial, polymer, catalyst, chemical, cosmetic and food industries, and are used by hundreds of leading laboratories and universities throughout the world.

Why us?

- Invented the DVS Technology with over 25 years of continuous innovation
- Every instrument is built upon the knowledge and experience of our industry leading sorption scientists
- Our service team provides uncompromising support to our customers and partners
- Outstanding instrument performance
- Most complete and intuitive Windows™ software for experimental control and analysis
- Winner of Innovation Award 2018 and ISO 9001:2015 Compliance

Surface Measurement Systems
World Leader in Sorption Science

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version 1.0
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SCAN ME

DVS
ENDEAVOUR

Surface Measurement Systems
World Leader in Sorption Science

The World's only Simultaneous 5X Parallel Gravimetric Dynamic Vapor Sorption Analyzer

Capabilities:

- 5 balance simultaneous parallel sorption analysis for high throughput/high fidelity data
- Real-time mass measurement kinetics
- Isolated individual sample chambers
- Water and organic vapor sorption kinetics
- Patented Speed of Sound vapor sensor measurement technology
- Co-adsorption with two vapors
- Water vapor sorption isotherms from 10 to 70 °C
- Organic vapor sorption isotherms from 10 to 70 °C
- Optional single channel 200x color video microscopy
- True0™ drying at 0.0% RH

DVS Endeavour

Key Measurement Capabilities

- Real-time simultaneous parallel mass measurements and sorption kinetics for up to 5 samples
- Individual sample chamber isolation preventing crossover contamination
- Sample masses from 1 mg to 5000 mg
- Water and organic vapor sorption kinetics and control using patented Speed of Sound vapor sensor measurement technology
- Multiple sorption/desorption, temperature and sample drying or activation cycles

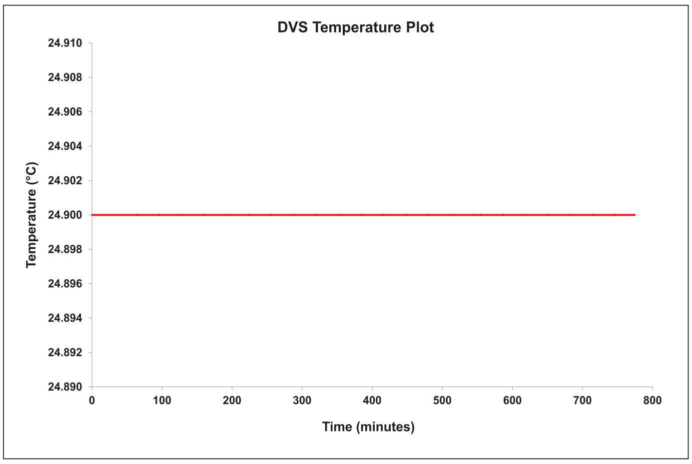
Key Hardware/Software Benefits

- 5 Individual balances operating in parallel
- Patented Speed of Sound vapor sensor measurement technology
- Open stainless steel stand design allows easy access to sample pans while minimizing static electric charging
- Broad temperature range (10-70 °C) from a single uniform and accurate temperature enclosure
- Next generation control and analysis software for the most advanced experimental design and data evaluation

Why Surface Measurement Systems?

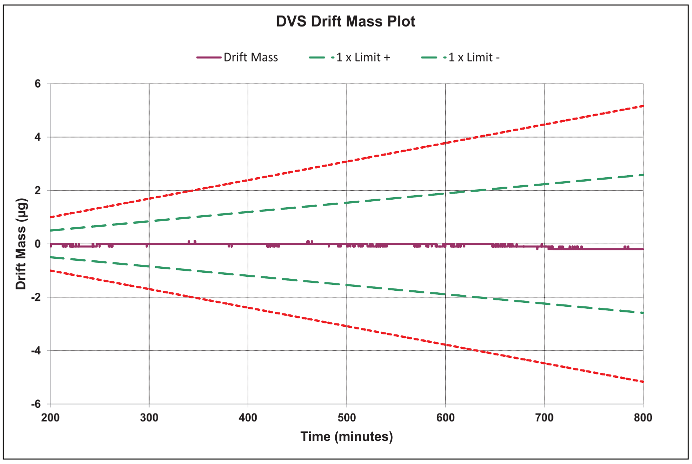
- We invented the DVS technology and continue to innovate in our world leading sorption instruments
- Our industry leading sorption scientists stand behind every instrument
- Our service team provides uncompromising support to our customers and partners

DVS Endeavour
Outstanding Performance



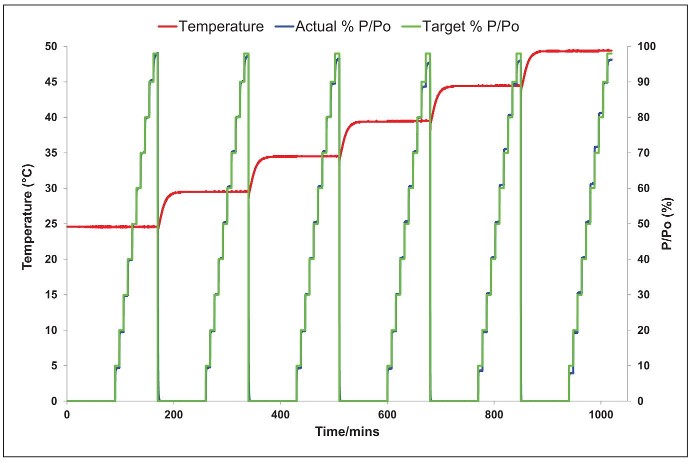
Long Term Temperature Stability

- Typical stability at 25 °C of ±0.02 °C
- Vapor generation and delivery at sample temperature prevents condensation issues found in instruments with multiple temperature zones
- Allows for accurate and stable vapor generation and delivery: a 1 °C temperature variation can alter humidity up to 4% at 25 °C.



Balance Sensitivity and Stability

- Typical balance sensitivity and baseline stability over 10 hours shown at right
- Mass resolution of 0.1 µg
- Root mean square noise of ≤ 0.3 µg
- Samples mass of 1-1000 mg
- Large mass sample option up to 5 grams

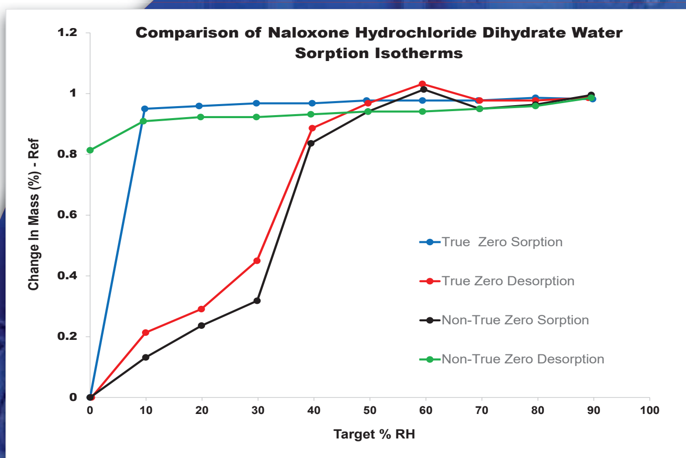


Humidity Performance

- Precision of ±0.1% RH of target humidity
- Highest level of humidity precision in any instrument in its class
- RH and Speed of Sound Probe options

True0™ RH

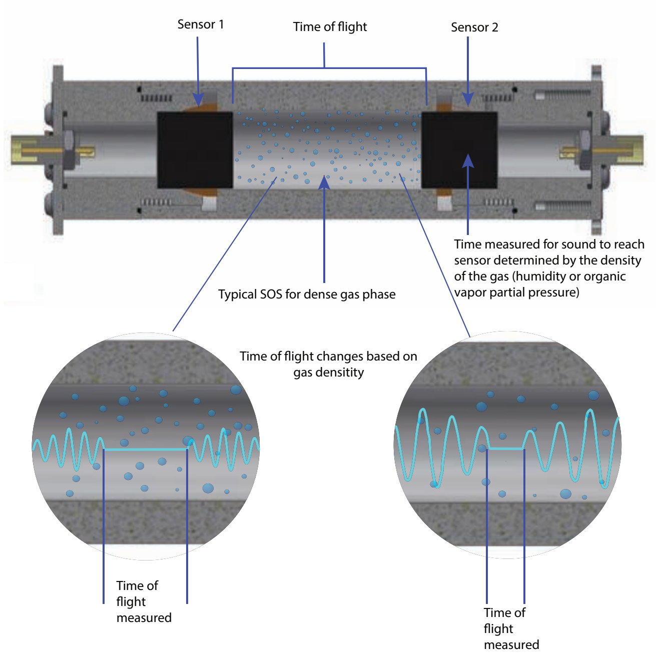
- Only DVS instrument offering True0™ RH
- Achieve partial pressure of water as low as 0.0% RH
- Hydration and dehydration kinetics below 1% RH can be readily studied
- Measurement of sorption/desorption isotherms at low RH or organic vapor concentration



The Only Technique to Directly Measure Solvent Concentration

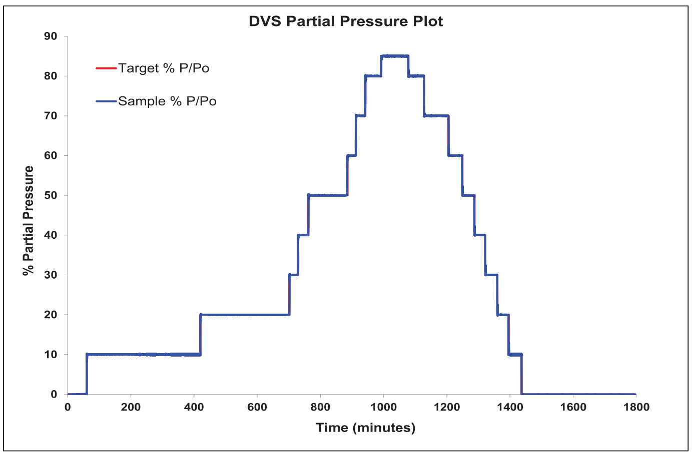
New Speed of Sound Vapor Sensor Measurement Technology

Previous methods for directly measuring solvent concentration have either been non-existent (i.e. predicting the solvent concentration) or in the case of the DVS Advantage, used a specially configured dew point analyser for the measurement. Increased customer demand for a more universal and accurate measurement technology has resulted in SMS developing a revolutionary new sensor. The patented Speed of Sound vapor sensor measurement technology in the DVS Endeavour measures directly the vapor concentration using the speed of sound (SOS). The SOS is directly related to temperature, the chemical species present, and the absolute vapor concentration of the gas phase species. This technology allows for true closed loop control by adjusting the mass flow controlled vapor delivery to the sample using the real time speed of sound measurement. Not only is the SOS measurement fast (1s), it is reliable and a fundamental gas phase property.



High Precision Organic P/P_o Delivery

- Unrivalled partial pressure concentration precision
- Closed loop experiment P/Po control



The Only Method to Directly Measure Solvent Concentration

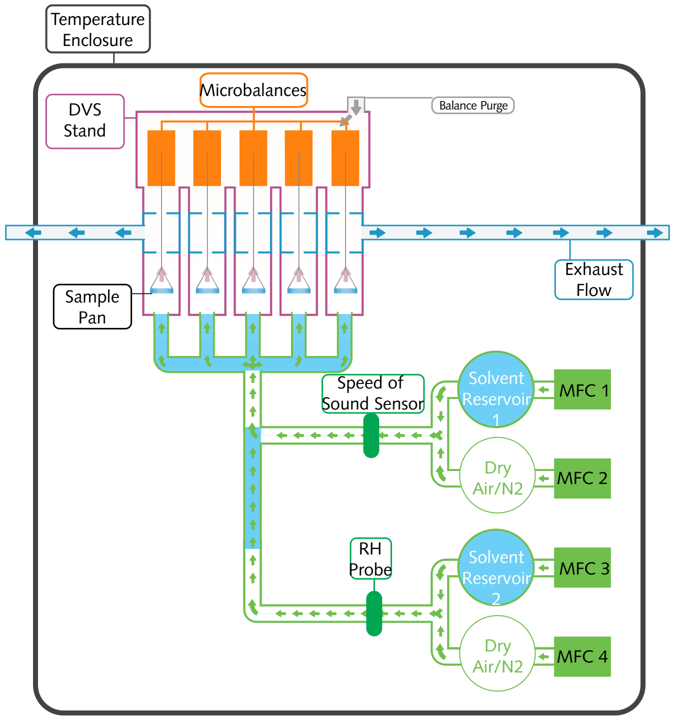
- Speed of sound is an intrinsic property of the vapor or gas measured. Calculates solvent concentration based on the speed of sound travelling through a fixed and known path length of solvent vapor or gas at a known temperature
- Dual Speed of Sound sensors available for complex organic solvent control or solvent co-adsorption measurements
- Automated path-length self-calibration prior to each experiment (1 second calibration time)

Modular Capabilities of DVS Endeavour

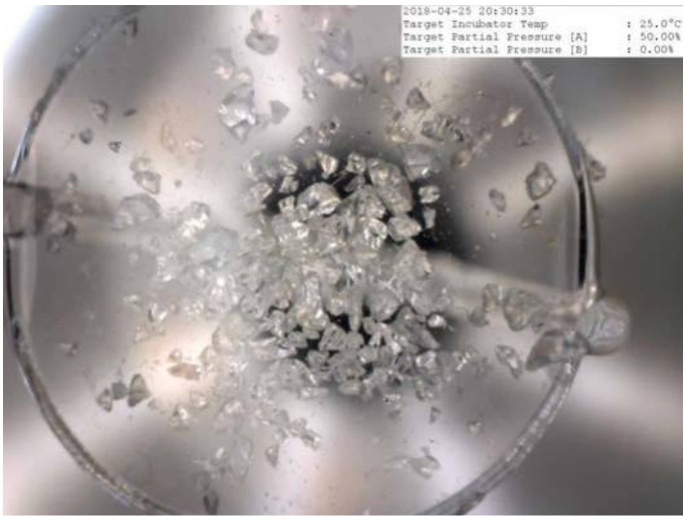
Solvent Delivery Configurations

Using the Speed of Sound Sensor, the DVS Endeavour measures and controls combinations of:

- Humidity
- Organic vapors
- Gases
- Two component systems
- Organic vapor with background humidity



DVS Endeavour Schematic



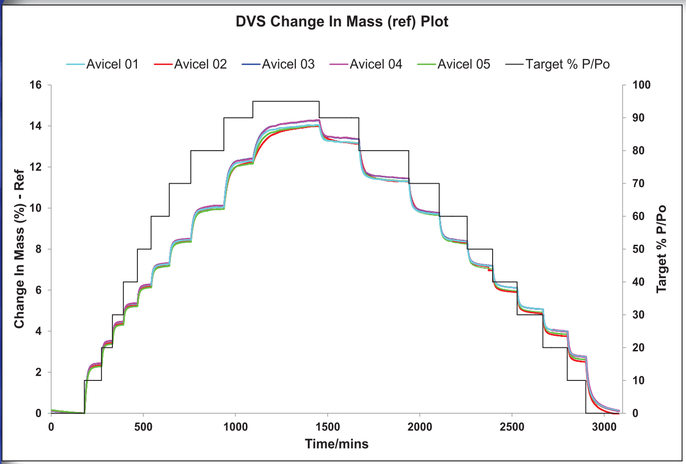
Optional Microscopy and Video

- Optional 1.3 megapixel color camera
- 200x resolution
- Images are time-date-temperature-partial pressure stamped
- Grid overlay and calibration for measuring dimensional change

High Temperature Preheater for Drying, Curing and Humidity Generation at Elevated Temperatures

- *In-situ* degassing/activation of samples up to 200 °C
- The temperature is measured by a Pt100 resistance thermometer directly below the sample pan

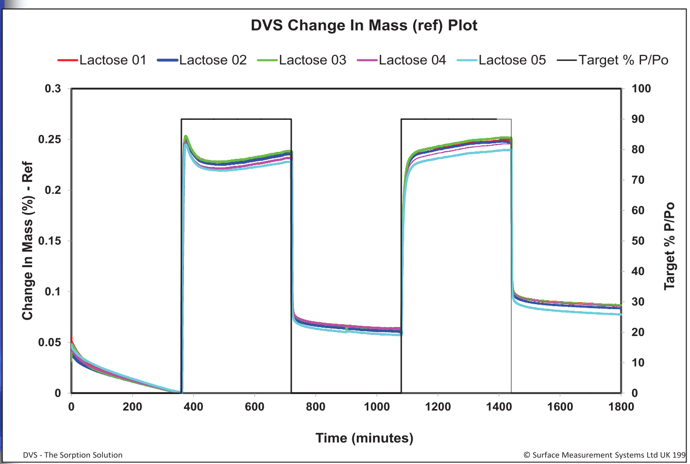
Applications of DVS Endeavour



Moisture Sorption/Desorption Variation Within the Same Lot of Crystalline Material

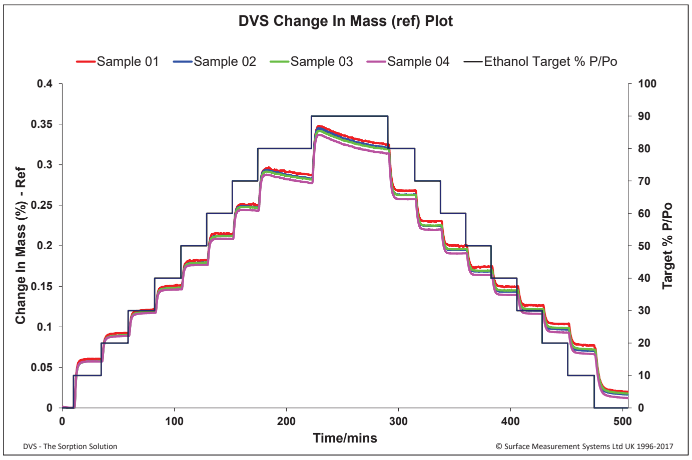
Only true high-throughput gravimetric dynamic vapor sorption kinetics for 5x samples in parallel. Multiple-method protocols incorporating:

- Isotherms and ramped humidity/organic vapor and experiments
- Vapor sorption, vapor exchange and dual vapor co-adsorption
- Experiments may include half, full or multiple partial pressure or temperature cycles
- Most versatile, yet simple experimental control interface



Lot-to-Lot Moisture Sorption/Desorption Variation of Microcrystalline Cellulose

- Investigation of batch-to-batch variation in raw materials and production technical support
- Ingredient compatibility
- Quality control for excipients, actives and formulations
- Stability studies in the selection of new compounds/candidates
- Large scale formulation studies



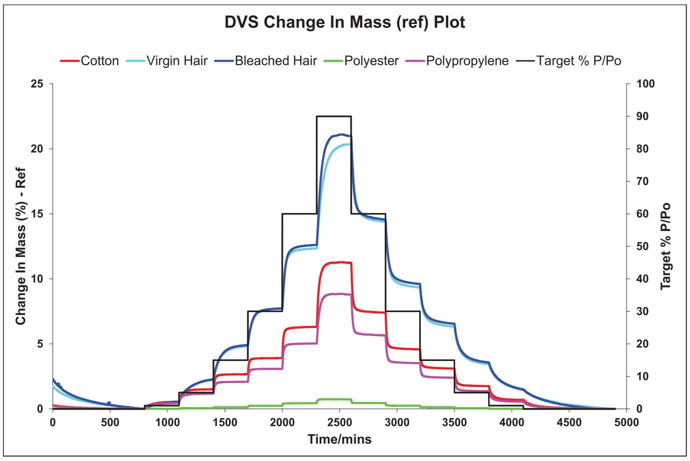
Processing Variations Affect Amorphous Content and Humidity Sorption/Desorption of Milk Powder

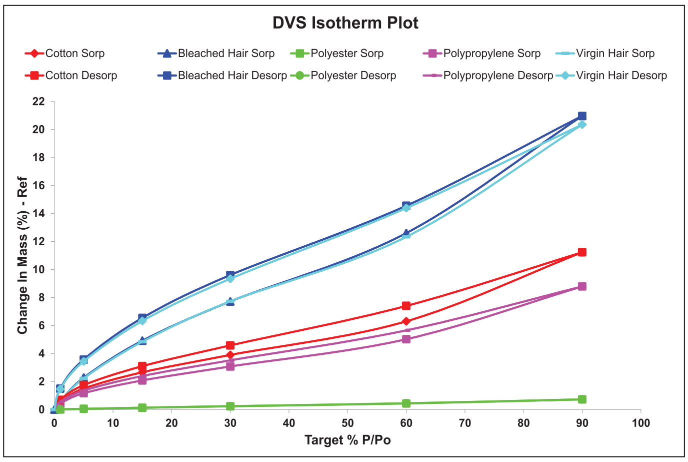
High sensitivity balance, temperature stability, and accurate vapor generation for measurement of rapid and/or small mass changes for process induced surface and bulk properties including:

- Amorphous content
- Hydrate/solvate formation
- Polymorph identification
- Moisture/solvent induced T_g
- Small sample size where only low quantity materials are available

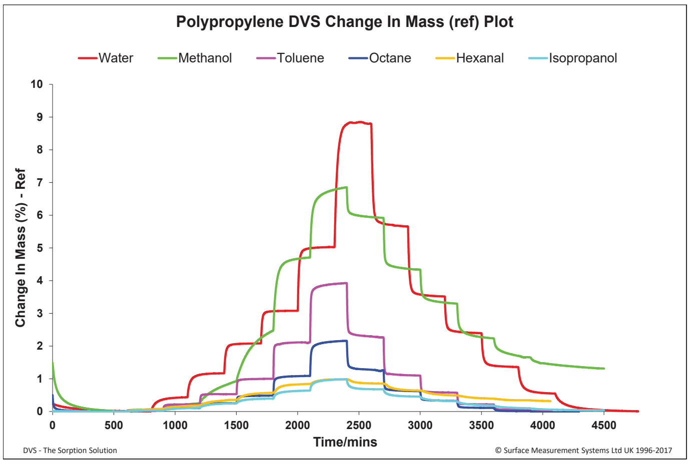
Applications of DVS Endeavour

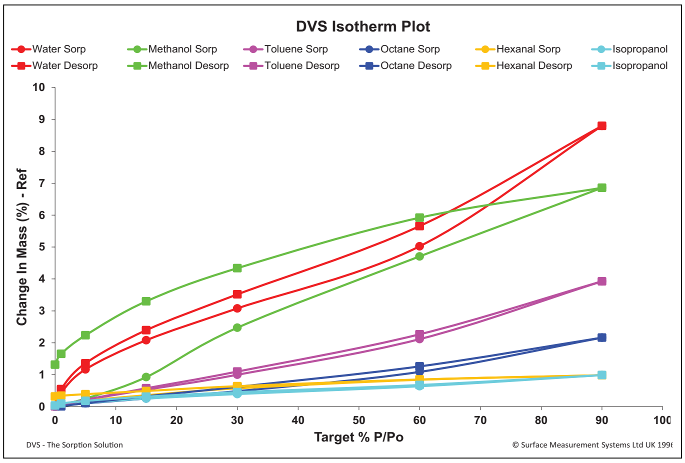
Variation in Fiber Humidity Sorption/Desorption Kinetics and Isotherms



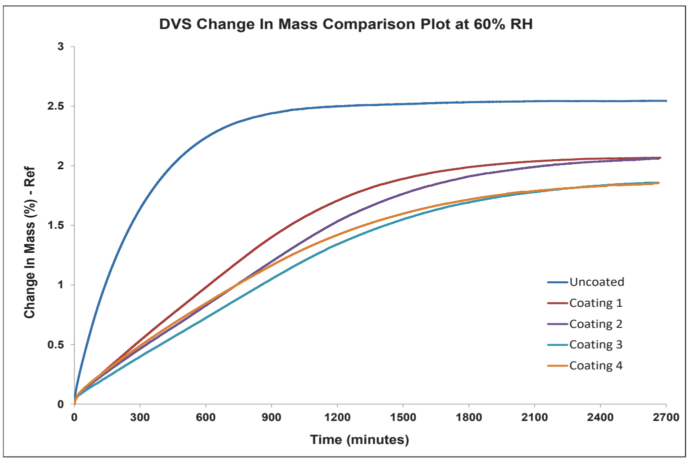


Fiber Sorption Isotherms for Moisture and a Series of Organic Vapors





Film Diffusion Coefficient for Moisture Barrier Coatings



Sample	Weight Gain 40° C/75% RH	Moisture (%)	Δ in Mass (%)	Average Coating Thickness (μm)	Diffusion Coefficient (cm^2/sec)
Uncoated	0.67	4.56	2.50	37.15	3.63×10^{-10}
Coating 1	0.68	4.30	1.90	33.61	1.14×10^{-10}
Coating 2	0.80	4.37	1.75	27.66	7.02×10^{-11}
Coating 3	0.67	4.34	1.55	33.79	6.50×10^{-11}
Coating 4	0.58	4.31	1.60	38.31	1.22×10^{-10}

Surface Measurement Systems

World Leader in Sorption Science

Temperature

Temperature controlled enclosure

Control range: 10 °C to 70 °C
Regulation precision: ± 0.1 °C
Enclosure also provides anti-condensation protection

High temperature pre-heater for drying samples up to 200 °C

200 °C (maximum local temperature)
Heating ramp rates: up to 10 °C/min
Temperature Sensor: Pt-100

Endeavour Stand

Manifold: 316 stainless steel
Seals: Viton
Tubing: 1/4 inch stainless steel

Solvent Reservoirs

Material: Pyrex glass
0.25 and 0.5L as standard
2 Reservoirs included

Relative Humidity Generation and Measurement

Flow control

High accurate digital mass flow controllers

Relative Humidity Sensor

Capacitance probe
Carrier Gas - Dry air or Nitrogen
Relative humidity range from 0 to 98%*
RH range accuracy from 10 - 60 °C ±0.5%*
RH range accuracy from 60 - 70 °C ±1%*

Organic Vapor Generation and Measurement Speed of Sound Sensor

Partial pressure range from 0 to 90% ¤ see note
P/P_0 range accuracy from 10 - 50 °C ±1% ¤¤ see note

Speed of Sound Sensor for organic vapors
Real time partial pressure measurement and control

Solvents Available include:

Water	Toluene	Dichloromethane
Pentane	Acetone	Isopropanol
Heptane	Chloroform	
Hexane	o-Xylene	
Octane	m-Xylene	
Nonane	p-Xylene	
Decane	Benzyl Acetate	
Cyclohexane	Ethyl Acetate	
Methanol	d-Limonene	
Ethanol	Tetrahydrofuran	
Phenol	Dioxane	

Instrument Platform

System Software

Next generation purpose built analysis and evaluation software for the most advanced experimental design and data control.

DVS Control Software

Multiple-method protocols incorporating:

- Sample pre-heating
- Vapor sorption
- Vapor exchange
- Dual vapor co-adsorption
- Temperature changes in a single experiment
- Ramp or step changes in relative humidity
- Automated video image acquisition
- Organic vapor sorption partial pressure
- Temperature stages may be based on fixed-time or user-defined dm/dt criteria
- Complex isotherm experiments
- Experiments may include half, full or multiple partial pressure or temperature cycles

DVS Analysis Software

- Isotherms
- Permeability and diffusion
- Kinetics information
- Surface area models
- Amorphous content
- Heat of sorption
- T_g determinations

Software Options

Standard

- DVS Control Software
- DVS Standard Analysis

Advanced

- DVS Advanced Analysis Suite
- DVS Isotherm Analysis Suite

21CFR Part 11 software solution is available for all DVS software upon request

Mass Measurement

Ultrabalance Low Mass

Low Mass SMS microbalance
Sample mass: between 1 and 1000 mg
Mass change: ±150 mg
Resolution (precision): 0.1 µg*
Root mean square balance noise: ≤ 0.3 µg*

Ultrabalance High Mass

High Mass SMS microbalance
Sample mass: between 10 and 5000 mg
Mass change: ±1000 mg
Resolution (precision): 1 µg*
Root mean square balance noise: ≤ 3 µg*

*For a current list of calibrated solvents or to request a solvent be calibrated contact sales@surfacemeausurementsystems.com

The instrument is factory calibrated at 25°C including salts calibrations. Calibrations at different temperatures including salts available upon request.
Speed of Sound sensor is calibrated for solvents listed in Solvents Available upon request.

Additonal solvent calibrations available upon request.

¤ depending on the solvent selected and temperature of the incubator
¤¤ depending on the solvent selected

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www.SurfaceMeasurementSystems.com

Surface Measurement Systems
World Leader in Sorption Science

DVS
Intrinsic PLUS

Surface Measurement Systems
World Leader in Sorption Science

Dynamic Gravimetric Vapor Sorption Analyzer

The smallest automated DVS for complex water sorption challenges

D V S I n t r i n s i c PLUS

The easy-to-use solution to complex water sorption challenges from Surface Measurement Systems:

- High quality water isotherms and efficient water activity measurements
- Step-by-step software wizards guide users through routine procedures
- Smallest, compact design that makes optimal use of limited bench space - only 26cm wide
- Advanced electronics and simplified user interface
- Accommodates wide variety of sample geometries and up to 5 gram capacity
- SMS UltraBalance™ provides unrivalled sensitivity and baseline stability
- Built-in Network Connectivity for easy data sharing and remote analysis
- Supports 21 CFR part 11

Ultrabalance™

Applications

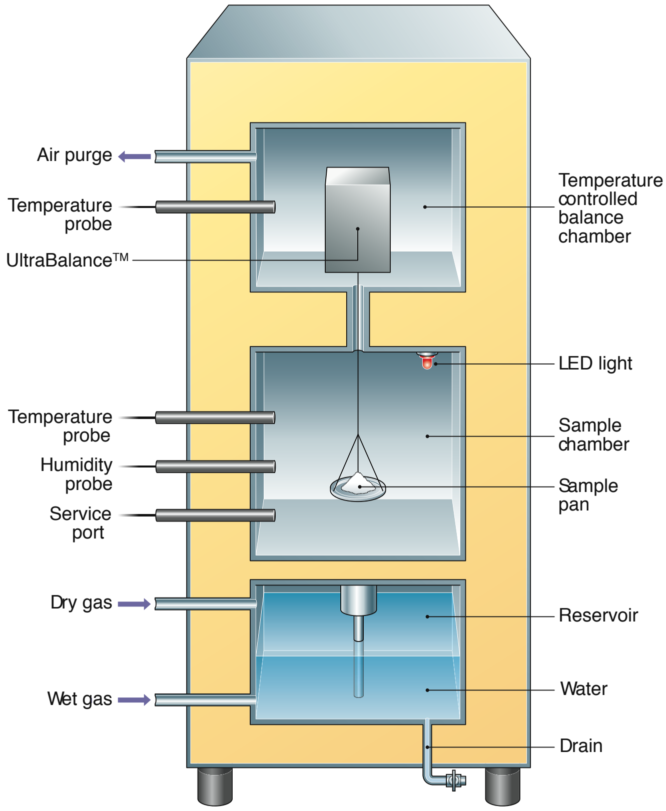
- Studying hygroscopicity of powders, fibers and solids
- Kinetics of water sorption and desorption
- Water induced morphology changes
- Food shelf-life prediction studies
- TEWL/ Transepidermal Water Loss
- MVTR/ Moisture Vapor Transmission Rate determination
- Calculation of Diffusion Coefficients
- Sorption Modeling
- Moisture Compatibility
- Water Activity Measurements

Materials Studied

- Pharmaceuticals: powders, tablets, API's and excipient materials
- Food: powders, processed food, biscuits
- Natural materials: grains/seed, wood, biomass
- Building materials: aggregates, cement, ceramics
- Personal care products: cosmetics, hair care, contact lenses
- Packaging materials: paper, plastics

The benefits of water sorption measurements

The water sorption properties of solid materials are recognized as critical factors in determining their storage, stability, processing and application performance. These properties are routinely determined for many natural and man-made materials and have traditionally been evaluated by storing samples in sealed jars containing saturated salt solutions of established relative humidity and then regularly weighing these samples until equilibrium is reached. The DVS Intrinsic provides a number of advantages over these methods:



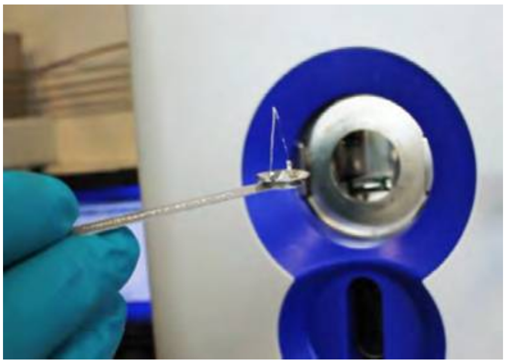
Schematic of the main components of DVS Intrinsic

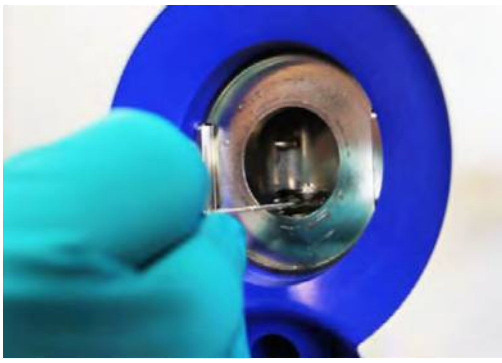
(i) The DVS Intrinsic technique, due to optimized vapor flow, reduces the time required to reach equilibrium, measured in minutes rather than days. Simultaneously the dynamic flow of moisture reduces the need for large sample sizes, requiring only a few milligrams of sample.

(ii) Due to the dynamic flow of vapor, the sample never needs to be removed from the instrument, eliminating errors and contamination associated with removing samples from storage containers in previous methods.

(iii) The DVS Intrinsic allows for kinetic water sorption/desorption data to be collected in real time, which is impossible in static methods.

(iv) The DVS Intrinsic technique reduces labor and operational costs by allowing skilled scientists and technicians to be more productive.



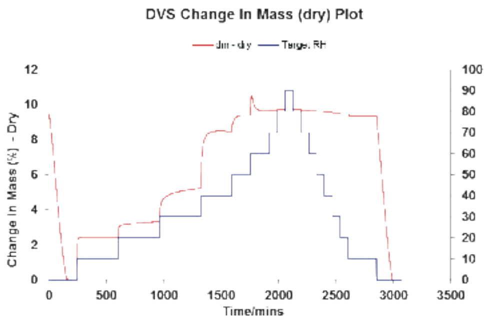


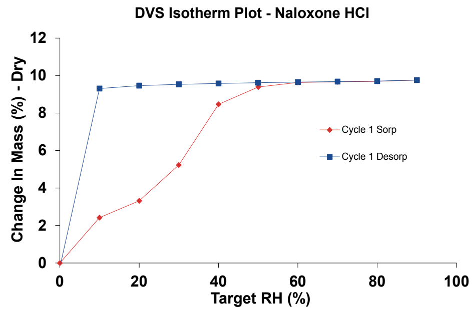
DVS Intrinsic sample metal pan

"The DVS Intrinsic Plus is a highly sensitive, accurate, and fast analyzer for the automated determination of moisture sorption properties of solids"

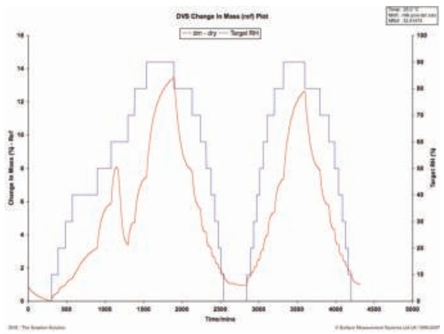
Applications

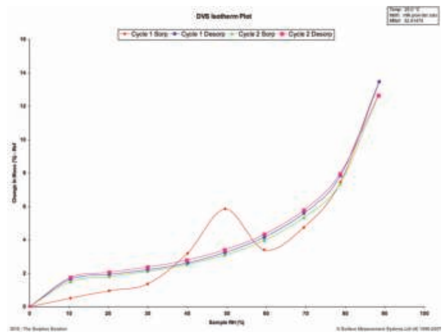
API hydrate formation and loss



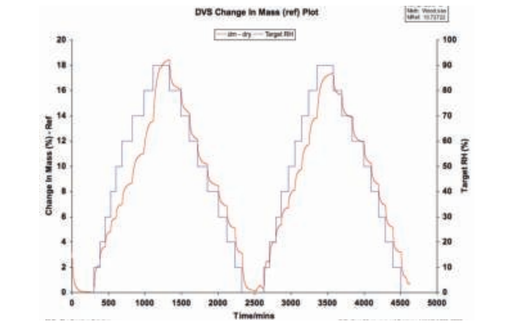


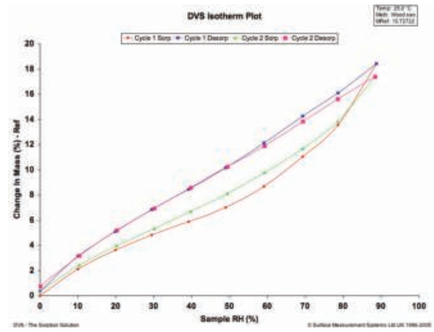
Amorphous lactose recrystallization



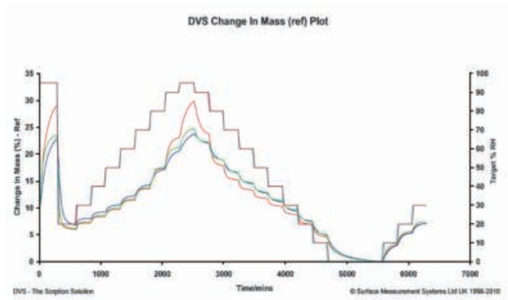


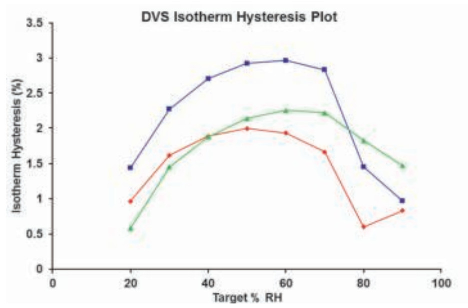
Wood fiber, two sorption cycles



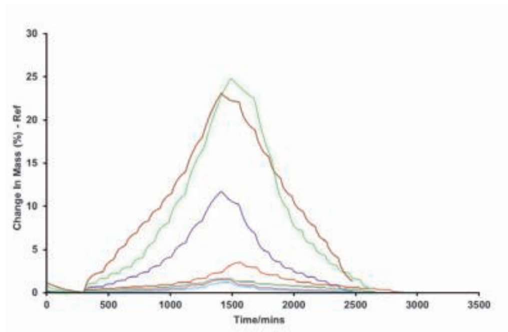


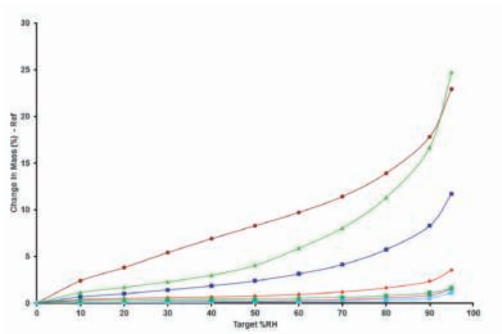
Hair with treatment comparison



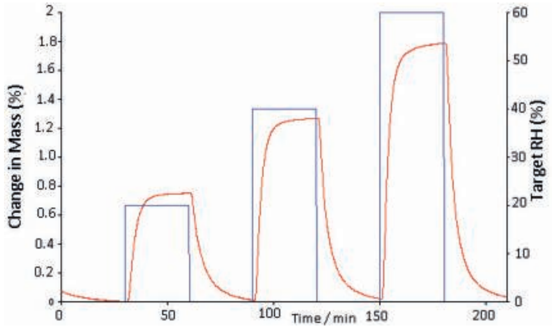


Building materials sorption compatibility

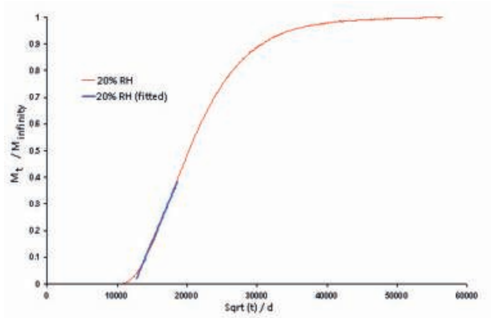




Moisture Diffusion/Permeation



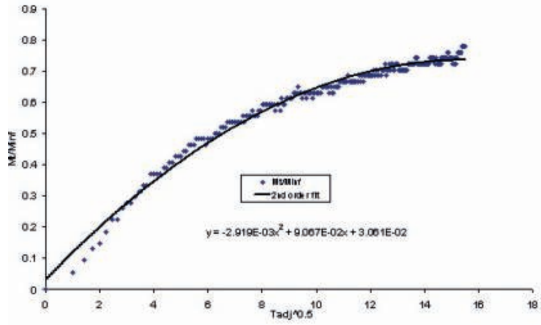
Sorption and desorption kinetics for a 7.5um polyimide film.



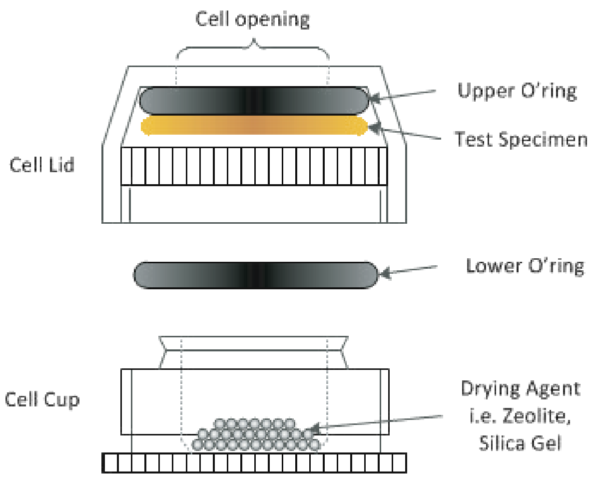
Diffusion plot for 0% RH to 20% RH step in humidity for a 7.5µm polyimide film.

Previous RH (%)	Target RH (%)	Diffusion Coeff. (cm2/s)	R-squared (%)
0.0	20.0	7.63E-10	99.55
20.0	0.0	4.38E-10	99.58
0.0	40.0	9.04E-10	99.52
40.0	0.0	6.05E-10	99.59
0.0	60.0	9.30E-10	99.54
60.0	0.0	6.55E-10	99.57

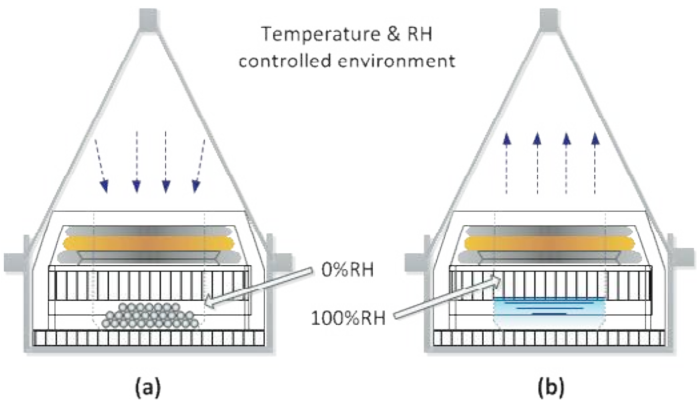
Diffusion coefficients from initial slopes.



Polynomial fit for particle diffusion calculation. Moisture diffusion coefficient for an amorphous pharmaceutical powder at 25C and 40%RH with σ = 0.11 x 10^{-11} cm^2/s.

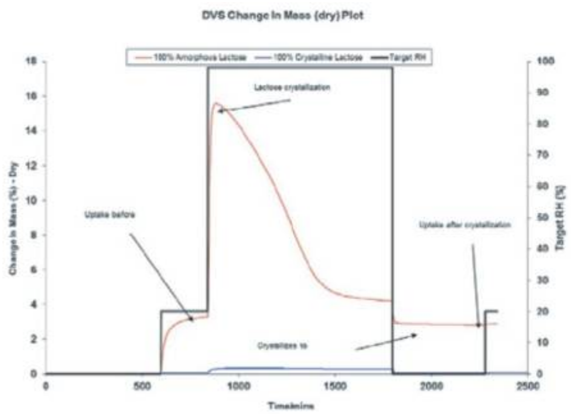


Experimental set-up for moisture vapour transmission rate measurement.

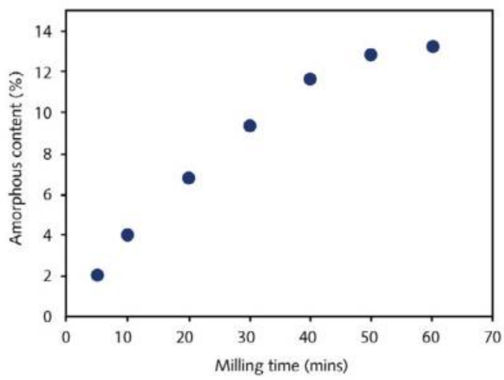


Payne type diffusion cell with DVS metal sample pan (C-WM-017) for (a) dry cup method and (b) wet cup method.

Amorphous Content



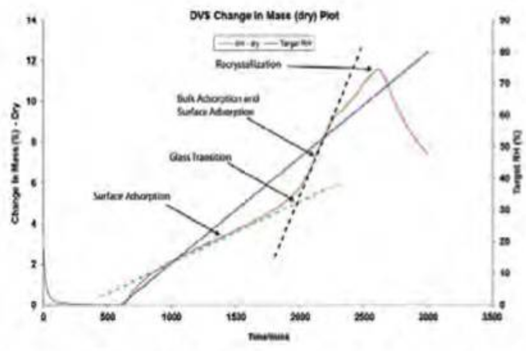
Amorphous content mass change and crystallization due to moisture.



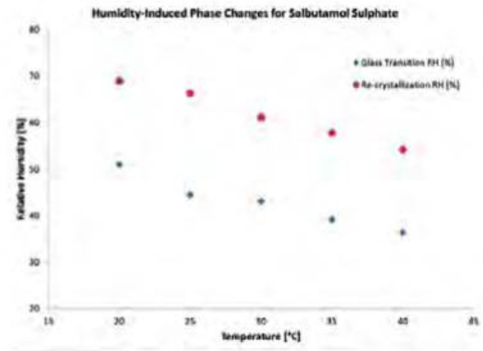
Influence of milling time on amorphous content of 100% crystalline lactose using DVS.

Applications Continued

Moisture Induced Glass Transition



Moisture induced glass transition and crystallization of salbutamol sulphate.



Glass Transition and crystallization RH (%) for salbutamol sulphate.

Technical Specifications

Temperature

Temperature controlled enclosure

Control range: 20 °C to 40 °C

Temperature accuracy: ± 0.2 °C

Flow Control

High accuracy digital mass flow controllers

Wide dynamic range - turndown ratio 1000:1

Carrier Gas: Dry air or Nitrogen

Inlet pressure: 2.5 to 4 bar

Regulated pressure: 2 bar

Flow rate: 0 -200 sccm

Relative Humidity

Relative humidity range from 0% to 98% (±0.5%) RH for 20°C to 40°C [1]

Relative humidity resolution: ± 0.1%

In-line real time Humidity Probe for Relative Humidity measurements and control

Mass Measurement

Ultrabalance Low Mass

Maximum load: 1000 mg

Mass change: ±150 mg

Resolution: 0.01 µg

Balance noise: ≤ 0.3 µg [2]

Ultrabalance High Mass

Maximum load: 5000 mg

Mass change: ±1000 mg

Resolution: 0.1 µg

Balance noise: ≤ 3 µg [2]

Solvent Reservoir

Reservoir capacity: 100ml

System Information

Dimensions:	470 mm (W) x 260 mm (H) x 390 mm (D)
	18.5" x 10.23" x 15.35"
Weight:	22 kg (48.8 lb)
Electrical:	100-240 V, 50/60 Hz, 300W

System Software

DVS Control Software

- Experimental stages may be based on fixed-time or a user-defined dm/dt criteria
- Experiments may include half, full or multiple partial pressure or temperature cycles

DVS Analysis Software

- Isotherms
- Permeability and diffusion
- Kinetics information
- Specific Surface Area
- Amorphous content
- Heat of sorption
- T_g determinations

Software Options

Standard

- Control Software
- Standard Analysis

Advanced

- Advanced Analysis Suite
- Isotherm Analysis Suite

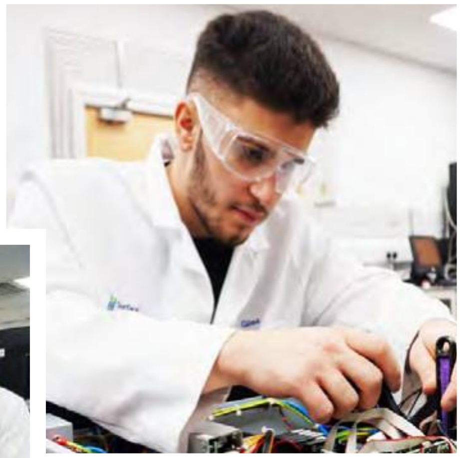
Footnotes

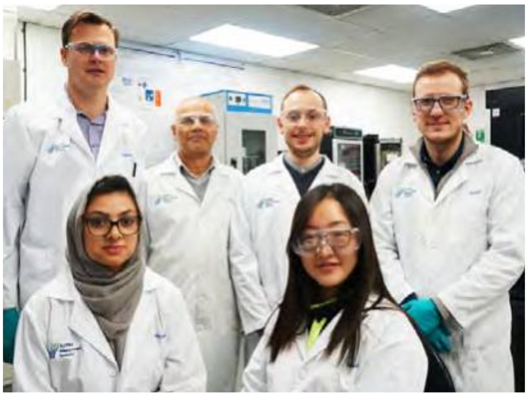
[1] 1-σ confidence level with %RH or P/Po calibration performance based on SMS factory certified methods (Salts Calibrations)

[2] Root mean square (averaged over 24 hours)

21CFR Part 11 software solution (optional)

About Us





Surface Measurement Systems Ltd. develops and engineers innovative experimental techniques and instrumentation for physico-chemical characterization of complex solids. Our range of characterization instruments and scientific/engineering techniques has helped solve difficult problems in the pharmaceutical, biomaterial, polymer, catalyst, chemical, cosmetic and food industries, and are used by hundreds of leading laboratories and universities throughout the world.

Why us?

- Invented the DVS Technology with over 25 years of continuous innovation
- Every instrument is built upon the knowledge and experience of our industry leading sorption scientists
- Our service team provides uncompromising support to our customers and partners
- Outstanding instrument performance
- Most complete and intuitive Windows™ software for experimental control and analysis
- Winner of Innovation Award 2018 and ISO 9001:2015 Compliance

Surface Measurement Systems
World Leader in Sorption Science

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version 1.0
15042020

SCAN ME

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Surface Measurement Systems
World Leader in Sorption Science

Inverse Gas Chromatography Surface Energy Analyzer

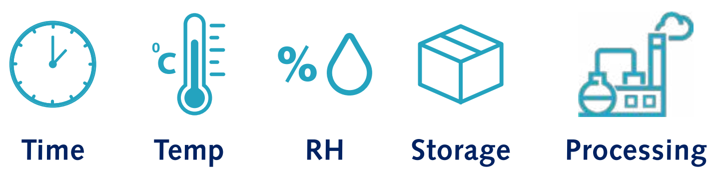
Purpose-built iGC instrumentation for advanced materials characterization

www.surfacemeasurementsystems.com

Surface Energy

Particulate solids, such as powders, fibers, and films, often exhibit issues during manufacturing, usage, and storage in various industrial sectors. To gain an understanding of the key factors that control their behavior and performance, surface energy γ is emerging as one of the most crucial solid material properties.

Discover how your materials are affected by:

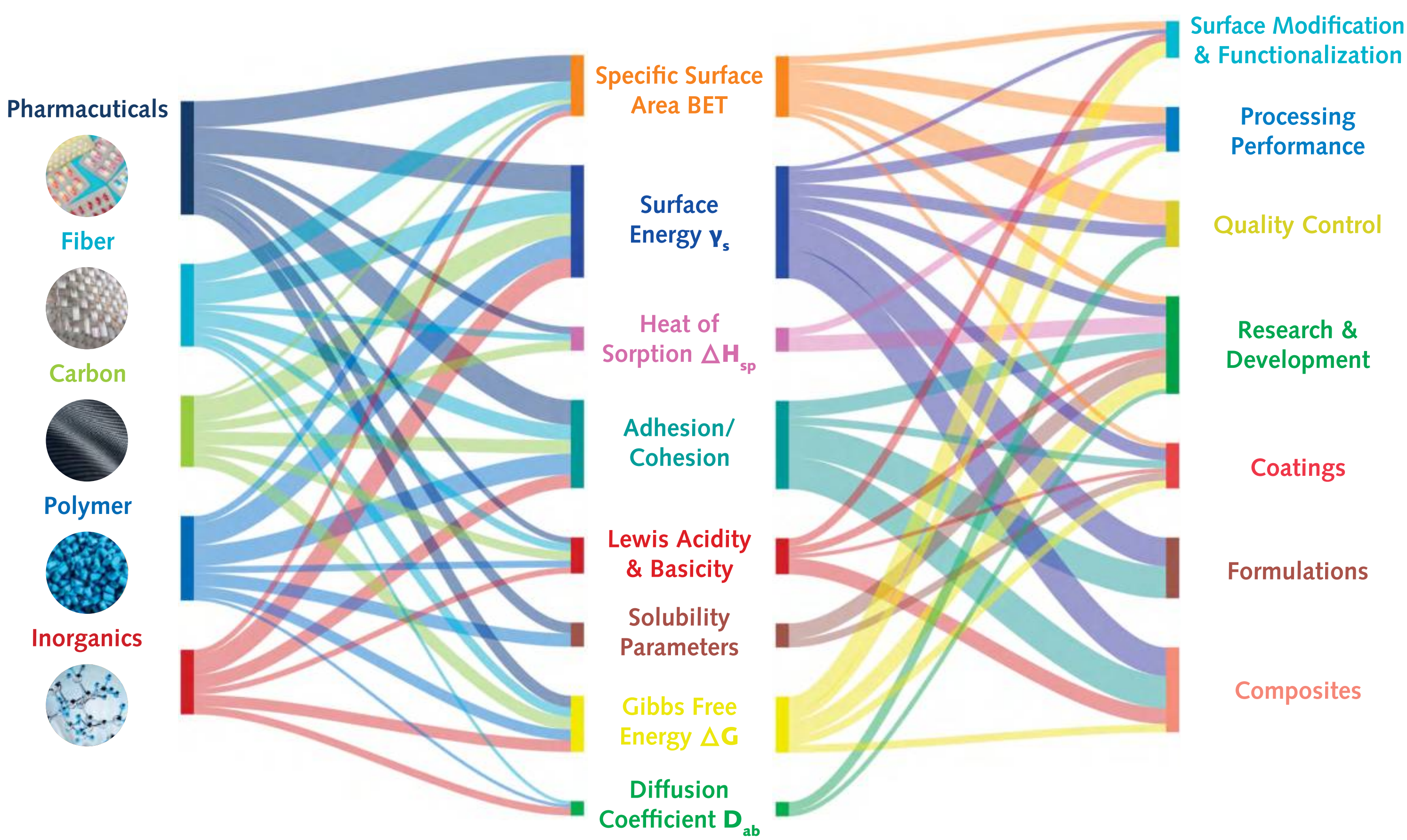


Surface energy γ is a measure of attractive intermolecular forces on a solid surface, similar to the surface tension of a liquid.

These intermolecular forces are responsible for the attraction between powder particles and other solids, liquids, and vapor molecules and can arise from long-range Van der Waals forces (dispersion forces) and shorter-range chemical forces (polar forces).

Surface energy values (dispersive and polar) are linked to several key solid properties, including wetting, dispersibility, powder flowability, agglomeration, process-induced disorder, adhesion/cohesion, static charge, adsorption capacity, and surface chemistry.

But how do we determine the actual surface energy of a specific solid material? This is where the iGC-SEA comes into play. This powerful instrument employs Inverse Gas Chromatography (iGC) to determine Surface Energy with exceptional detail and accuracy via gas phase adsorption.



What is iGC?

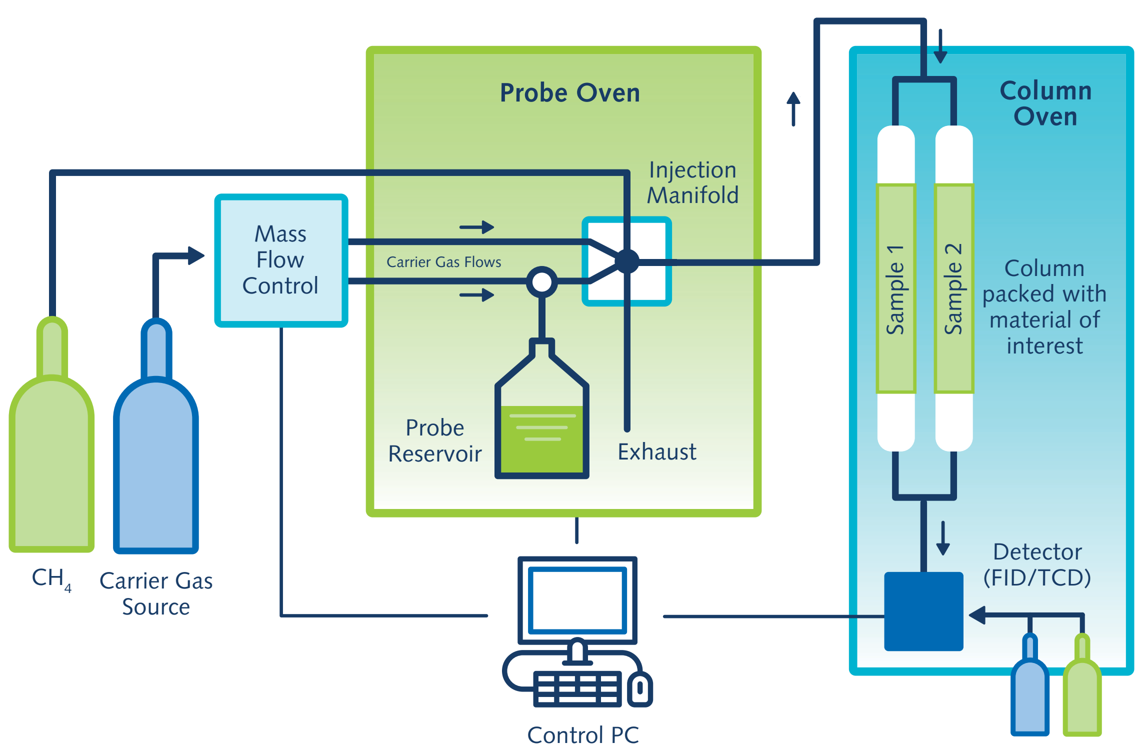
Inverse Gas Chromatography (IGC) is a technique used to characterize the surface and bulk properties of materials by analyzing the interactions between a solid sample and various gases.

What is the **iGC-SEA**?

The iGC-SEA, or Inverse Gas Chromatography-Surface Energy Analyzer, is an instrument that operates on the principles of iGC. Inverse Gas Chromatography is a gas-solid technique used to characterize the surface and bulk properties of solid materials (For more details, see page 10). Due to its reliability and precision, IGC has become the preferred method for surface energy characterization in laboratories worldwide for particulate materials.

At the core of the iGC-SEA's innovation is the patented gas injection manifold system. This system generates accurate solute pulse sizes across a wide concentration range, producing isotherms at both high and low sample surface coverages. enables precise determination of surface energy heterogeneity distributions.

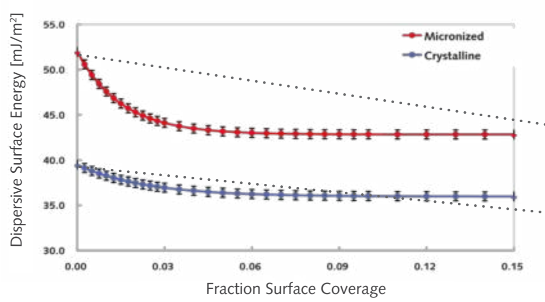
The iGC-SEA offers a humidity control option, allowing the assessment of humidity and temperature's impact on the physicochemical properties of solids, including moisture-induced Tg, BET specific surface area, surface energy, wettability, adhesion, and cohesion. The system can also conduct bulk solid property experiments employing probe-bulk interactions and solubility theory. Equipped with a purpose-built data analysis software suite, the iGC-SEA stands as an unparalleled instrument, providing a broad spectrum of accurate and reliable surface and bulk property measurements.



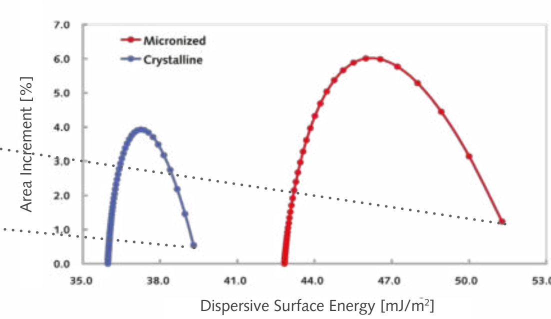
iGC-SEA schematic

Applications

Surface Energy Heterogeneity Profiling



Dispersive Surface Energy **Profiles** for Powder Budesonide

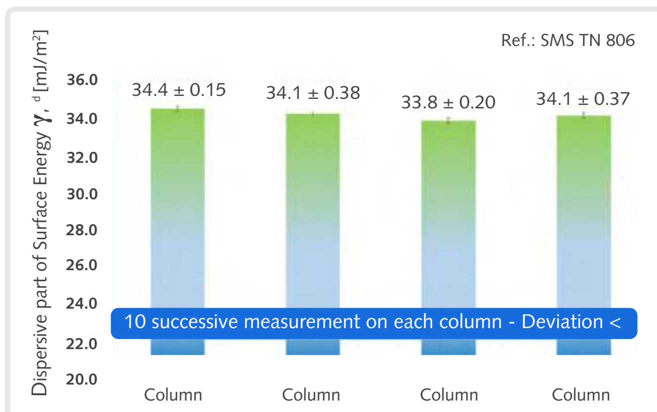


Dispersive Surface Energy **Distributions** for Powder Budesonide Samples

Analyzing surface energy profiles involves determining both dispersive and polar components as a function of the fractional surface coverage of the packed material. The surface energy distribution is the integration of the surface energy profile across the entire range of surface coverages and is analogous in principle to a particle size distribution.

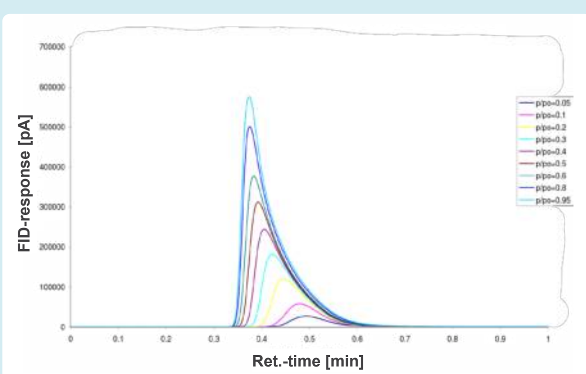
Reliable SE Reproducibility

The unique features of the iGC-SEA instrument enable unparalleled reproducibility in Surface Energy determination. An average standard deviation of less than 0.8 % is observed on 10 successive measurements across 4 columns.

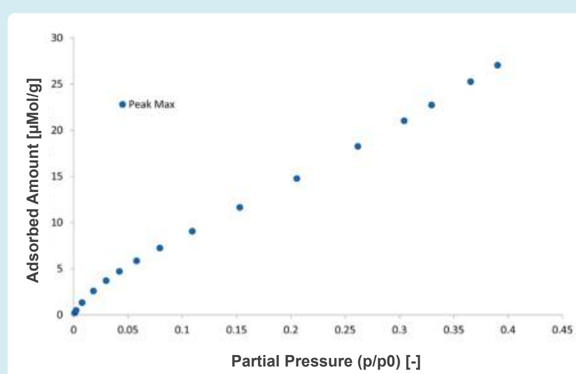


Technical performance of iGC-SEA on commercial Paracetamol (Sigma-Aldrich)

Adsorption Isotherms, Heat of Adsorption and Henry Constants



*Series of pulses for a multiple injection experiment (variable concentration) on *M745 with hexane at 303 K*



*Sorption isotherms of hexane by pulse injections on *M745*

*The M745 is an **α-**alumina, which is used as a certified reference material. Reference: SMS Application Note 208.

Instrument Platform

iGC-SEA Hardware

✓ Patented integrated gas phase injection systems with humidity generation

✓ Sample Volume: 0 - 23.56 cm^3

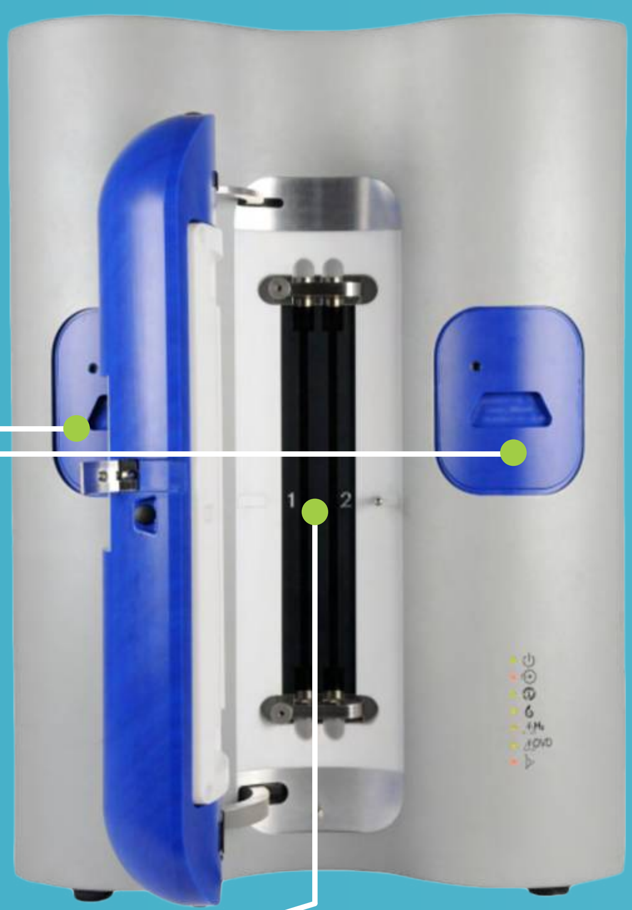
✓ 12 interchangeable reservoirs: Easy access drawers

✓ Sequential twin-sample column design

✓ 50 solvents database built-in-ability to add more

* Temp range dependent on instrument variant

460 mm (W) x 530 mm (D) x 650 mm (H)



✓ Sample column oven: 10 °C to 500 °C *

✓ Flame Ionization Detector (FID): Adjustable gain

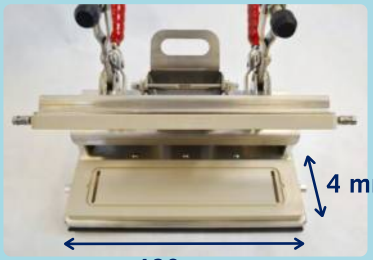
✓ Use of Nitrogen or Helium as carrier gas

✓ Purpose-built and fully integrated iGC

✓ H_2 & Organic Vapor Leak Detector

Sample Columns

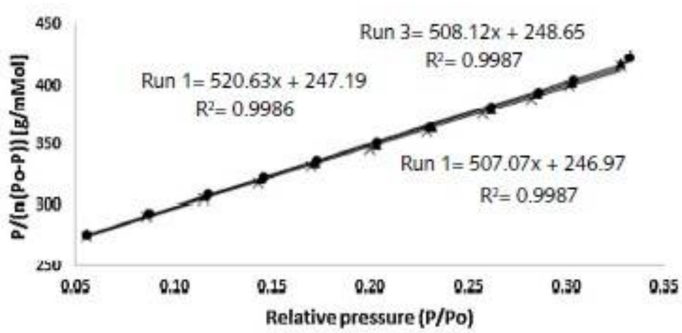
Film Cell Holder



iGC-SEA Advanced

- Analyzes surface energy and specific surface area for fine powders, fibers, and non-porous particulate solids.
- Fully-automated iGC system
- Patented headspace injection system with humidity generator
- User-friendly control & analysis software with CFR 21 Part 11 capabilities

Surface Area Analysis Case Study



*Linearized BET analysis for 3 experiment runs**

Commercial natural fibres studied

Sample	Variety	Fibre processing
Cellulose	BioMid® (ENC International, South Korea)	Dry jet-wet spinning process
Kenaf	KK60 (Thailand)	Water retting
Flax	Linseed flax (Canadian)	Mechanical decortication by scrutching

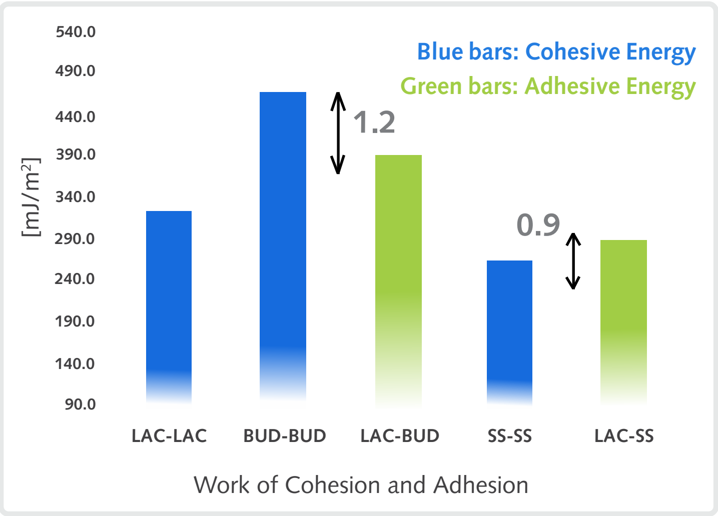
Reproducibility BET experiment.

BET specific surface area (Octane) ($m^2 g^{-1}$) at 30 °C and 0% RH					
Specimen	Run 1	Run 2	Run 3	Mean	Std (%)
BioMid®	0.546	0.545	0.543	0.545	0.1
Kenaf	0.503	0.494	0.501	0.500	0.5
Flax	1.373	1.423	1.440	1.412	3.5

*Reference: "Inverse gas chromatography for natural fiber characterization: Identification of the critical parameters to determine the Brunauer-Emmett-Teller specific surface area" (Journal of Chromatography A, 1425 (2015) 273-279).

Predict Blending Performance for DPI Formulations

The surface energy derived Cohesion-Adhesion Energy can be used to predict blending performance. As shown, the CAB model can effectively predict the interactive powder mixing behavior of small particles along with the compatibility/flow behavior of resultant interactive mixtures at certain excipient proportions.



LAC: Lactose **BUD:** Budesonide **SS:** Salbutamol Sulfate

	W_{coh}	W_{adh}	CAB with lactose
BUD	490	400	$W_{coh} > W_{adh}$
SS	270	290	$W_{coh} \leq W_{adh}$

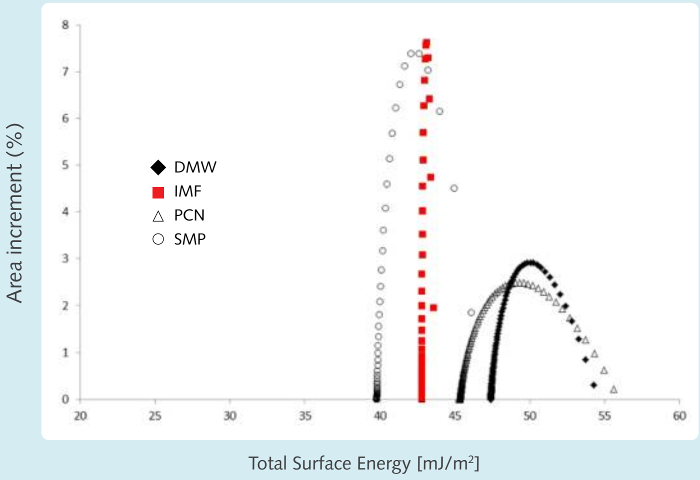
Formulation	Content uniformity RSD (%)
SS+LAC	4.2
BUD+LAC	28.1

(Data by R.Price, Univ. of Bath, UK)

Similar study: "Applying surface energy derived cohesive–adhesive balance model in predicting the mixing, flow, and compaction behavior of interactive mixtures" (European Journal of Pharmaceutics and Biopharmaceutics 104 (2016) 110–116).

Investigating Flow Behavior of Powders

The iGC-SEA is a valuable tool, providing insight on powder flowability using surface energy analysis. More energetically homogeneous powders (narrow energy curves, see graph below) display improved flow behavior. A standard technique for measuring powder flowability corroborates the IGC analysis findings, showing that Infant Milk Formula (■) has better flowability.



*Normalized surface energy distribution of milk powder**

	GEA TEST [s] Mean± SD	Brookfield Flow Tester Flow function 1/slope Mean± SD	Brookfield Flow Tester Classification
DMW	23[a] ± 2.8	4.93[a] ± 0.26	Easy-flowing
IMF	23[a] ± 0.7	10.50[b] ± 1.29	Free-flowing
PCN	103[b] ± 8.5	4.15[a] ± 0.25	Easy-flowing
SMP	21[a] ± 0.7	9.19[b] ± 0.19	Easy-flowing

DMW: Demineralized whey powder, IMF: Infant milk formula powder, PCN: Phosphocasein powder, SMP: Skim milk powder. *GEA Powder Flow Method A23a (1978).*

*Reference: "Relationships between surface energy analysis and functional characteristics of dairy powders" (Food Chemistry 237 (2017) 1155–1162).

iGC-SEA Nova

- New high temperature oven: 30 °C - 500 °C
- *In-situ* preconditioning and surface energy analysis within a single instrument
- Fully-automated iGC system
- Patented head space injection system with humidity generator
- User-friendly control & analysis software with CFR 21 Part 11 capabilities

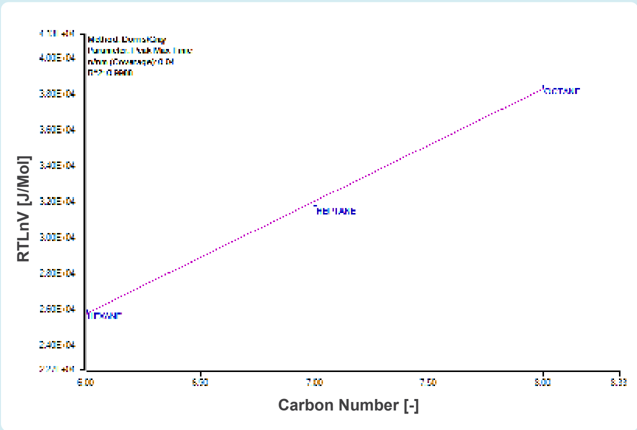
Carbon Black Surface Characterization for Energy Storage

Vulcan XC 72 is a carbon black with high electrical conductivity for diverse applications, such as batteries, fuel cells, conductive paper, and catalyst support. This material provides exceptional conductivity even at low loading levels and plays a crucial role as a support in anode and cathode electrodes, particularly in polymer electrolyte membrane fuel cells (PEMFC). BET SSA: 128.8 m²/g.

Results:

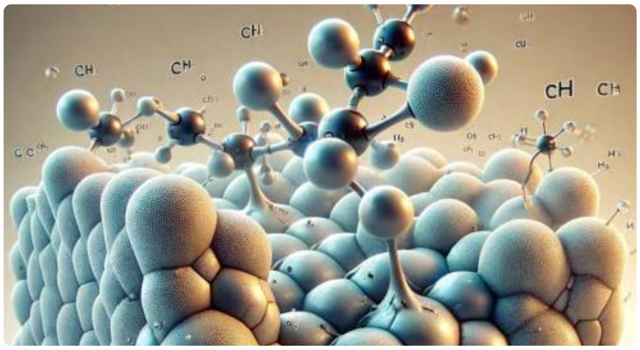
Humidity: 0%RH
Sample mass: 8mg

Experiment Temperature:	523K	573K
Dispersive Surface Energy (γ_d^{50}):	139 mJ/m²	240.1 mJ/m²

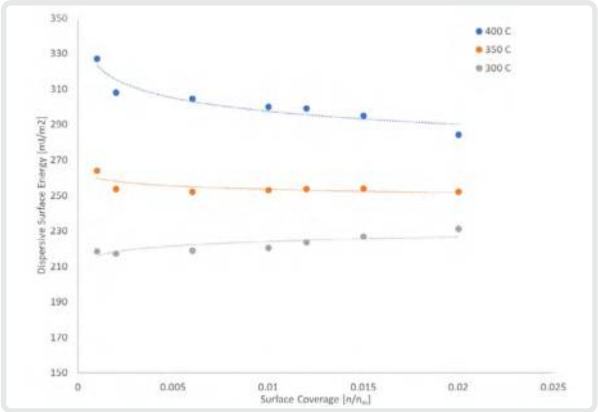


Dorris-Gray Method

Dispersive Surface Energy of Zeolite 13x

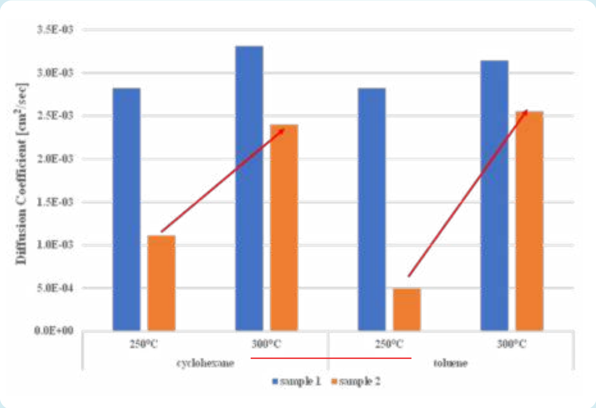


The yd surface energy of Zeolite 13X at 300°C and 350°C reveals homogeneity, with a consistent and uniform surface energy distribution. However, when the temperature rises to 400 °C, some degree of heterogeneity begin to appear.



Dispersive Surface Energy of Zeolite 13x

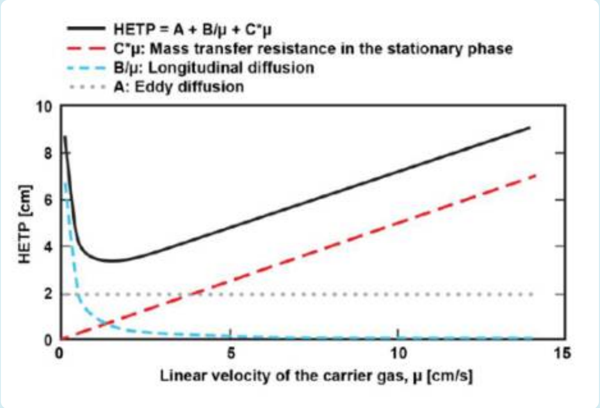
High Temp Diffusion Coefficient



Diffusion of Microporous Catalysts

Diffusion and mass transfer are critical in the design and optimization of catalytic processes, operations processing of materials, and in adsorptive separation. The van Deemter model is a continuation of the plate theory and involves the dynamic response of HETP as a function of the average linear velocity of the carrier gas (see Figure 1), distinguishing the three types of diffusion types: eddy diffusion, longitudinal diffusion, and mass transfer resistance.

Comparing the changes in diffusion coefficients as a function of temperature, the values are higher at 300°C than 250°C for both vapors on both samples. This is expected since higher temperatures lead to faster diffusion rates. When comparing the cyclohexane and toluene results on the same sample, the cyclohexane diffusion coefficients are higher for both samples and temperatures.

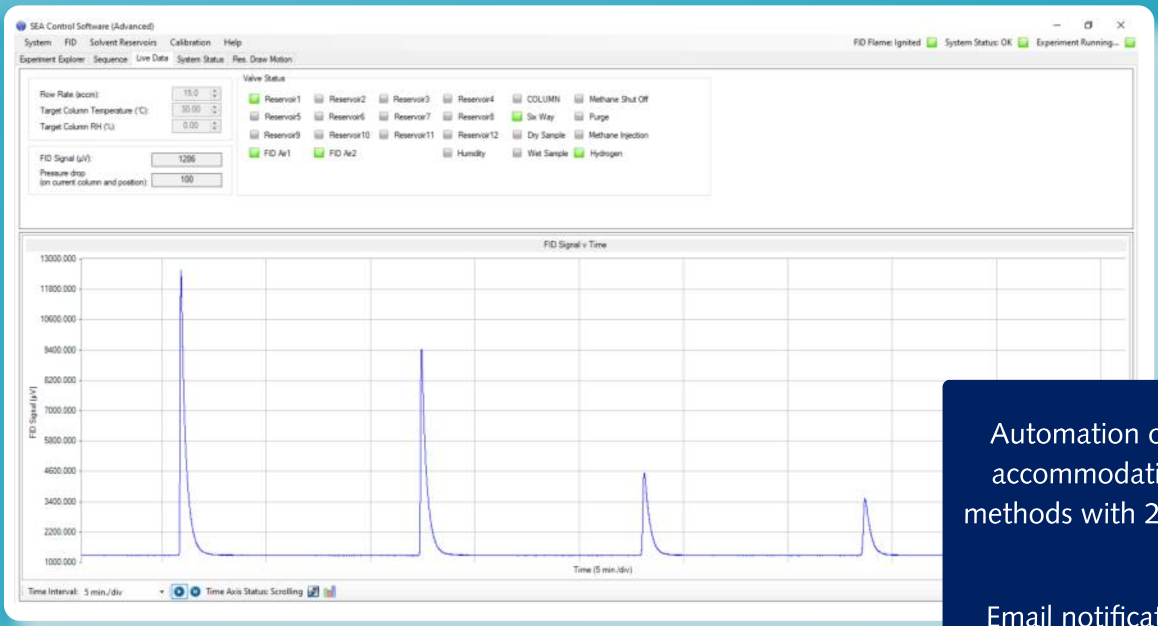


Van Deemter Plot: The 3 Components of the van Deemter Equation

Purpose-Built Software

Our proprietary software includes a seamless system control software and robust data analysis software featuring advanced tools tailored for research-focused users.

Control Software



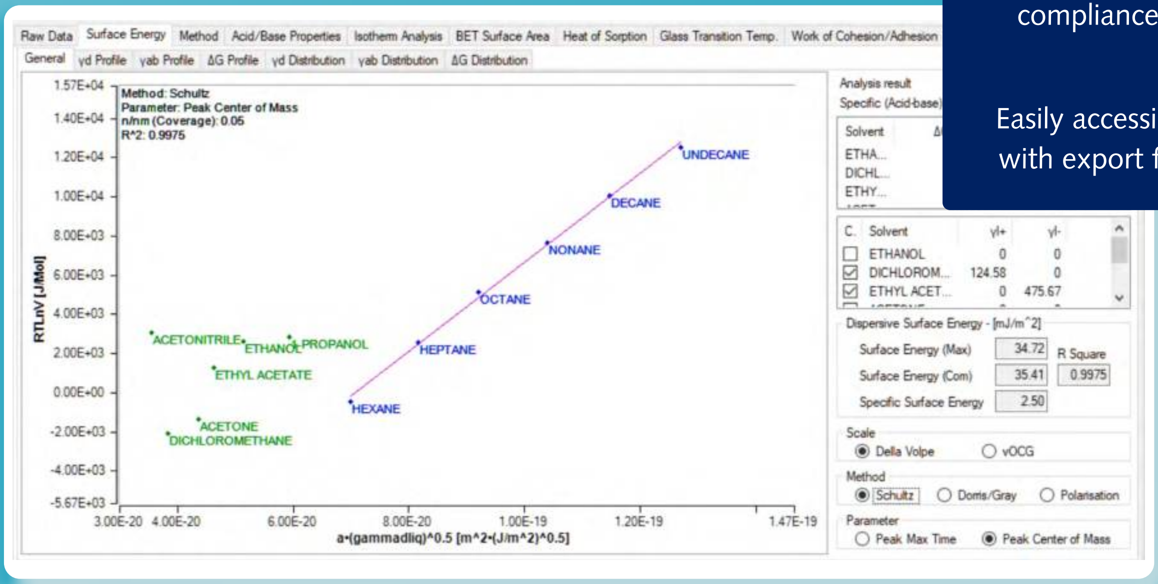
Automation of sequences, accommodating up to ten methods with 2,500 injections

Email notifications for real-time experiment updates

CFR 21 Part 11 compliance validation

Easily accessible raw data with export functionality

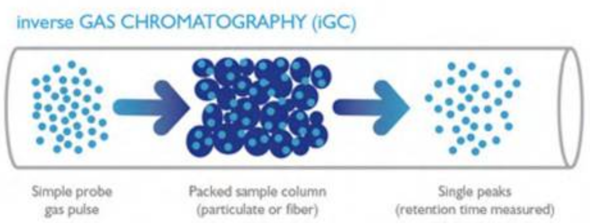
Analysis Software

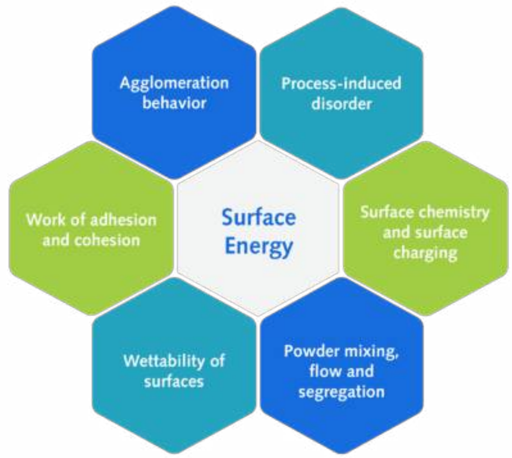


The Inverse Gas Chromatography Technique

The iGC-SEA is a purpose-built Inverse Gas Chromatography (IGC) system designed to measure surface energy and various physiochemical properties. In this process, the sample is packed into a column and positioned within a controlled environment where temperature and humidity are regulated. During an IGC experiment, different vapor probe molecules are introduced to account for both the dispersive (non-polar) and acid-base (polar) components, allowing the determination of intermolecular force strength during vapor-sample interactions.

Once the surface energy of a solid has been determined, these values can be correlated to several key solid properties, including wetting, dispersibility, powder flowability, agglomeration, process-induced disorder, adhesion/cohesion, static charge, adsorption capacity, and surface chemistry.





Surface Measurement Systems, Your iGC Specialists

- We are the producers of world's first purpose-built IGC instrument
- We are pioneers in vapor sorption instrumentation with over 30 years of continuous innovation.
- Every instrument is built upon the knowledge and experience of our industry leading sorption scientists.
- Our service team provides uncompromising support to our customers and partners.
- We ensure outstanding instrument performance.
- The iGC-SEA is accompanied by a complete Windows® software for experimental control and analysis.
- Industries using iGC-SEA: building materials, personal care, chemical, pharmaceutical, energy, food, and more.

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Surface Measurement Systems
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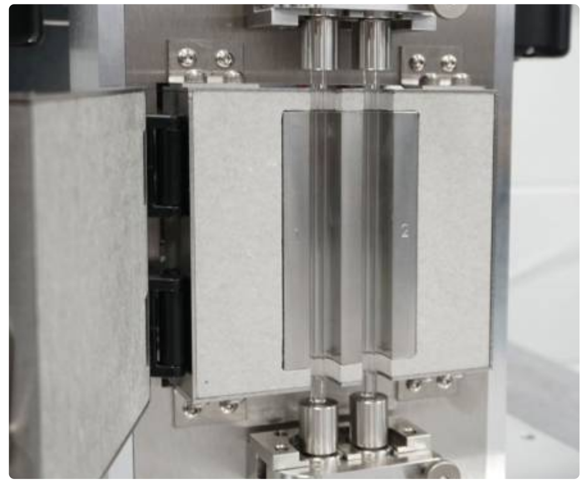
BTA Frontier

Self-Contained Multi-Component Breakthrough Analyzer

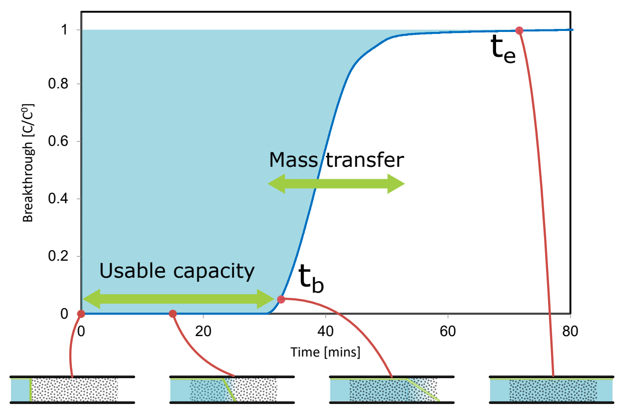
Single & multi-component sorption analysis for direct insights into breakthrough and equilibration times

BTA Frontier

The **BTA Frontier** revolutionizes sorbent testing with its cutting-edge packed bed breakthrough analysis, delivering unmatched precision in real-world process conditions. Engineered for versatility, it accurately measures competitive adsorption with a wide range of gases and vapors across multiple sample types. By seamlessly mixing single or multi-component adsorbate streams and passing them through a packed sample column, the BTA Frontier quantifies retention of the component. This allows crucial sample properties and process-relevant key performance indicators to be determined.

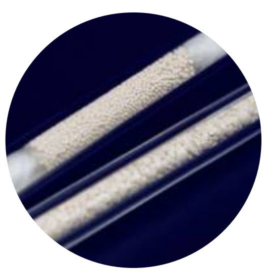


User-friendly and efficient, it features advanced sensors for CO_2, H_2O, organic vapors, and a thermal conductivity detector (TCD) with an optional mass spectrometer. Purpose-built for precise gas-phase mixing, adsorbate detection, temperature, and flow control, the BTA Frontier ensures unmatched accuracy and confidence in your results.

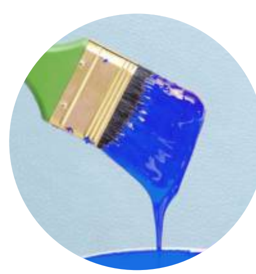


Industry Applications:

Carbon Capture, Utilization, & Storage



Material Characterization



VOC Capture & Remediation

Adsorbent Process Scale-up

What is BTA?

Breakthrough Analysis (BTA) is a sorption technique for determining breakthrough curves, tracking how a sorbent material interacts and captures vapors/gases over time. It reveals key adsorption insights, including breakthrough times, saturations points, and equilibrium data.

Key Features

Single-Component Breakthrough Curve

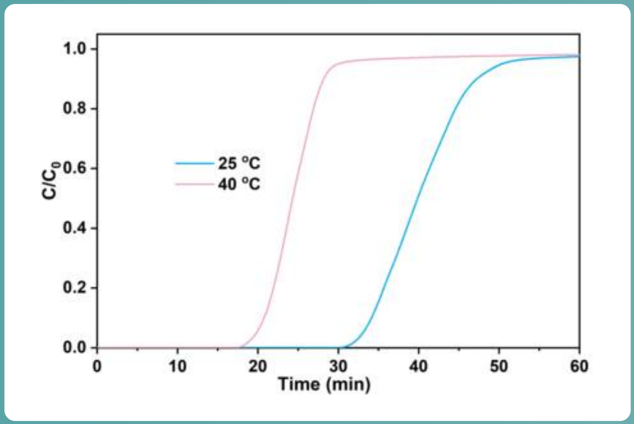


Figure 1. Water (50%RH) adsorption on 13X zeolite

Single-Component Breakthrough Curve

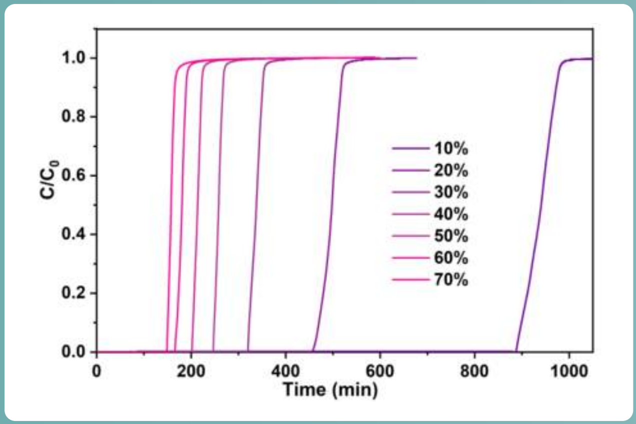


Figure 2. Water adsorption on 13X zeolite at 25 °C

Thermal Desorption

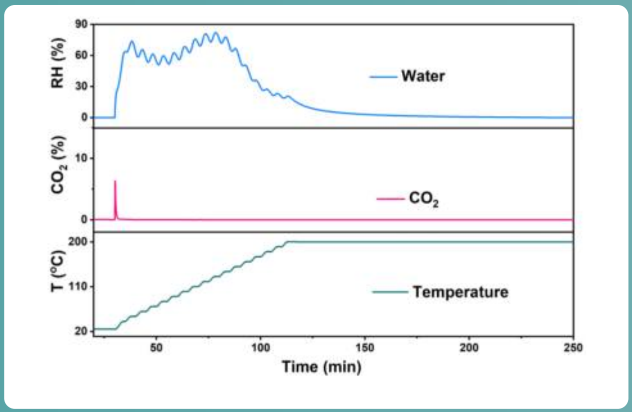


Figure 3. Desorption of CO_2 and water after adsorption on zeolite 13X

Sorbate Cycling

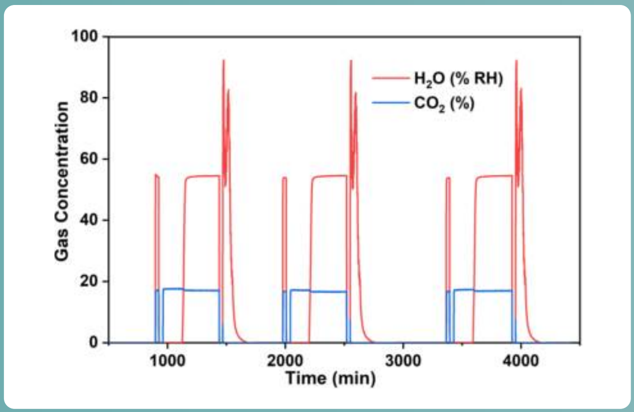


Figure 4. Cyclic adsorption

Comprehensive Gas and Vapor Breakthrough Measurements:

- Conduct single (Fig 1 & 2) or multi-component sorption (Fig. 3), sample regeneration, and cycling (Fig. 4) experiments.
- Analyze true multi-component sorption, assessing the uptake of one gas/vapor (e.g., CO_2, VOCs) in the presence of another (e.g., water, N_2).
- Utilize independent control over a wide range of gas/vapor concentrations.

Self-Contained, Scalable System:

- Generate uniform gas mixtures for accurate testing across different materials.
- Simultaneously perform multiple measurements using a standard sensor array.
- Measure VOCs from ppb to high %, and CO_2 from ppm to high %, with fast sample loading and changing.

Unmatched Flexibility and Accuracy:

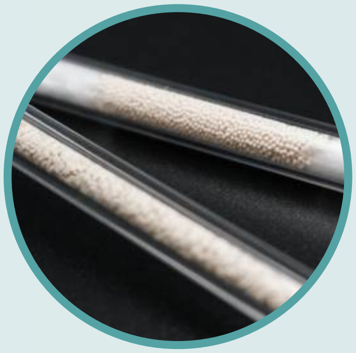
- Parallel activation in two columns for increased throughput. Accommodate samples masses from 30 to 3000 mg and a range of sample morphologies (e.g., pellets, monoliths).
- Utilize up to six gas inlets, two heated liquid reservoirs, and a temperature-controlled incubator.
- Optimized flow path with automatic dead volume determination, dedicated mass flow meter for outlet flow measurement, and pressure transducers at the columns end pressure indicators before and after columns.
- Optional mass spectrometer is available for analyzing complex gas mixtures.

Hardware

Versatile Sample Set-up

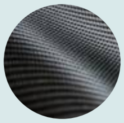
The BTA Frontier's innovative two-column design allows for sequential experiments, enabling simultaneous sample activation while significantly increasing throughput to optimize your analysis schedule. Whether you're conducting small-scale studies or large-scale projects, the BTA Frontier adapts seamlessly to your research needs.

Engineered for consistency, it delivers precise, repeatable results, making it an ideal choice for advancing gas separation research and material characterization.



- Sample columns available in multiple internal diameter sizes: 2mm, 3mm, 4mm, and 1 cm and in both stainless steel & silanized glass.
- Accommodates a variety of samples, including powders, granules, pellets, fibers, and more.



- Used with or without a temperature probe embedded in the bed

Precise Temperature Control for Optimal Performance

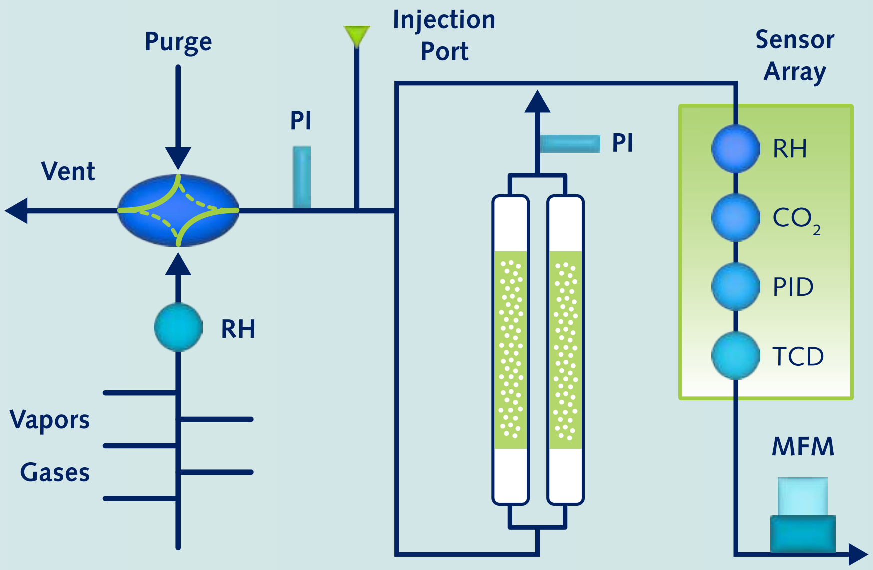
The BTA Frontier's column oven and dynamic control features provide precise, stable temperature regulation throughout automated experiments, offering several key advantages:

- Delivering reproducible and reliable outcomes
- Ensuring stable organic and water vapor generation
- Preventing unwanted condensation
- Studying temperature-dependent reactions
- Enhancing sensitivity for detecting low-abundance analytes

Gas Generation/Mixing System Available

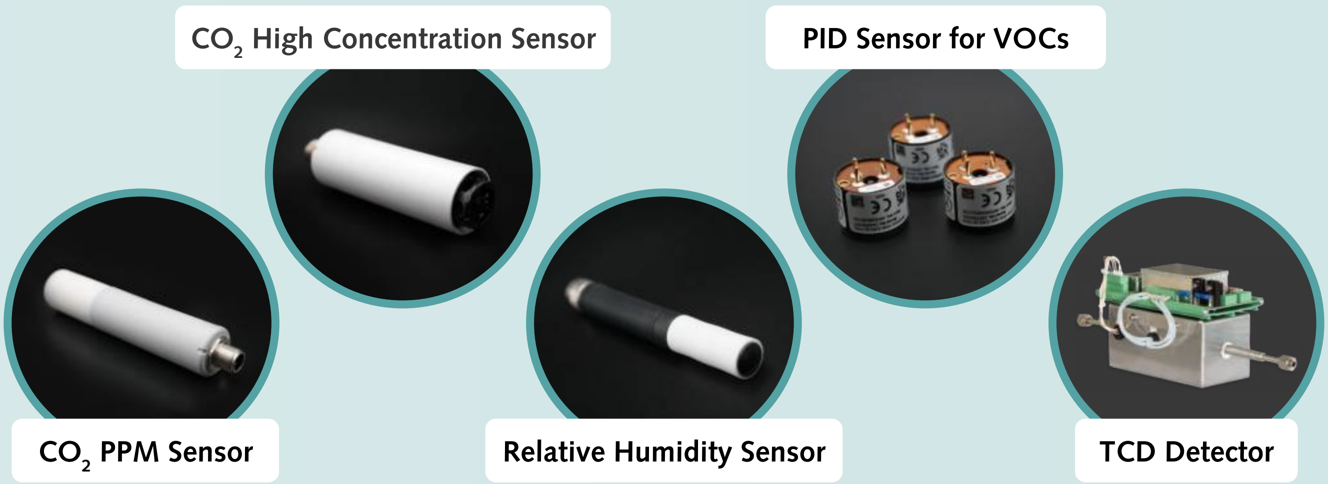
The BTA Frontier features precise gas generation and mixing with multiple inlets for flexible vapor or gas assignment. Its integrated sensor and purging system ensure uniform gas distribution, delivering reliable data across various applications.

- Achieve unmatched accuracy with uniform gas generation.
- Gain flexibility with multiple purging options before or during gas preparation.
- Prepare gas mixtures while performing other functions like simultaneous cooling and activation.



Advanced Sensors for Enhanced Application Potential

The secret to the self-contained nature of the BTA Frontier is its internal series of top-range sensors, enabling the instrument to engage in precise gas and vapor detection, giving the instrument its varied application potential.



Hardware Setup

Pressure Transducers

Located before and after the columns to accurately evaluate pressure drop.

Relative Humidity Sensor

Capacitive detector for relative humidity measurement (0-100% RH)

PID Sensors for VOCs

Detection of wide range of VOCs from ppb to % level (upper limit: 10,000 ppm)

CO_2 Sensor

- NDIR sensor capable of detecting CO_2 at concentrations relevant for PCC and DAC applications
- Two options targeted for High Concentration (%) and low concentrations (ppm) level

Two Heated Reservoirs

- The built-in reservoirs are located in the temperature-controlled incubator (5-60°C)
- Thoughtfully designed for generating water and organic vapors

TCD Sensor

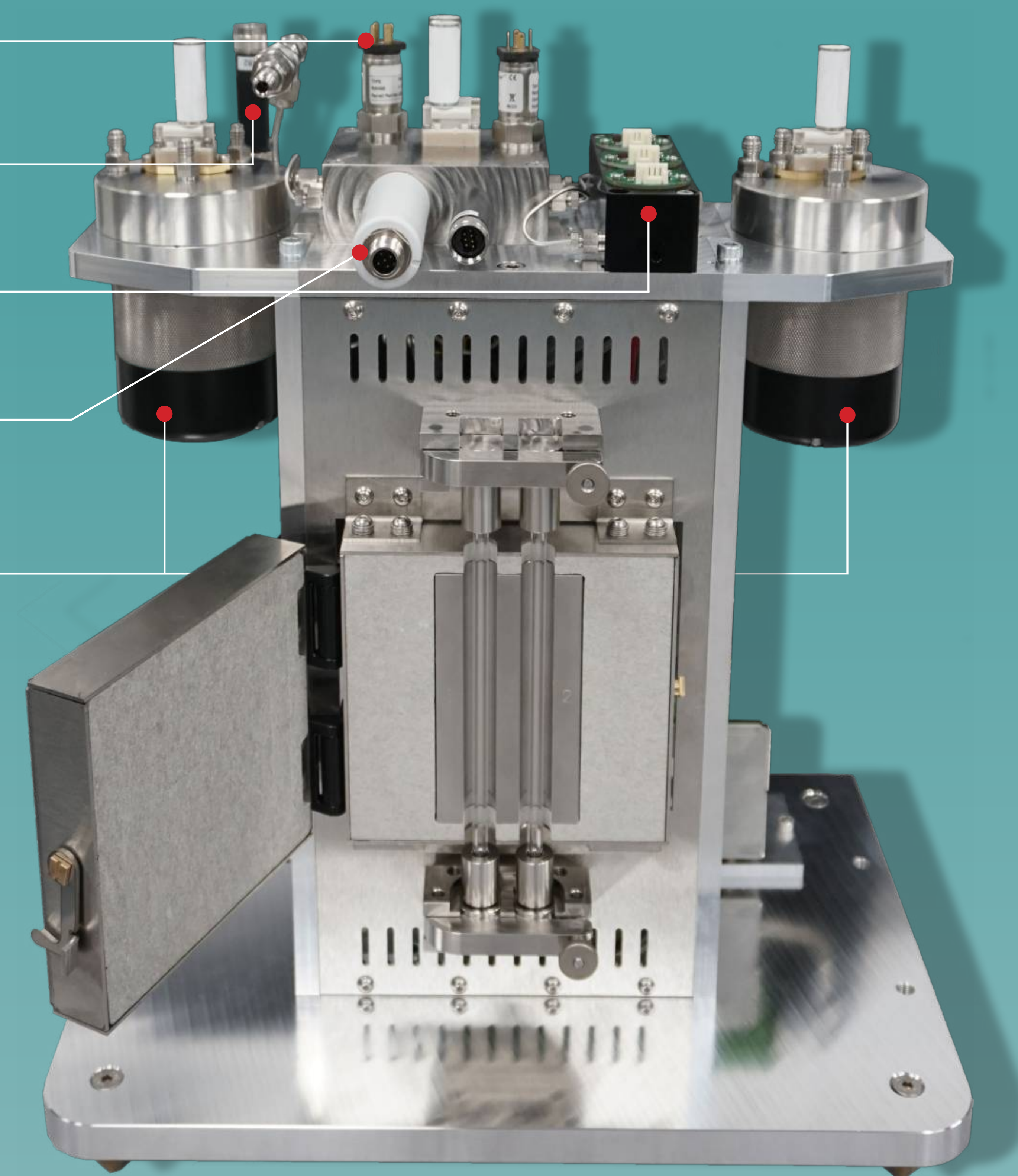
- General purpose, universal sensor for measuring concentration changes through thermal conductivity variations
- Self-contained heated unit with quick response time (T90 < 3s) and tunable ranges

Gas Inlets

- Equipped with five high-precision MFCs for seamless control of solvents, humidity, and gases on the feed side
- Dedicated purge gas MFC ensures system purity
- Effortless, software-driven gas calibration for all MFCs

Flow Path

- Advanced two-path bypass system with purge for both column and sensor train
- Minimizes dead volume while ensuring stable and complete feed gas mixing

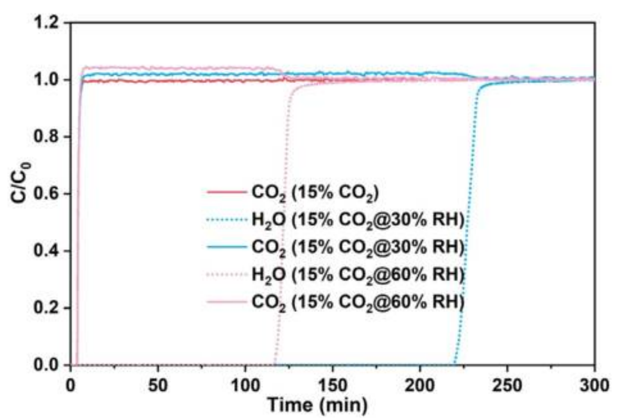


Case Study 1 : Carbon Capture with Zeolite 13X

Zeolite 13X, an aluminosilicate zeolite, is a promising material for carbon capture, particularly in Post-Combustion Capture (PCC) and Direct Air Capture (DAC).

In this case study, shaped zeolite 13X pellets (0.4 mm diameter) were packed into a 4 mm ID column. The sample was activated at 300 °C for 10 hours under a 200 sccm nitrogen flow, followed by cooling to ambient conditions (25 °C). Using the BTA Frontier, its gas adsorption properties were thoroughly analyzed. The system's precise control and multicomponent analysis provided valuable insights into zeolite 13X's gas adsorption efficiency under real-world conditions.

Post-Combustion Capture



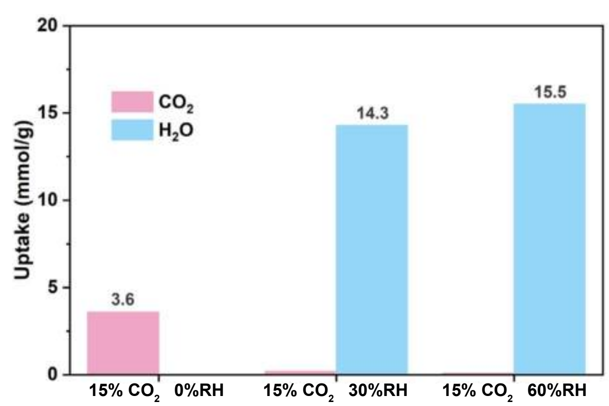


Fig 5 & 6. 15% CO_2 breakthrough (left) and uptake (right) at different relative humidity (0%, 30% and 60%) on zeolite 13X at 25 °C

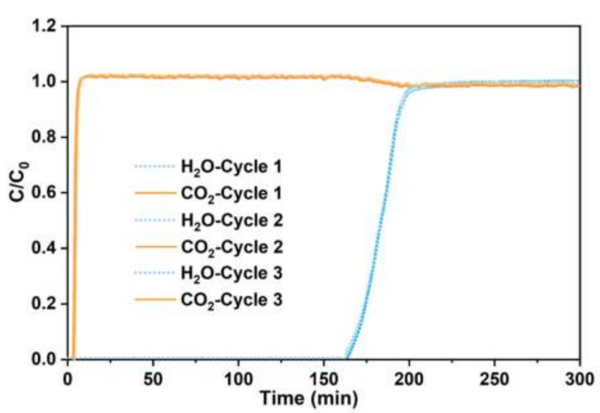


Figure 7. Cyclic CO_2 adsorption on 13x zeolite at 25ºC (15% CO_2 @ 60 %RH)

Post-Combustion Capture (PCC):

For point-source conditions a 15 vol% CO_2 flue gas analogue was selected. After activation, inlet conditions of 15% CO_2 at 0% (dry), 30%, and 60% RH were prepared using the BTA Frontier's gas generation and mixing system. Water vapor concentration was precisely controlled by bubbling nitrogen through deionized water via a heated reservoir, ensuring accurate and consistent humidity levels.

Case Study 1 : Carbon Capture with Zeolite 13X

Direct Air Capture (DAC)

Under DAC conditions, CO_2 concentration is as low as 400 ppm—four orders of magnitude lower than in typical PCC scenarios, making CO_2 capture measurements more challenging. After sample activation, the system was switched to dry or wet 400 ppm CO_2 for testing.

To ensure accuracy, a secondary blank test was performed, demonstrating the rapid response of the sensors and minimal dead volume, which highlights the BTA Frontier's precision in detecting low-

Direct Air Capture

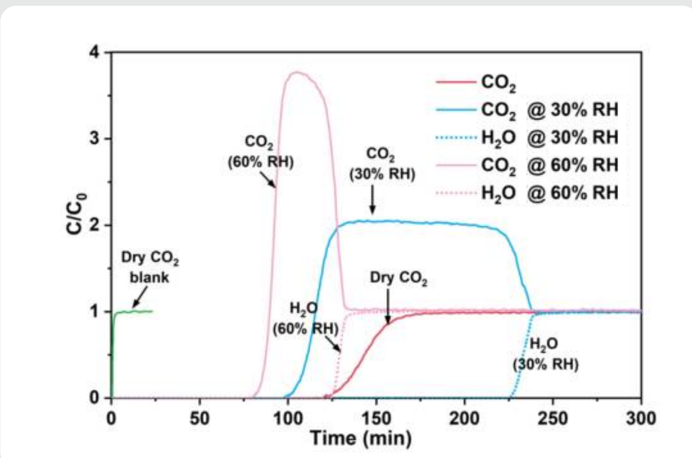


Figure 8. 400ppm CO_2 adsorption at different relative humidities on 13X zeolite at 25°C

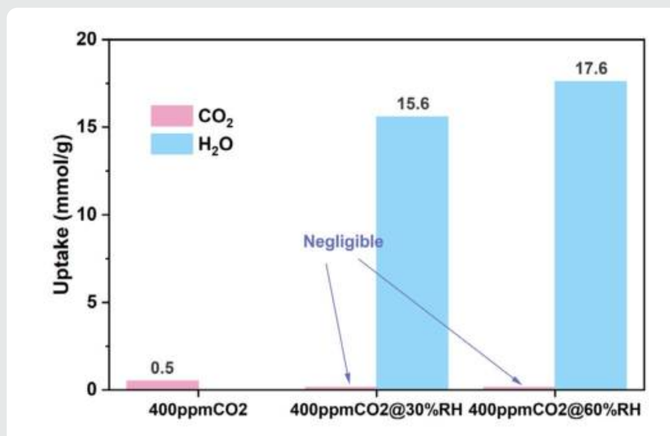


Fig. 9. Toluene (815ppm) adsorption on zeolite 13X under humid conditions (30 % RH) at 25 °C

Key Discoveries:

- **High CO_2 adsorption:** Zeolite 13X demonstrates strong CO_2 adsorption under dry conditions, achieving a capacity of 3.6 mmol/g for PCC and 0.5 mmol/g for DAC experiments.
- **Co-adsorption with water:** In both PCC and DAC scenarios, CO_2 is nearly (PCC) or fully (DAC) displaced by H_2O during co-adsorption on zeolite 13X.
- **Limitation in wet conditions:** The hydrophilic nature of zeolite 13X limits its effectiveness for CO_2 capture in humid environments.

Overall, while zeolite 13X performs well in dry conditions, its water sensitivity highlights the need for alternative materials for effective carbon capture in humid environments.

Case Study 2 : VOCs removal by Zeolite 13X

Volatile Organic Compounds (VOCs) are significant contributors to air pollution, making their removal a pressing concern. The efficiency of porous material filters in remediating VOCs from ambient air is often influenced by ambient humidity.

In this case study, we utilized the BTA Frontier to evaluate the adsorption efficiency of a common VOC, toluene, on a packed column of zeolite 13X. By leveraging the system's multicomponent capabilities, we further explored the effects of humidity on adsorption performance.

The zeolite 13X pellets were packed into a column and placed in the system. The sample was activated *in-situ* at 300°C for 10 hours under 200 sccm of N_2 before conducting adsorption experiments at a constant flow of 815 ppm toluene in N_2 at 25°C, under both dry (Fig. 10) and wet (Fig. 11) conditions.

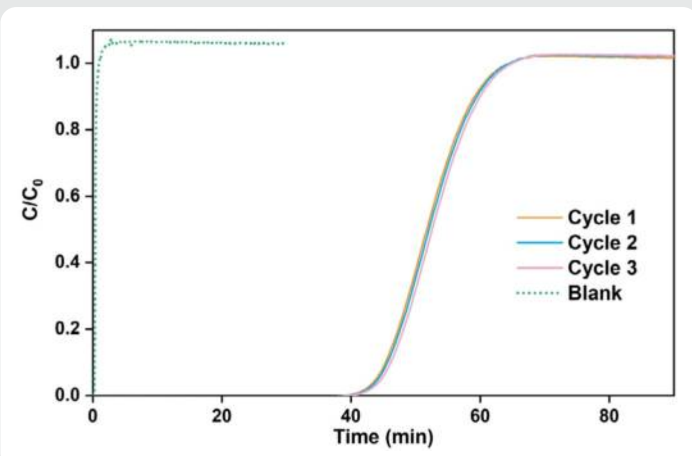


Fig. 10 Toluene (815ppm) adsorption on Zeolite 13X under dry condition at 25 °C

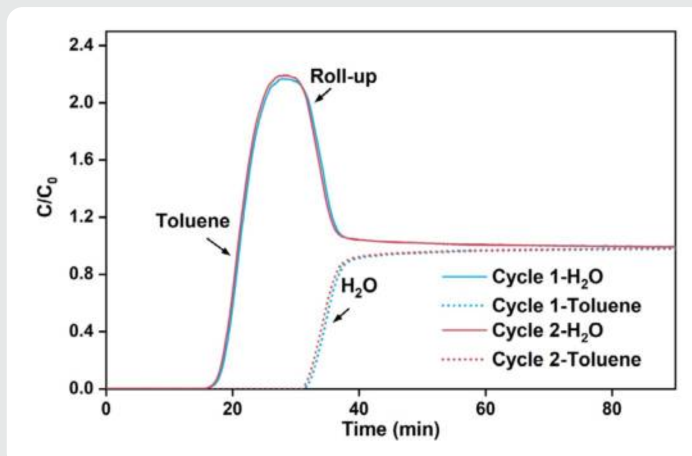


Fig. 11. Toluene (815ppm) adsorption on zeolite 13X under humid conditions (30 %RH) at 25 °C

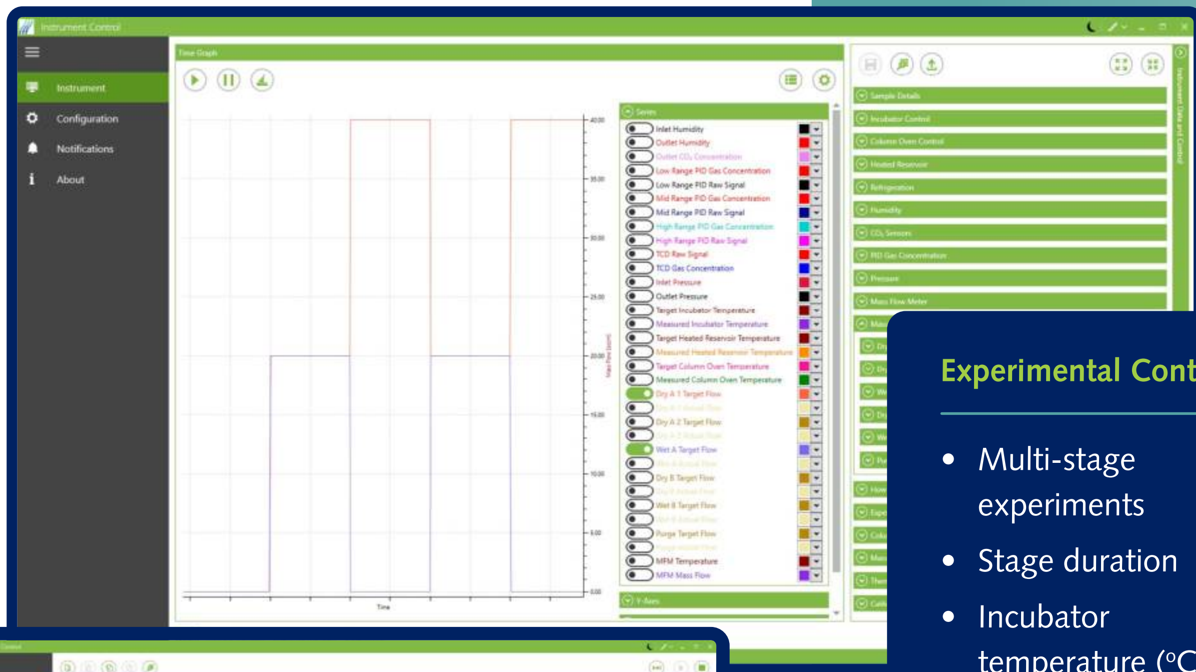
Key Discoveries:

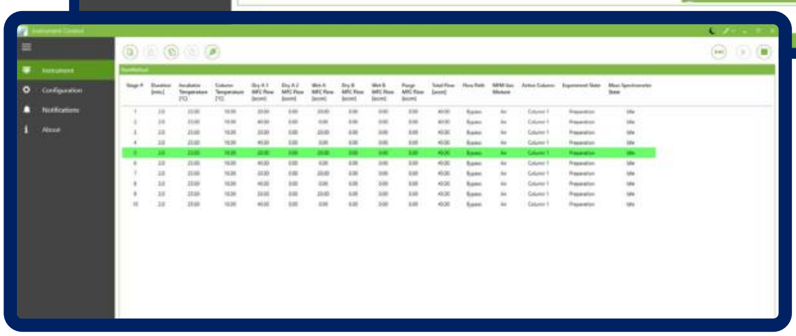
- **Dry conditions:** Zeolite 13X maintains a stable toluene adsorption capacity of 1.95–1.98 mmol/g over three cycles.
- **Wet conditions:** (30 %RH), the toluene adsorption capacity drops significantly from 1.95 mmol/g to around 0.17 mmol/g, due to the hydrophilic nature of zeolite 13X, which has a high water uptake of 15.5 mmol/g. The roll-up effect in the co-sorption curves (Fig. 11) shows that water displaces toluene.

The BTA Frontier accurately performs breakthrough curves for both single and multicomponent systems, offering insights into sorption phenomena, equilibration times, and competitive sorption between vapors.

Purpose-built Software

The Instrument control software package provided with the BTA Frontier allows the live plotting of data from all the MFCs, sensors, and heating units and comes with a method and sequence builder for creating complex experiments.





Experimental Controls:

- Multi-stage experiments
- Stage duration
- Incubator temperature (°C)
- Sample column temperature (°C)
- Dry and wet MFC flows

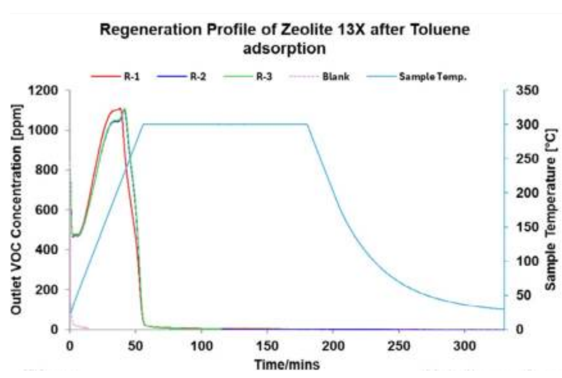


Figure 12: Graph produced from the BTA analysis suite macro

The powerful analysis software automatically compensates for dead volume and calculates uptakes using data from system sensors or any integrated mass spectrometer, ensuring accurate and precise results. With customizable graph options for plotting and data export in text format, this comprehensive analysis suite is ideal for presenting your findings.

Specifications

Construction Materials

Custom-built manifold: 316 stainless steel
Seals: Viton® or equivalent, Kalrez® optional
Tubing: 1/16 or 1/8 inch 316 stainless steel

Inlet Flow Control

Up to six gas inlets
Control Range per MFC: FS up to 200 sccm
Turn-down Ratio up to 1000:1
Calibrated gases include:
N_2, CO_2, He, O_2, Ar, & more

Temperature Control

Incubator Control
Controls entire gas mixing, vapor generation and measurement system
Control Range: 5 °C to 60 °C
Control Accuracy: ±0.1°C

Column Oven
Control Range: Up to 500 °C
Control Accuracy: ±.2 °C

Sample and Configuration

Two-column quick-connect system
Small sample amounts (~30 to 3,000 mg)

Column Sizes:
Length: 165 mm
Glass: 2, 3, 4, 10 mm ID
Stainless Steel: 3- 4 mm ID

Pressure Transducer

Pressure transducers at column inlet and outlets. Full scale 0-2.5 bar
Accuracy better than 1.0 % F.S.
Temperature compensated.

Vapor Generation

Liquid Reservoirs
Up to two 50 mL easy-change reservoirs
Heated to avoid evaporative cooling

Vapor Generation Range
0 – 90% for 5-60 °C [1]
Accuracy: ± 0.5 p/p0 [2]

Gas & Vapor Sensors

Humidity Measurement
Measurement Range 0-100 %RH
Accuracy (5-40 °C) ± 0.8 %RH
Accuracy (40-85 °C) ± 1.5 %RH

CO_2 Measurement
% level 0-20% vol, atmospheric pressure, accuracy down to 0.1 %vol CO_2
Ppm level 0-30,000 ppm, accuracy down to ±40 ppm CO_2

Thermal Conductivity Detector (TCD)
Quick response - T90 ≤ 3 sec
High corrosion resistance
High temperature capability up to 180 °C.

Organics Measurement (PID)
0 – 98% for 5-60 °C
Low-range sensor: 1 ppb-40 ppm [3]
Mid-range sensor: 0-4,000 ppm [3]
High-range sensor: 0-10,000 ppm [3]

System Information

Dimensions: 520 mm (W) x 980 mm (H) x 610 mm (D)
Weight: 80 kg (180 lb)
Electrical: 200 – 240 v, 50/60 Hz, 1,500 VA

System Software

Instrument Control Software

- Live data view and plotting
- Full control over parameters
- Powerful custom methods and sequences
- Multiple component detection
- Multiple concentrations or temperature cycles
- Temperature changes in a single experiment

BTA Analysis Software

- Easy plotting of normalized and molar concentration and other custom plots.
- Dead volume corrected capacities of multiple components.
- User defined control of calculation parameters.
- Software equipped with the calculation of dead volume corrected capacities of multiple components.

Footnotes
*10 – 40 °C
[1] Humidity factory calibrated at 25 °C and 60 °C. Calibrations at other temperatures upon request.
[2] Achievable with humidity – for other liquids depends on vapor pressure
[3] As calibrated with isobutylene

Surface Measurement Systems
World Leader in Sorption Science

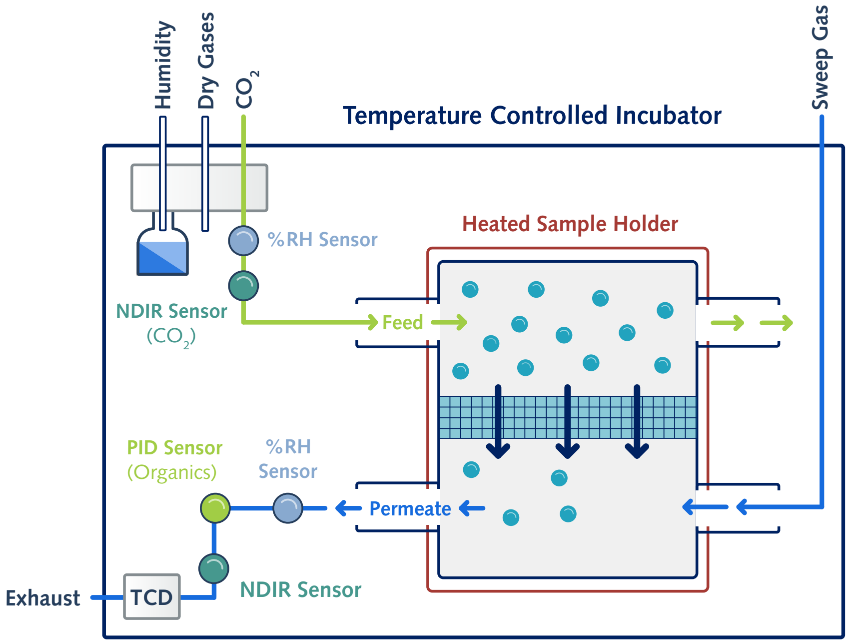
MPA Horizon

Self-contained Membrane Permeation Analyzer

Advanced cross-flow analysis to measure multi-component permeation through thin-films and membranes

MPA Horizon

The cross-flow permeation instrument for membrane analysis



The Membrane Permeation Analyzer (MPA Horizon) is a self-contained cross-flow gas permeation instrument, purpose-built for investigating the competitive permeation of gases and vapors through membranes and barrier films.

In most membranes, the nature of the environment can influence the permeation of gases or vapors, especially the temperature and humidity. The new MPA Horizon enables the analysis of gas/vapor permeation and permeation kinetics under real-world conditions.

Applications:

Fuel Cells: Ion Exchange

Food & Drink Storage

Electrical Insulation

Carbon Capture & Storage

Pharmaceuticals & Cosmetics

What is Permeation?

Permeation is the movement of molecules from one side of a material to another. The MPA Horizon monitors gas and vapor permeation through membranes using its versatile sensor suite.

Key Features

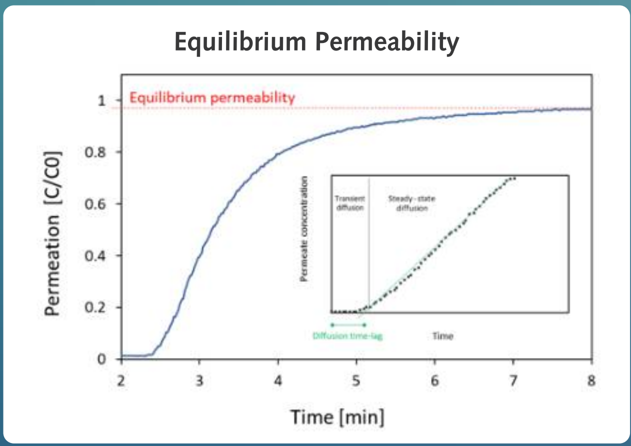


Figure 1: Example permeation plot and the metrics that can be obtained

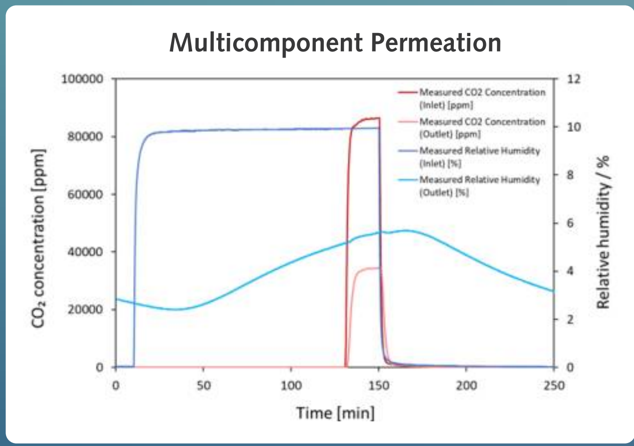


Figure 2: Multi-component permeation of humidity and CO_2 through a hydrophilic membrane

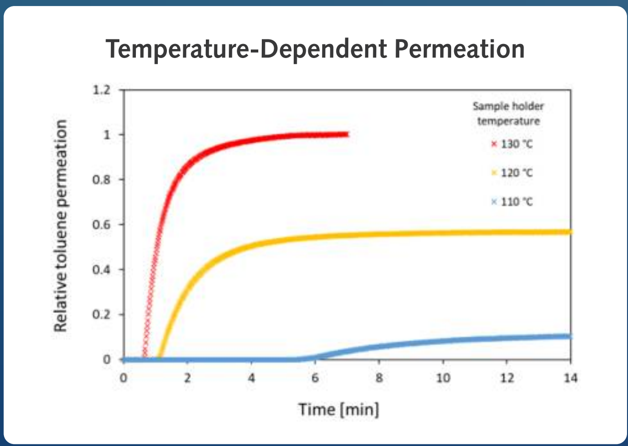


Figure 3: The effect of PET sample temperature on toluene permeation

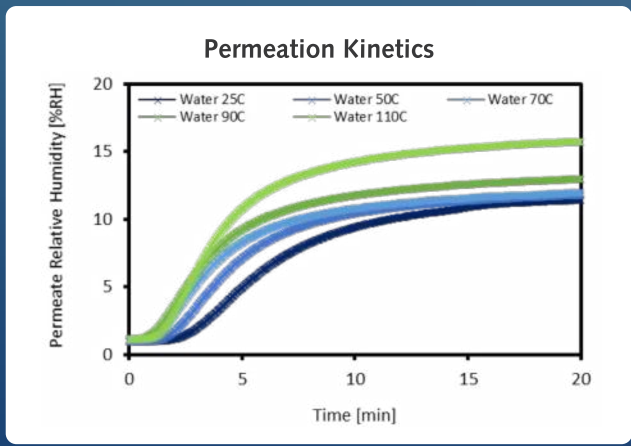


Figure 4: The change in humidity permeation kinetics with temperature through PET

Multicomponent membrane permeation

- Individual control of gases, vapors, and humidity concentrations
- Concentrations and temperatures manipulated in precise steps
- Complex multi-component methods and cycling can be performed with ease

Local heating of the sample

- Sample can be locally heated between 10-150 °C to simulate real-world conditions
- Determination of permeation and diffusion activation energies

Cross-flow gas system

- Inlet and outlet sensors for a complete understanding of permeation
- Capability to detect both the feed or permeate stream

Versatile gas/vapor sensor suite

- Individual detectors for humidity, organics, CO_2
- Universal detector for other gases using a TCD
- Further specific gas detectors, such as O_2, available on request

Hardware

Sample Holder

Our sample holder is a custom-built, simple-to-use double O-ring seal designed to accommodate small and large sample sizes up to 11 cm in diameter. Samples can be up to 0.5 mm in thickness, with thinner samples supported to prevent deformation.

Temperature Controlled Incubator

The sample holder, sensors, and humidity generation are all held within a temperature-controlled incubator to prevent any condensation and to aid temperature-sensitive experiments with fast and stable sample holder temperature equilibration.

This incubator also greatly increases the sensor reading stability when compared to products without temperature control.

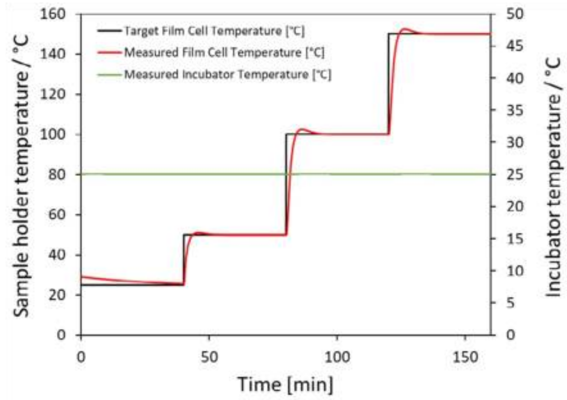


Figure 5: Sample holder heating control and incubator stability

Sensors: What Can You Run?

The MPA Horizon has a multi-purpose sensor suite built into the instrument meaning extra detectors are not required. As standard, the MPA Horizon sensors can detect:

- Humidity using capacitance probes
- CO_2 using near-infrared sensors
- VOCs (organics) using photo-ionization detectors
- Thermal conductivity detector for all other gases
- O_2 using an electrochemical optical sensor

The MPA Horizon has integrated humidity generation with fast and accurate concentration fronts thanks to precisely tuned temperatures, mass flows, and sensor calibrations.

- Achieves high humidity (<85%) even at high temperatures (60 °C) , without condensation
- High humidity stability ±0.1%RH over 6 hours
- Heated 50 mL water reservoirs

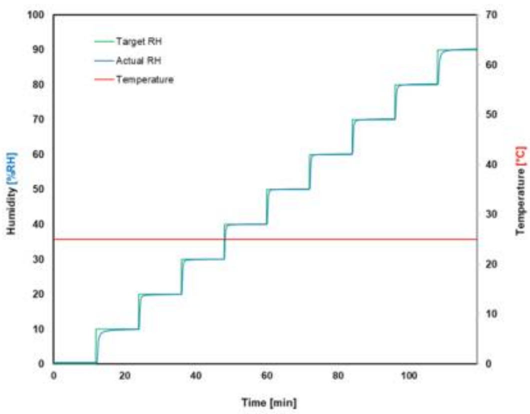


Figure 6: Stability and range of humidity at 25 °C

Controlling Your Flows

The MPA Horizon's unique design allows users to switch the gas calibrations of their MFCs via the software, allowing users to vary the gases and vapors that can be exposed to the sample. This is all housed within the self-contained unit for simple control.

The MPA uses a cross-flow system at atmospheric pressure, with the cross-flow and carrier gas tunable by the user.

As standard, users can switch the MFC calibrations for the carrier gas between:

- ✓ Oxygen
- ✓ Nitrogen
- ✓ Argon
- ✓ Carbon dioxide
- ✓ Helium

With six customizable MFCs, with flow rates up to 200 sccm, the MPA Horizon can mix wet and dry gases to create intricate concentration mixtures with humidity.

	MPA Horizon	Industry Standard
Generation of single component flows of O_2, CO_2, or water vapor (>90 %RH) in a carrier gas	✓	✓
Local heating of the sample	✓	✓
Cross-flow gas system at atmospheric pressure	✓	✓
Multicomponent permeation and detection for gases, vapors, and humidity	✓	
Sample bypass for the accurate quantification of feed gas concentrations	✓	
Alternating between detecting the feed and permeate streams	✓	

Case Study 1: Membranes for Carbon Capture

An attractive type of membrane materials for carbon dioxide removal are polymers of intrinsic microporosity (PIMs) – forming flexible and easy-to-manufacture single component membranes with high gas permeabilities and selectivity for CO_2 over N_2 and O_2 – primary components of flue gas. However, whereas the selectivity and permeability of membranes are often measured in ideal conditions, real flue gas contains a mixture of contaminants such as water which can impact the transport properties of a membrane.

The MPA Horizon was used to observe the multi-component permeation of flue gas constituents through two membranes: PIM-1 (hydrophobic) and cPIM-1 (hydrophilic). The purpose of this study was to reveal how modifying the polymer structure to be more hydrophilic changes the impact of humidity on CO_2 permeation and selectivity.

A selected humidity was first introduced to the top of the membrane for three hours, followed by a 30-minute step of 10% CO_2 whilst maintaining the selected humidity. The concentration of CO_2 permeating through the membrane was monitored to observe how the presence of humidity affects CO_2 permeation.

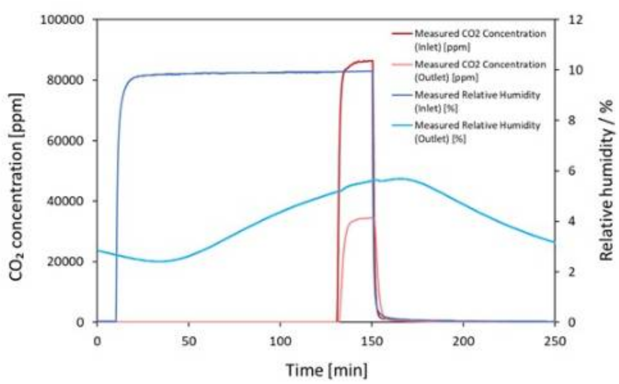


Figure 7: The impact of humidity on PIM-1 and cPIM-1 CO_2 permeation

The effects of humidity on the CO_2 permeability were evaluated for PIM-1 and cPIM-1, for which CO_2 permeability was seen to decrease by 20% and 40% respectively at 60 %RH.

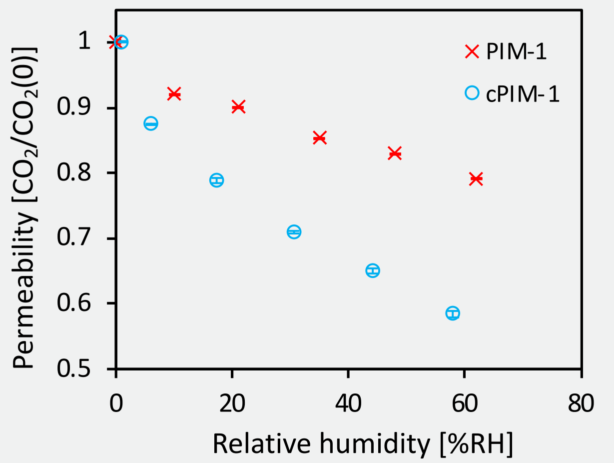


Figure 8: The impact of humidity on PIM-1 and cPIM-1 CO_2 permeation

The effects of humidity on the CO_2 / N_2 selectivity were also evaluated for cPIM-1. As shown in Figure 9, the selectivity of cPIM-1 decreases with humidity.

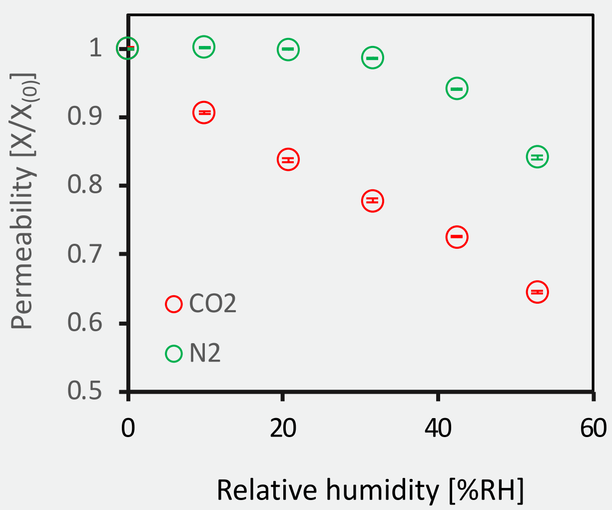


Figure 9: The impact of humidity on cPIM-1 CO_2 and N_2 permeation

Case Study 2: Permeation Through Barrier Membranes

Analyzing water permeation through barrier membranes under application conditions is vital. In the real world, the temperature and environment of a polymer can vary significantly, and these properties can have a significant impact on the polymer barrier properties. Temperature can make the polymer more flexible, increasing permeation, and VOCs can dissolve in the polymer changing its properties.

The permeation of toluene and water through two polymer films, PET and Kapton, have been evaluated under a range of temperatures and humidity. For PET, the humidity permeation was found to increase at temperatures above the glass transition temperature (>70 °C). This increase in water permeation was not observed in Kapton due to the much higher glass transition temperature of 300 °C. Therefore at higher temperatures, Kapton behaves as a better water barrier.

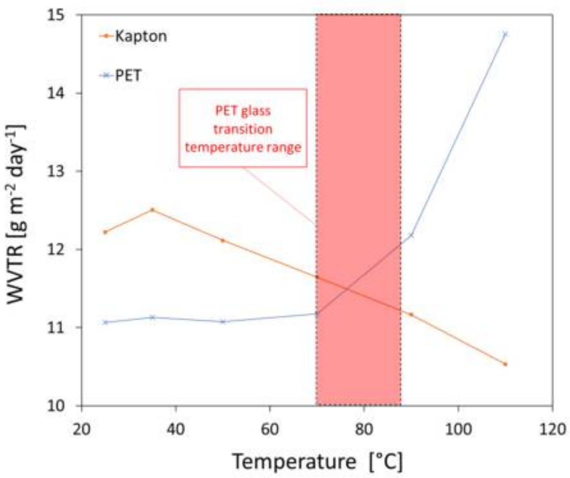


Figure 10: The change in water vapor transmission rate (WVTR) through PET and Kapton with temperature.

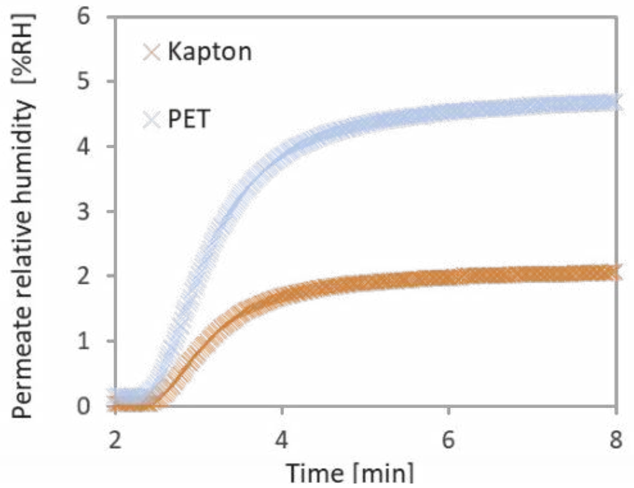


Figure 11: The permeation of humidity through Kapton and PET films at 130 °C

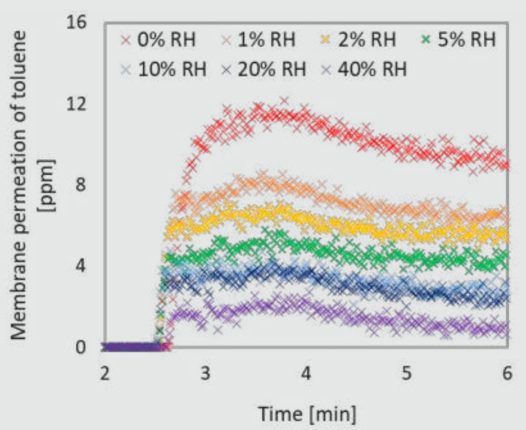


Figure 12: The change in toluene permeation through PET at 130 °C with increasing humidity.

Multi-component permeation

The permeation of toluene through PET with increasing humidity was observed by analyzing changes in the feed gas concentration after passing over the membrane. This analysis mode allows for the detection of ppm level changes in permeation. As shown in Figure 12, the permeation of toluene through PET decreases with increasing relative humidity.

Modular Capabilities

Gas Inlets

- Five gas mixing inlets on the feed side for solvent, humidity, and gases
- Check valves to prevent line contamination
- 5 feed gas MFCs with changeable gas calibrations via our software
- 1 sweep gas MFC with changeable gas calibrations via our software

Flow Path

- Accurate determination of feed gas concentrations via a sample bypass line
- Switchable flow path to detectors between feed and permeate side

Heated Reservoir

- The heated reservoir is held within the controlled enclosure
- Designed for accurate humidity generation [95%RH] up to 60 °C
- Reservoir held within a heated jacket to avoid evaporative cooling

Heated Sample Holder

(For sample activation and local heating)

- *In-situ drying of the sample between 10-150 °C*
- Local control of temperature for the observation of glass transitions

Optional:

Oxygen Sensor

A fully integrated optical oxygen sensor for the determination of oxygen permeation

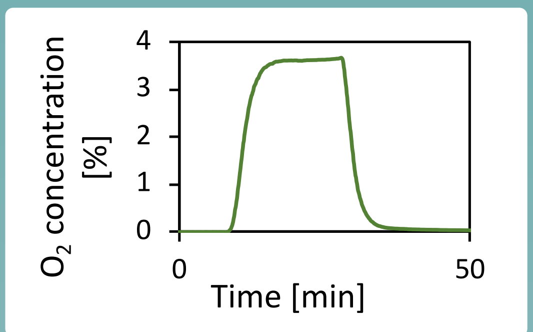
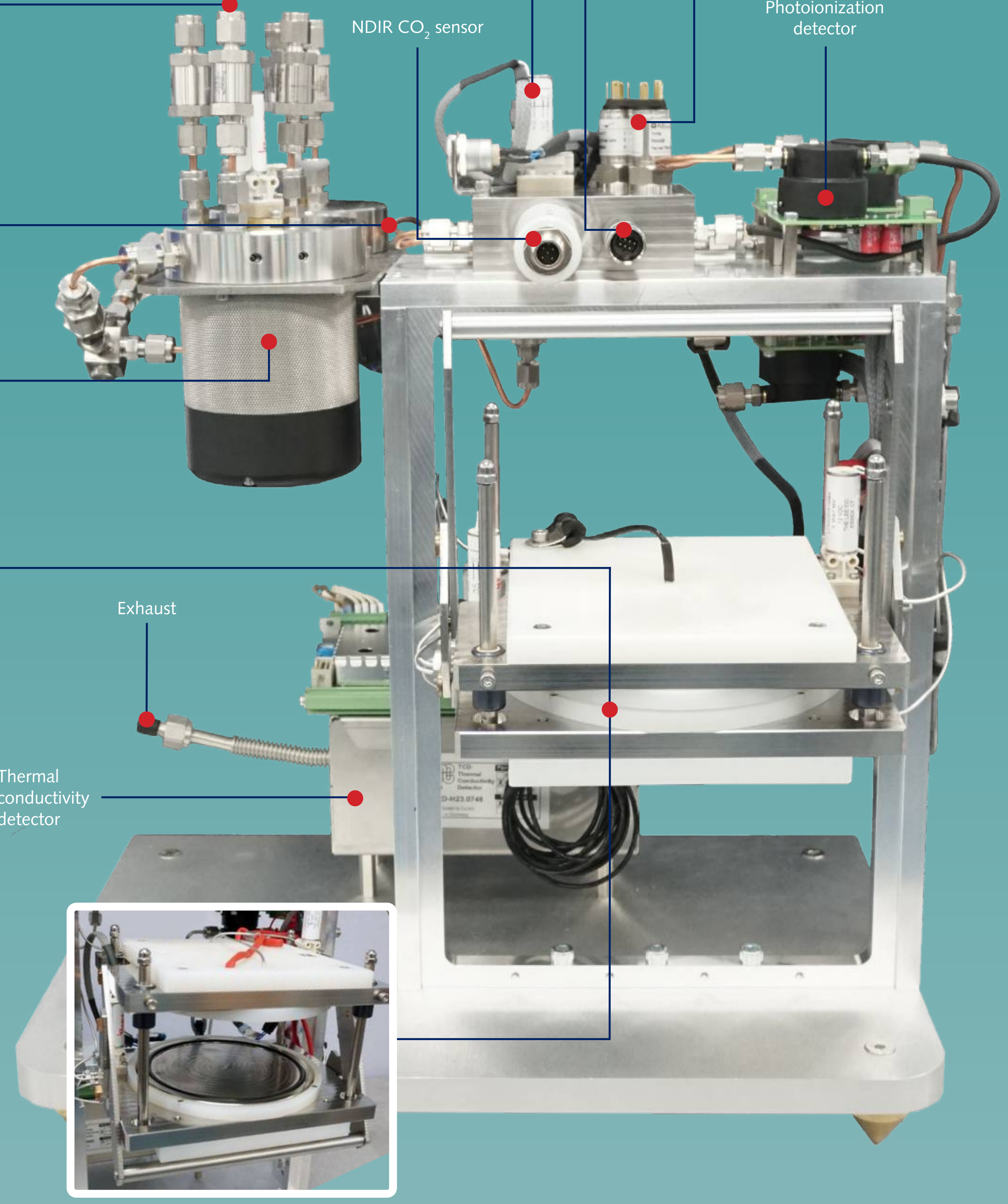


Figure 13: Oxygen permeation detected using a built-in optical sensor

Injection Port

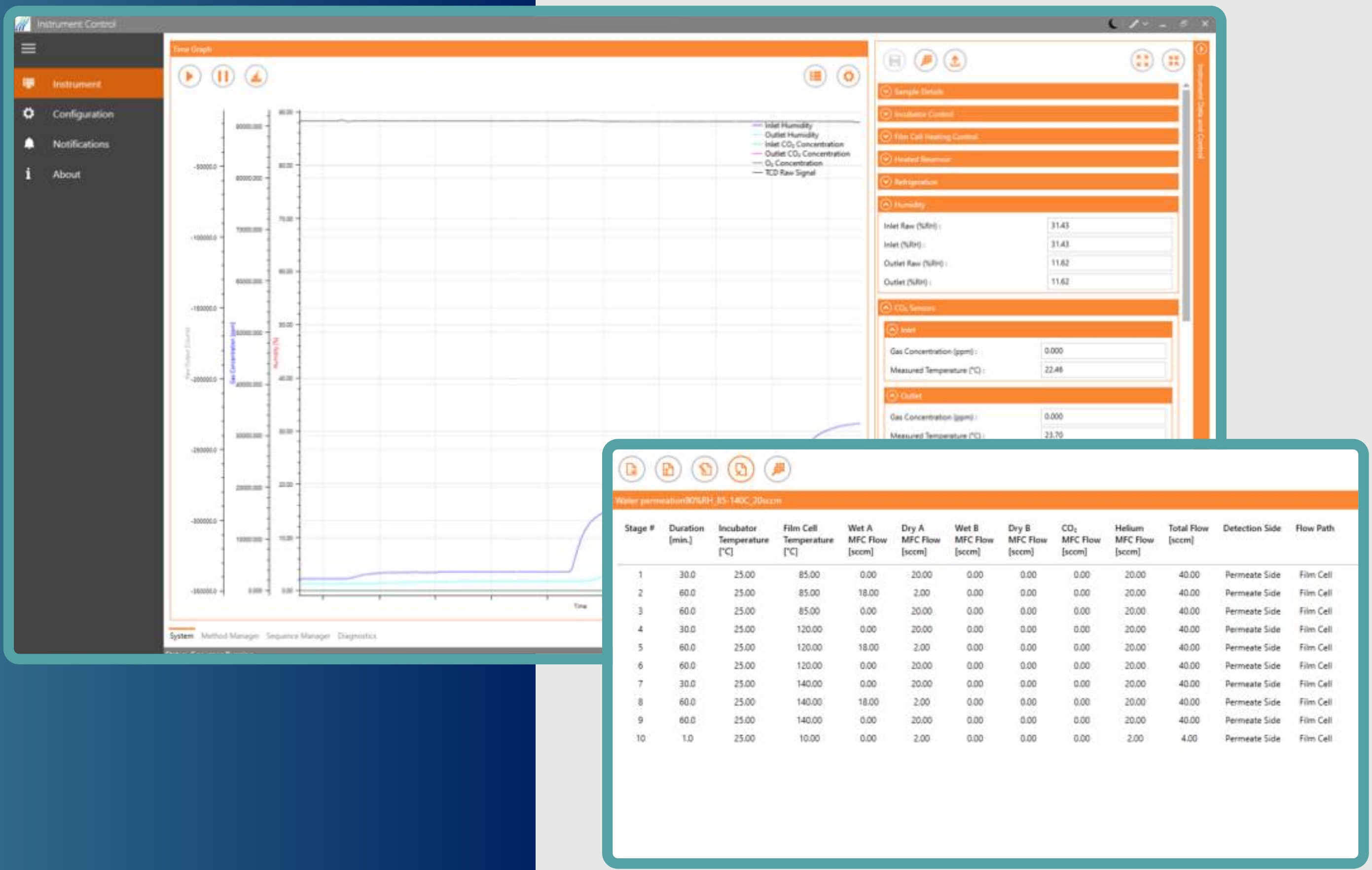
An in-line port for the injection of headspace concentrations or contaminants



Purpose-built Software

The instrument control software package provided with the Horizon allows the live plotting of data from all the MFCs, sensors, and heating units and comes with a method and sequence builder for creating complex experiments.

Control Software



Analysis Software

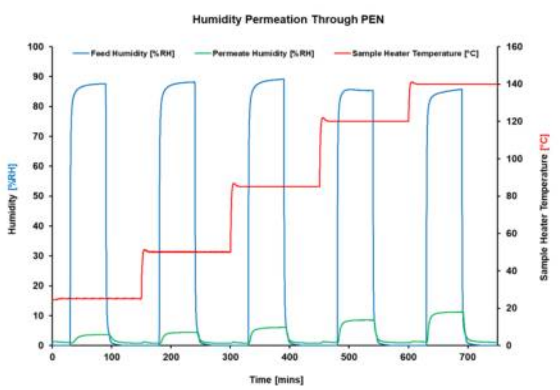


Figure 14: Graph produced from the MPA analysis suite macro

All data files from the MPA are saved as encrypted files for extra security. These files may be exported as text using the software and opened in our analysis software Excel macro.

This macro allows for easy data analysis and graphical plotting, an example graph is shown to the left in figure 14. It also includes calculations of permeation and WVTR.

Specifications

Construction Materials

Custom-built manifold: 316 stainless steel
Seals: Viton® or equivalent, Kalrez® optional
Tubing: 1/16 or 1/8 inch 316 stainless steel

Gas Flow Control

Control Range 0-200 SCCM
Turn-down Ratio up to 1000:1
Calibrated gases N_2, CO_2, He, O_2, Ar

Temperature Control

Incubator control
Controls entire generation and measurement system
Control Range: 5°C to 60°C
Control Accuracy: ±0.2°C

Local sample heater
Control Range: 10°C to 150°C
Control Accuracy: ±1 °C
Heated diameter: 120 mm

Sample Dimensions

Variable sample diameter
Standard width 11 cm
Thickness <0.5 mm

Humidity Generation

Water Reservoirs
50 mL easy-change reservoir
Heated to avoid evaporative cooling

Generation
0 – 98% for 5-60 °C [1]

Measurement
Measurement Range 0-100% RH
Accuracy (5-40 °C) ± 0.8% RH
Accuracy (40-85 °C) ± 1.5% RH

Gas & Vapor Sensors

CO_2 Measurement
0 – 25% vol, atmospheric pressure
Accuracy: Exact [3] or 0.05 %vol. of inlet concentration

Organics Measurement
0 – 98% for 5-60 °C
Low-range sensor 1 ppb-40 ppm
Mid-range sensor 0-4000 ppm
High-range sensor 0-10000 ppm

TCD Gas Measurement
Measurement Range 0-100%

O_2 Measurement
Measurement range 0-100%
Accuracy* (<1% O_2) ± 0.02%
Accuracy* (100% O_2) ± 2%

WVTR Measurement
(80 cm^2) 0.05 – 500 g m^{-2} day^{-1}

System Information

Dimensions: 520 mm (W) x 980 mm (H) x 610 mm (D)
Weight: 80 kg (180 lb)
Electrical: 200 – 240 v, 50/60 Hz, 1500 VA

System Software

Instrument Control Software
- Live data view and plotting
- Full control over parameters
- Powerful custom methods and sequences
- Multiple component permeation and detection
- Multiple concentration or temperature cycles
- Temperature changes in a single experiment

MPA Analysis Software
- Easy graphical plotting
- Permeability calculations
- Calculation of water vapor transmission rate (WVTR)
- Calculation of diffusion lag time

Footnotes
*10 – 40 °C
[1] Humidity factory calibrated at 25 °C and 60 °C. Calibrations at other temperatures upon request.

What is water vapor transmission?

The water vapor transmission rate (WVTR) is a measure of humidity permeation through a membrane of a specific area over 24 hours (g_{water}/ m^2/ day). On the MPA horizon, we measure this WVTR using the capacitance humidity probes, and the WVTR can be measured between 0.009 - 800 g / m^2 / day over a wide range of temperatures, with multiple components present. Parameters such as O_2 and CO_2 transmission rate can also be calculated using the MPA Horizon.

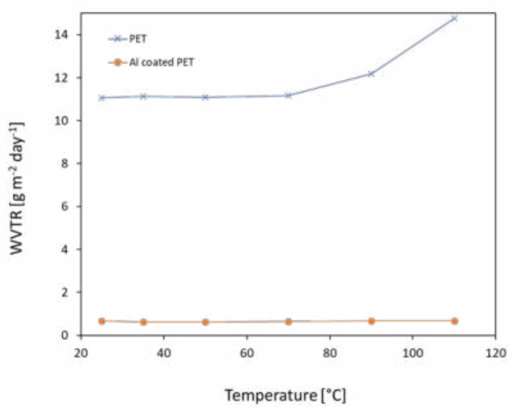


Figure 15: Water vapor transmission obtained from the MPA Horizon

Why do we need multi-component permeation?

The real-world application of membranes and films introduces materials to any number of fouling or swelling agents that affect the performance of these materials to act as efficient barriers or sieves.

Therefore, to realistically assess the film or membrane performance, the permeation of target gases and vapors must be performed in the presence of the multiple components to simulate real-world conditions.

About Surface Measurement Systems

Surface Measurement Systems Ltd. develops and engineers innovative experimental techniques and instrumentation for physico-chemical characterization of complex solids, solving difficult problems in materials research. With over 30 years of continuous innovation, every instrument is built with the cumulative knowledge and experience of our world-leading team of sorption scientists. This makes us the preferred sorption partner of universities, research institutes, corporate R&D, and global government organizations, who we work with and support in pioneering the future of materials research.

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Vapor Pressure Analyzer

Knudsen Effusion Method

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Vapor Pressure Analyzer

The VPA measures the vapor pressures of solids, liquids, and oils using the Knudsen Effusion Method.

The vapor pressure is an important physical property that defines the amount of vapor phase material that exists in equilibrium with the original material. All materials enter the vapor phase by sublimation (solid – gas) or evaporation (liquid – gas). The vapor pressure of a material at thermodynamic equilibrium is a fundamental property of the material and is only a function of temperature.

Knowledge of vapor pressure is highly desirable for many materials including pesticides and pharmaceutical samples in order to avoid the atmospheric accumulation of toxic compounds. Indeed, the vapor pressure is directly related to the Gibbs free energy of the original solid/liquid material.

The vapor pressure of material is required to be registered with the EPA (US Environmental Protection Agency) or the EC (European Community). The Knudsen effusion method used here is approved by the Organization for Economic Cooperation and Development and it is outlined in its Vapor Pressure OECD/OCDE Guideline 104.

Knudsen Effusion Method

The Knudsen Effusion Method is a dynamic gravimetric technique based on the rate of escape of vapor molecules through an orifice of known dimensions in a Knudsen cell into a vacuum at a known temperature. The rate of mass loss through the orifice is measured by the Surface Measurement Systems' UltraBalance within the VPA system. Sample masses from 1 to 100mg can be studied typically in the temperature range from 10°C to 400°C.

In a typical experiment, the sample is placed in a Knudsen cell made of titanium containing an orifice of known area and heated to experimental temperatures. The rate of mass loss is related to vapor pressure of condensed phase, P, by the Knudsen equation (Equation 1), where dm/dt is the rate of mass loss inside the Knudsen cell with time, M is the sample molar mass (mol/g), R is the universal gas constant, A is the area and T is the temperature.

$$P = \frac{\left(\frac{2\pi RT}{M}\right)^{1/2}\left(\frac{dm}{dt}\right)}{A} \qquad (1)$$

Dm/dt is the slope value from a least square regression fit to the experimental mass data over a certain period of time. The dm/dt is used to calculate the vapor pressure using Equation 1. A series of measured vapor pressures at differing temperatures are used to determine the constants A and B in the **Clausius–Clapeyron** equation (Equation 2) and thus calculate enthalpy of vaporization, ΔH, or heat of sublimation.

$$\log_{10}(p/Pa) = A - \frac{B}{T/K} \qquad (2)$$

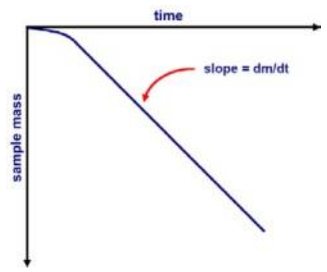
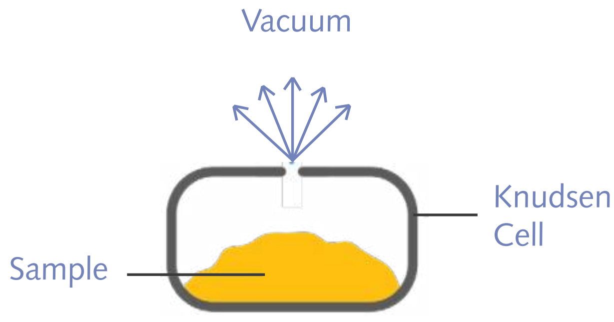


Figure 1 Mass versus time data from Knudsen cell

Knudsen cell



Experimental Data

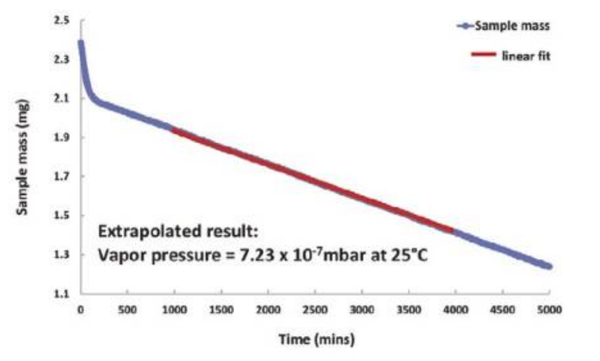


Figure 1. Vapor pressure of Bifenthrin is too low to be measured at 25°C, but can be extrapolated from the DVS Vacuum data recorded at 65°C.

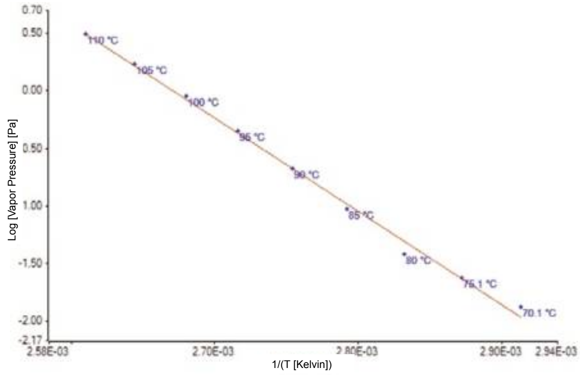


Figure 2. Plot of vapor pressure data for Bifenthrin.

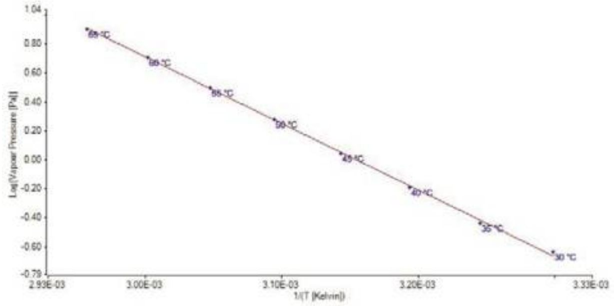


Figure 3. Plot of vapor pressure data for benzoic acid.

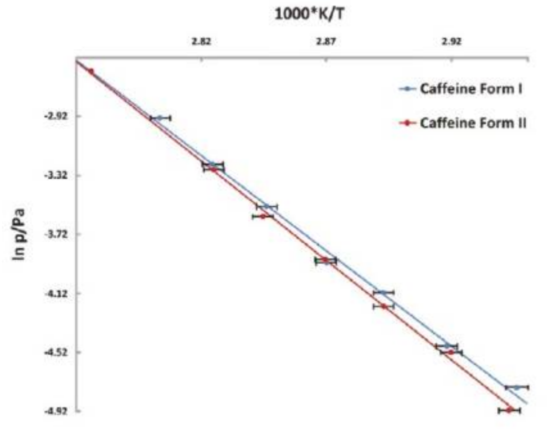


Figure 4. Vapor pressure of Caffeine vs temperature stability study. The lower vapor pressure of Form II shows it to be the more stable polymorph.

Software

VPA analysis software

includes the following features:

Windows®-based graphical interface

Knudsen vapor pressure analysis

One-click data analysis and report generation

Flexible data-range selection

Easy importing of results from different methods for simultaneous analysis

VPA control software

includes the following features:

Windows-based interface

Quick-set methods

Easy saving and restoring of methods

Real-time display of experiment progress

Data saved in a tab-separated values (TSV) file

Turbo pump control

Balance tare and calibration wizards

Incubator and pre-heater temperature control

Specifications and Schematic

Temperature

Temperature controlled enclosure
Control range: 10°C to 70°C
Temperature accuracy: ± 0.2°C

High-temperature pre-heater

400°C (maximum local temperature)
Temperature sensor: Pt-100

Vacuum generation

Roughing pump can produce minimum vacuum pressure of 1 x10^{-3}Torr.

Turbomolecular pump in combination with roughing pump provides vacuum pressure down to 1x 10^{-8}Torr.

Vacuum pressure measurement

Vacuum pressure transducer: full scale from 1x10^{-8}Torr up to atmospheric pressure.

Vacuum stand

Material: 316 stainless steel
Seals: Viton® and Kalrez®
KF flanges and VCR fittings

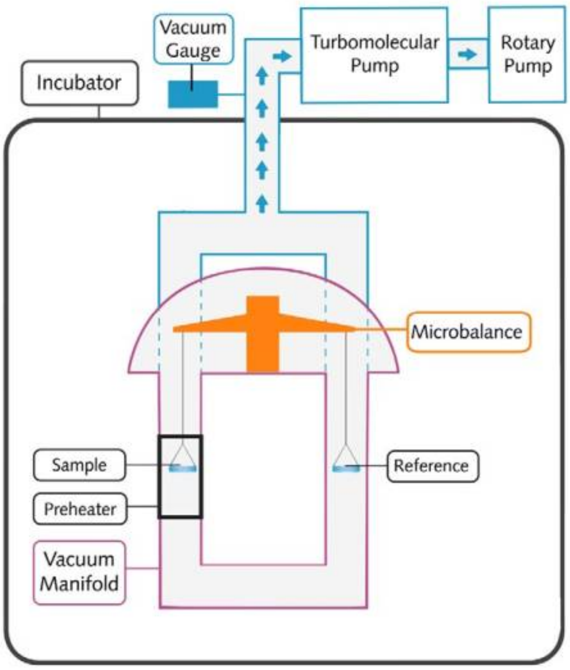
Mass Measurement

Ultrabalance Low Mass

Sample mass:	between 1 and 1000mg
Mass change:	up to ±150 mg
Resolution (precision):	0.01 µg
Root mean square balance noise:	≤ 0.3 µg

Ultrabalance High Mass

Sample mass:	between 10 and 5000mg
Mass change:	up to ±1000 mg
Resolution (precision):	0.1 µg
Root mean square balance noise:	≤ 3 µg



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