

Introduction to Dynamic Vapor Sorption and its Uses for Advanced Characterization of Porous Materials

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Characterization of Solids

- **Energy as a Probe**

1. Spectroscopy
2. Light, x-rays, lasers, etc.
3. Analytical and structural information

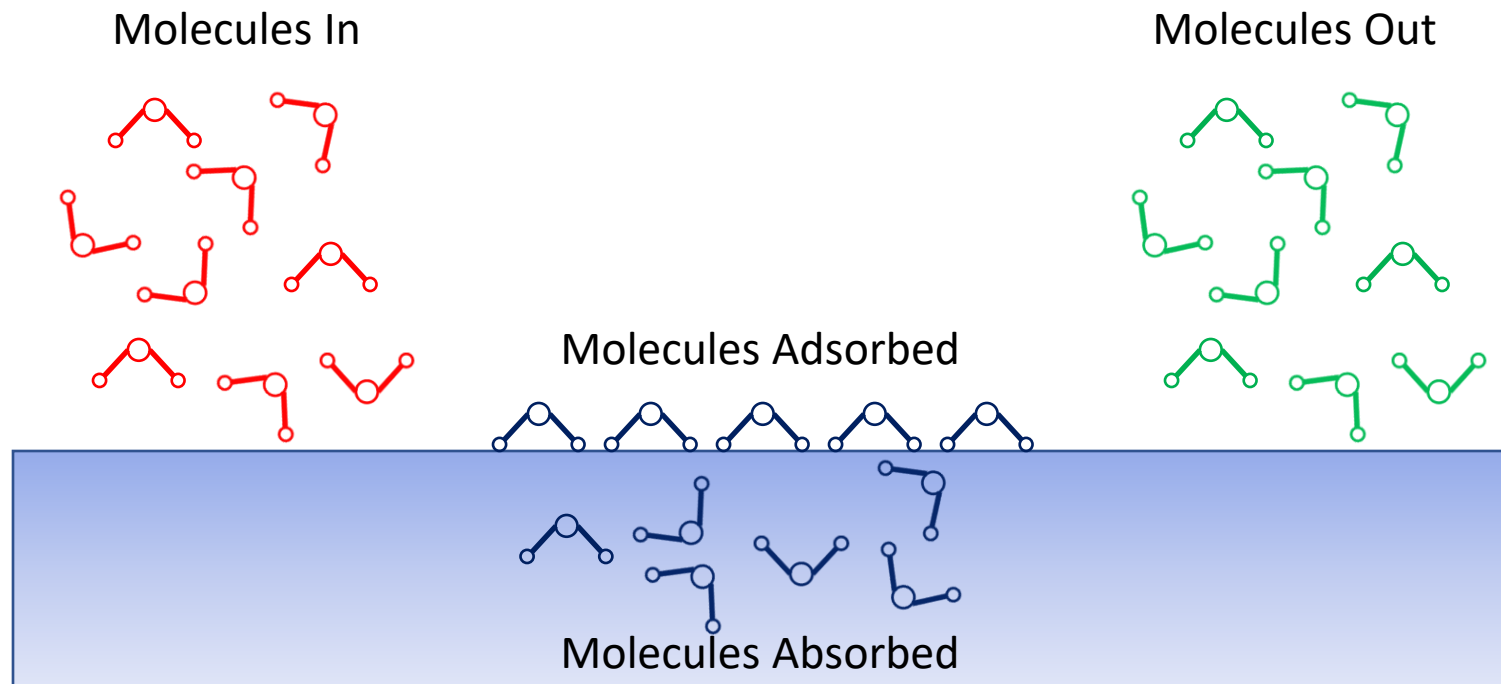
- **Heat as a Probe**

1. Calorimetry
2. Thermodynamic information

- **Molecules as a Probe**

1. Sorption techniques
2. Thermodynamic, chemical, and structural Information

Molecules as a Probe



Molecules as a Probe

- **Chemical Interactions**

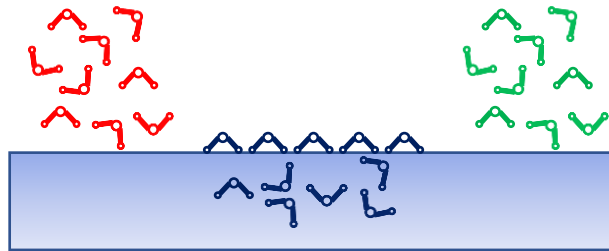
- IGC, DVS, Wetting, Chemisorption analyzers

- **Physical Structure (surface area, pore size, density etc.)**

- DVS, IGC, Volumetric sorption (i.e. BET analyzers), Chemisorption, Pycnometer

- **Thermodynamic Information**

- IGC, DVS, Thermal Analysis Methods

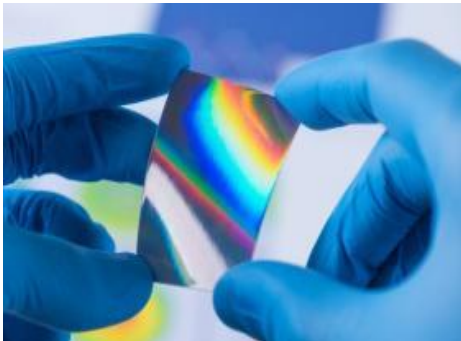
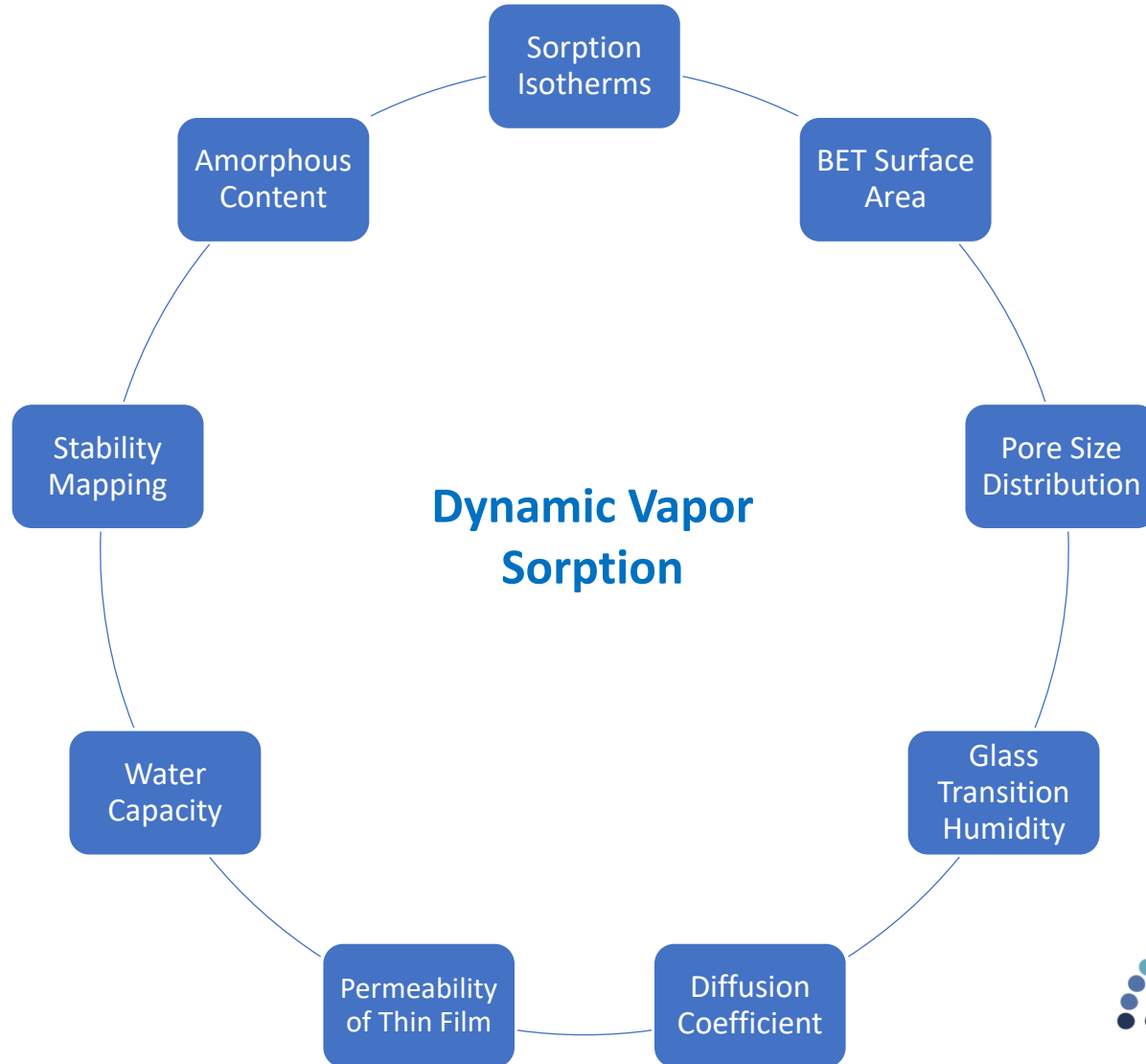


Moisture Sorption Technique Comparison

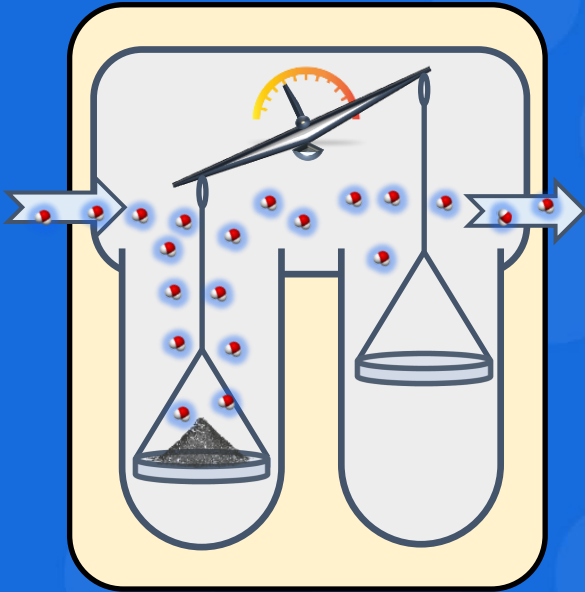
	Dynamic Vapor Sorption	Desiccator Jars	Volumetric Sorption
Sample Morphology and Preparation	Any sample placed on a sample pan	Manually weighing of samples	Sample must be placed in glass tube; semi-solids cannot be analyzed
Minimum Sample Sizes	0.5 - 10 milligrams	500 – 1000 milligrams	100 – 500 milligrams
Sorption Kinetics	Direct and automatic measurements as fast as every second	Data points only obtained on scale of hours or days	Not directly attainable
Reproducibility	Excellent reproducibility (up to 0.01 microgram resolution)	Low due to manually removing samples from chambers and weighing 'off-line'	Poor for samples with surface areas below 1.0 m ² /g
Measurement Conditions	Wide temperature and full humidity range under dynamic flow	Typically only ambient temperatures and discrete RH conditions	No carrier gas and typically samples must be exposed to high vacuum



Properties Measured by DVS



Dynamic Vapour Sorption (DVS) Introduction



Dynamic gravimetric Vapor Sorption (DVS)

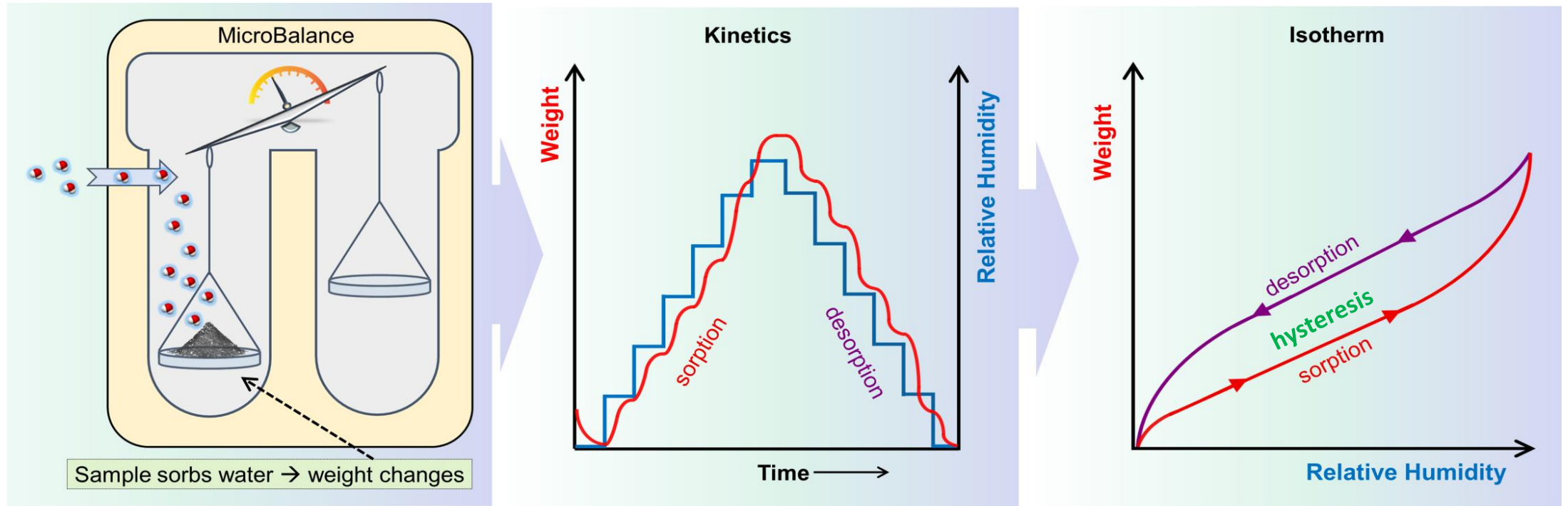
A **gravimetric sorption** method that **optimizes kinetics** by utilizing a **continuous flow** of adsorbate alongside a **highly sensitive balance**.

- **High sensitivity:** down to **0.1 μg** \rightarrow from **10 mg** of sample
- **Fast equilibrium:** significantly improved kinetics over static systems
- **Uptake** as a function of concentration or temperature (or both!)
- **Kinetics by default**

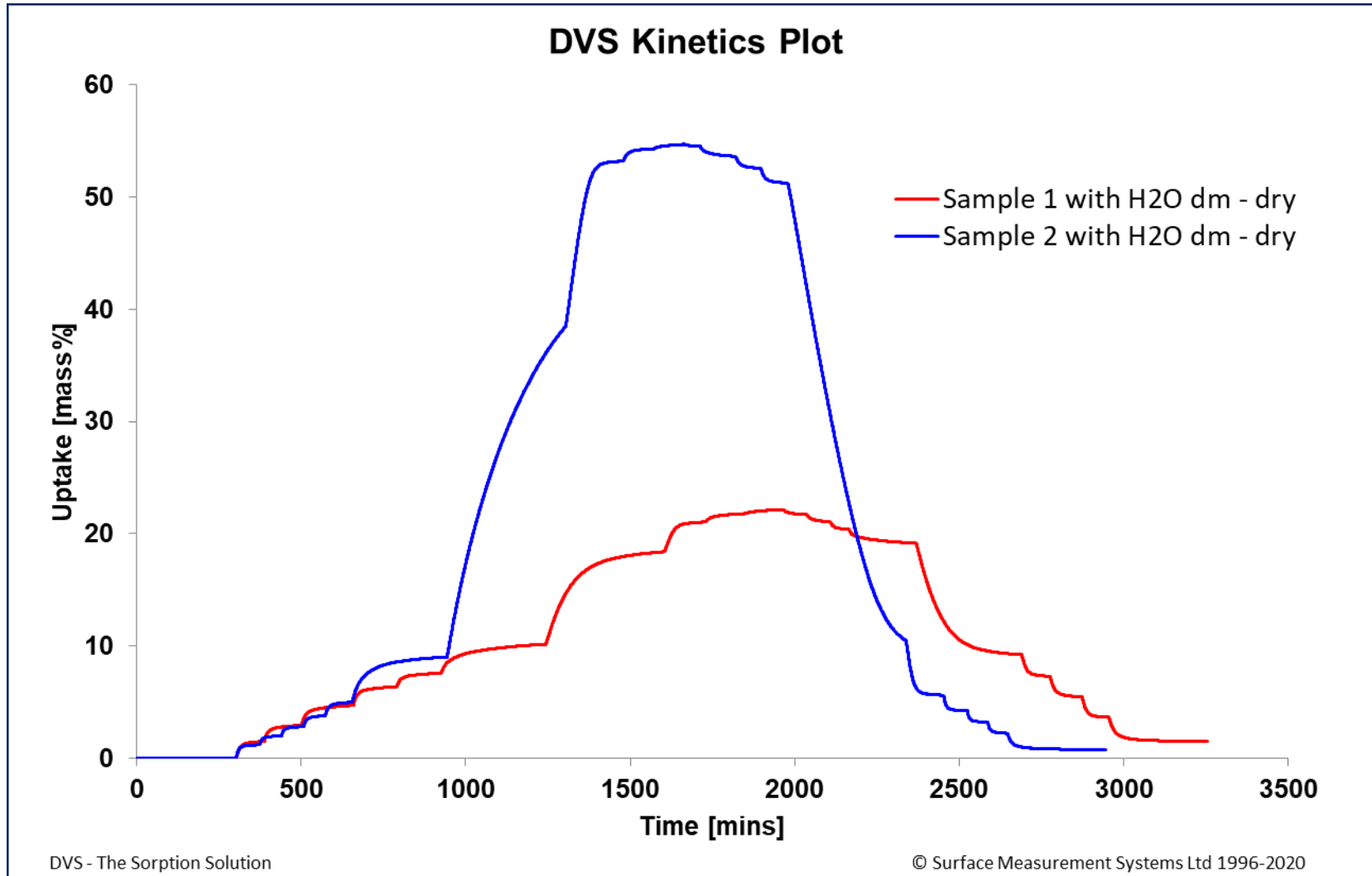
Research or Real-World conditions

- Carrier and pure flow configurations.
- Wide range of vapors and gases
- Wide range of temperatures: p_0 range up to 85C, local heating to 400 C
- Wide range of pressures: atmospheric down to **10^{-6} Torr**

How the DVS Technique works



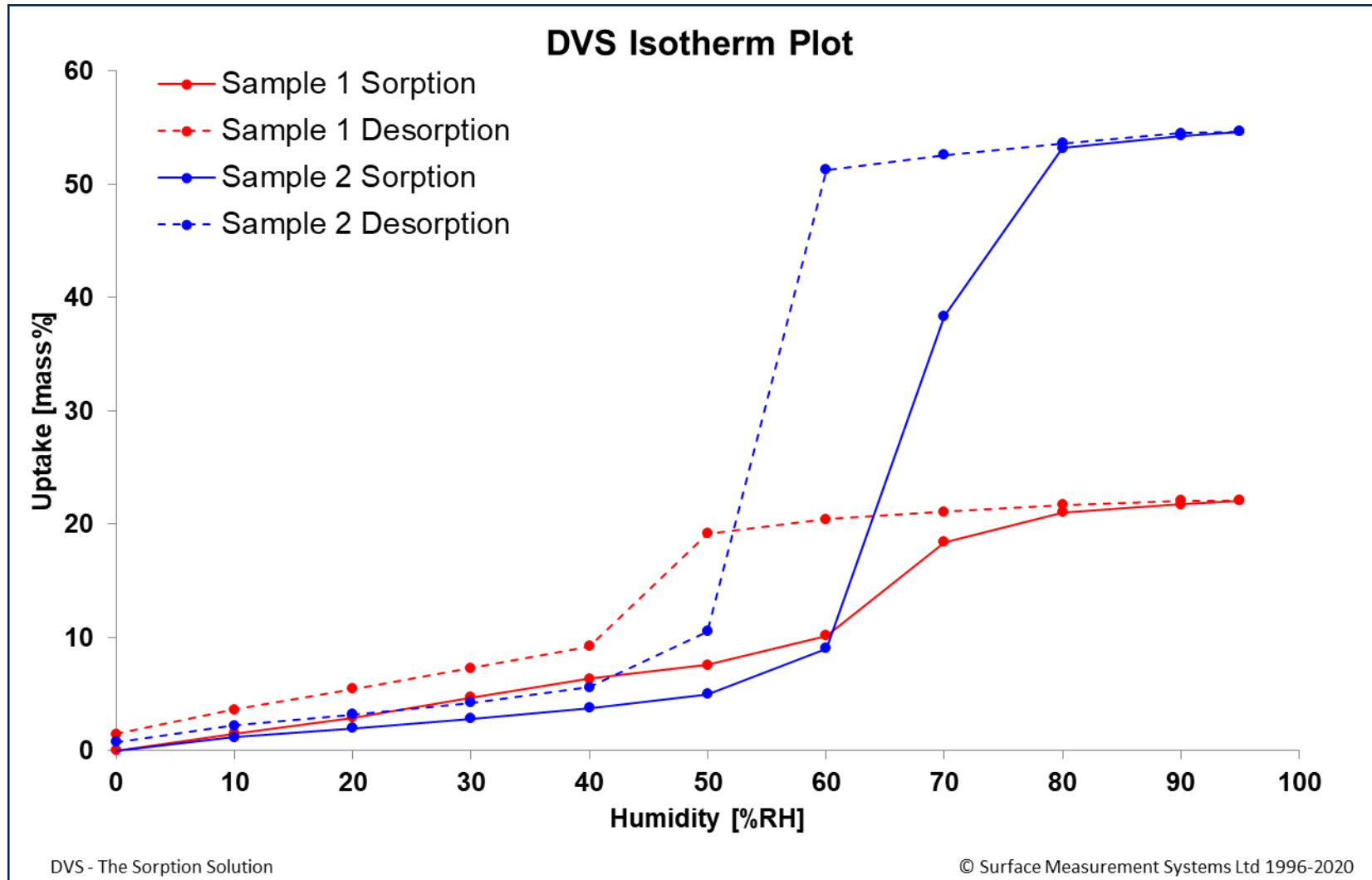
Water Sorption Results – Kinetic Plots



Provide with full water sorption / desorption kinetics information.

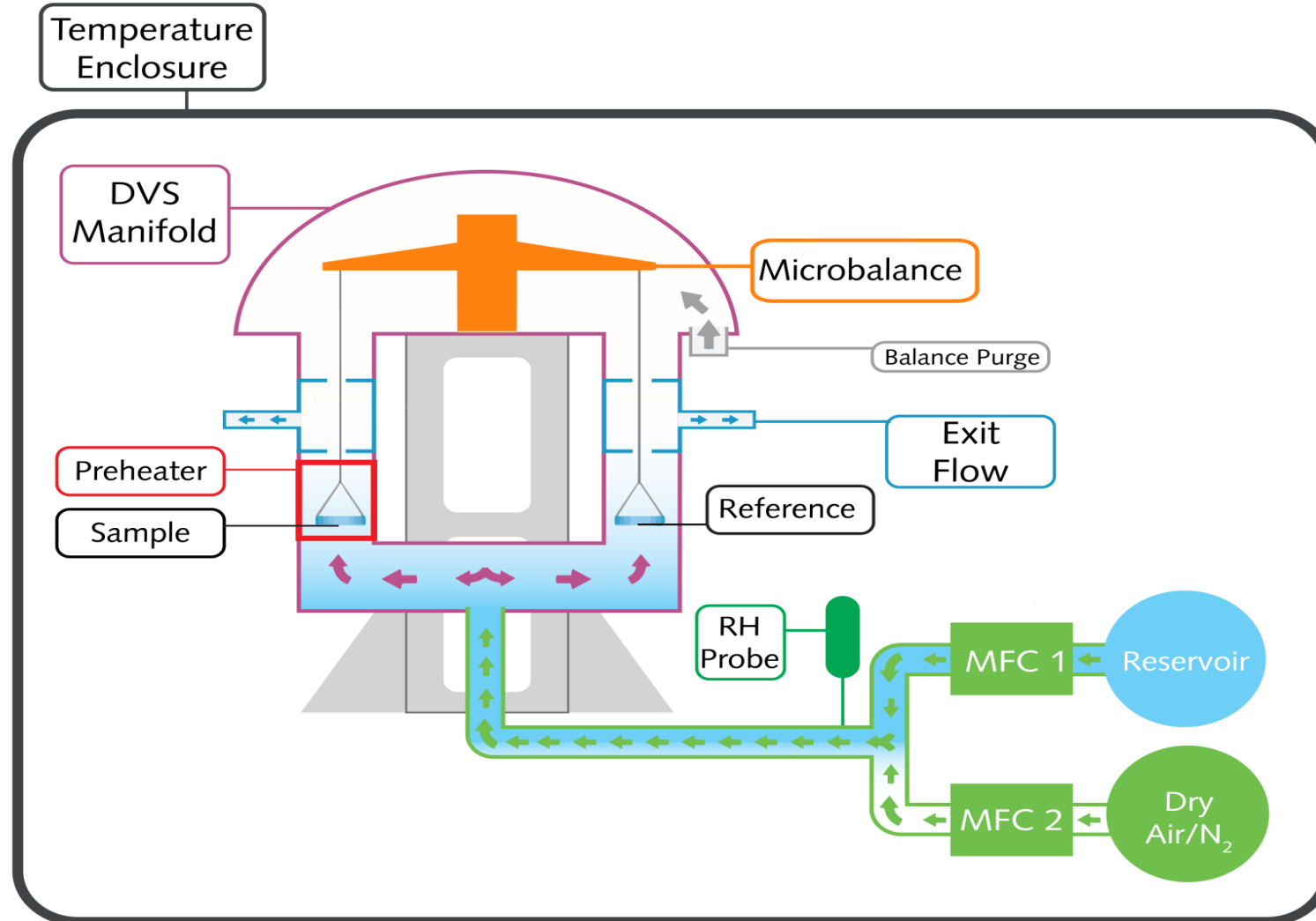
Two samples show great difference in moisture sorption capacity and kinetics.

Water Sorption Results – Isotherm Plots



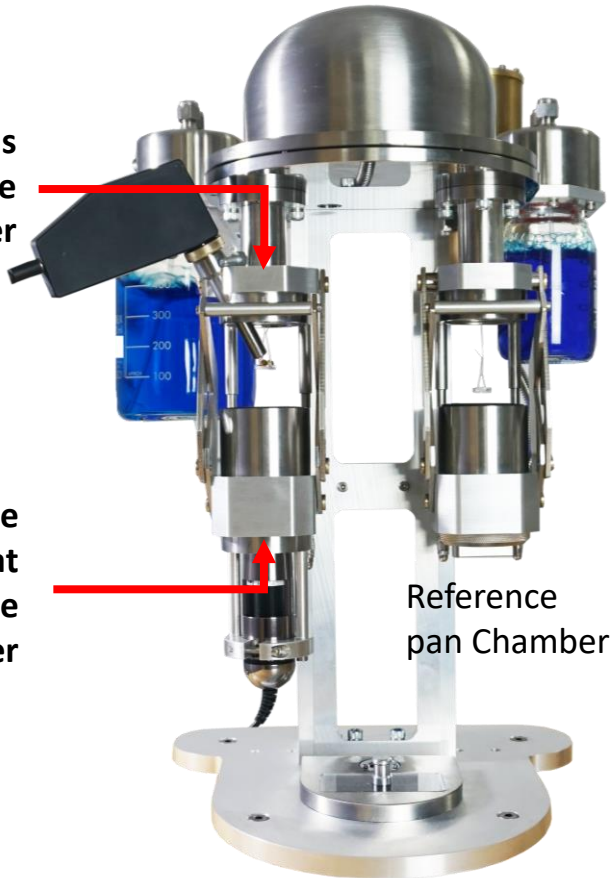
Easy access to moisture sorption / desorption isotherms and hysteresis measurements

Schematic of DVS Adventure Instrument



System Accessories

UltraBalance Enclosure



The Raman probe is installed at the top of the sample chamber

The camera or the preheater are installed at the bottom of the sample chamber

Reference pan Chamber

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



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DVS Product Line

DVS Intrinsic Plus

DVS Adventure

DVS Resolution

DVS Endeavour



DVS Discovery

DVS Carbon

DVS Vacuum



DVS Product Line

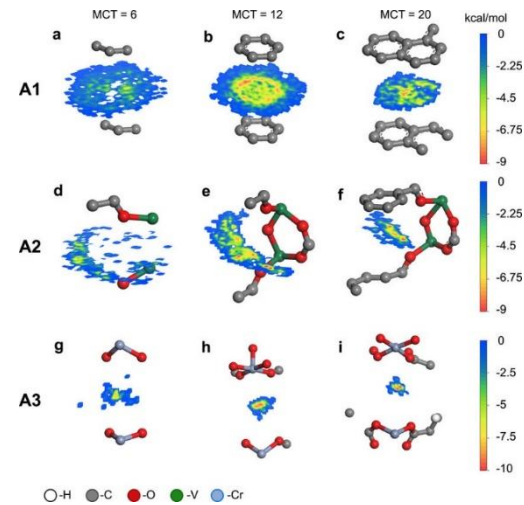
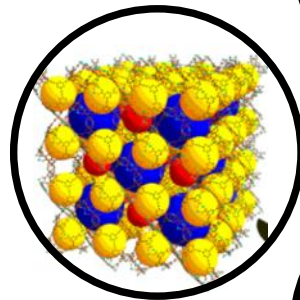
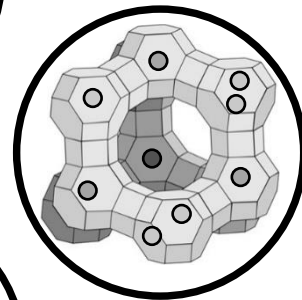
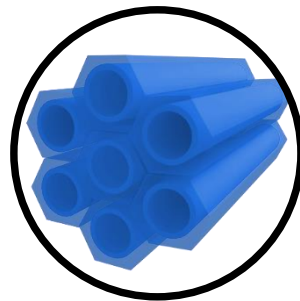
Available Features	DVS Intrinsic+	DVS Adventure	DVS Resolution	DVS Endeavour	DVS Discovery	DVS Carbon	DVS Vacuum
Temperature range (°C)	20-40	5-85	5-85	10-70	10-70	5-85	10-70
Optional in-situ sample preheater (°C)		200	200	200	200	200	400
Simultaneous sample measurement	1	1	1	5	2	1	1, 2
Co-adsorption of two molecules			2 vapors	2 vapors		H ₂ O/CO ₂	2 gases/vapors
Water vapor sorption kinetics & isotherms	✓	✓	✓	✓	✓	✓	✓
Carrier gas – atmosphere flow based	✓	✓	✓	✓	✓	✓	
200x color video/microscopy accessory		✓	✓	✓	✓		
Fiber optic/Raman spectroscopy accessory		✓	✓	✓	✓		
Organic vapor sorption kinetics & isotherm			✓	✓	✓		✓
Speed of sound organic vapor sensor			✓	✓	✓		
CO ₂ gas sorption						✓	✓
Compatible with NH ₃ , SO ₂ , H ₂ S							✓
High vacuum capabilities							✓

Advanced solutions for assessing the performance of materials for CCUS



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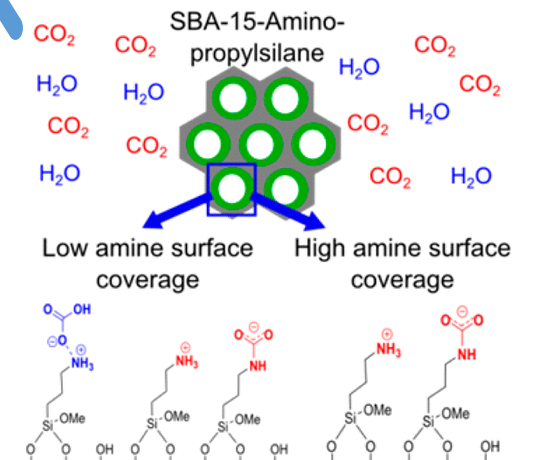
Solid sorbents for CCUS



Boyd, et al. (2019). Data-driven design of metal-organic frameworks for wet flue gas CO₂ capture. *Nature*. 576. 253-256. 10.1038/s41586-019-1798-7.

Physisorption

Chemisorption



Didas et al, *The Journal of Physical Chemistry Letters* 2014 5 (23), 4194-4200, DOI: 10.1021/jz502032c

- Three powerful instruments for screening materials
 - Fundamental properties with the **DVS Vacuum**
 - Real-world conditions with the **DVS Carbon**
 - Breakthrough analysis with the **BTA Frontier** and **MPA Horizon**



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Powerful instruments for realistically evaluating different stages and materials for CCUS

Fundamental

Process



DVS Vacuum

Gravimetric Sorption Analyzer
Any probe: Water, Organics, Gases

Fundamental properties, uptakes, enthalpy of sorption, surface area, vacuum activation



DVS Carbon & Carbon⁵

Gravimetric Sorption Analyzer
CCUS: CO₂ and Water

Real-world conditions, independent humidity control, cycling, temperature-swing programs



BTA Frontier & Horizon

Packed Bed & Membrane Analyzer
Flexible: CO₂, Water, Organics, Gases

Multicomponent adsorption uptakes, variable flow mixing, breakthrough point determination



DVS Vacuum

DVS for Fundamental Studies

DVS
VACUUM



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Key features of the DVS Vacuum



- Gravimetric sorption analyzer
 - Small sample sizes needed
 - Pure flow system – no carrier
- Thermostatted chamber – no condensation
- Vacuum and high temperature capable
- Dynamic and static operations modes
- Multi-vapour and/or gas injection (2 ports)
- Single or dual sample configurations
- *In-situ* activation / drying kinetics
- Real-time adsorption/desorption kinetics
- Adsorption / desorption isotherms
 - (T constant, P variable)
- Adsorption / desorption isobars
 - (T variable, P constant)
- Cycling and complex programs

DVS Vacuum Summary

A powerful gravimetric instrument with vacuum capabilities, ideally adapted to recording pure isotherms (CO_2 , SO_2 , H_2O etc), fundamental material parameters and gauging impact of one component on another in co-sorption.

DVS CARBON

Gravimetric CO₂ / H₂O Analyzer



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Key features of the DVS Carbon



- **Gravimetric dynamic sorption analyser**
 - Flow system – uses an inert carrier gas at different flowrates
 - Minute (mg) to medium (g) sample sizes - μg changes in mass
 - Kinetics are available by default
- **CO₂, humidity, temperature, independently controlled**
 - Single – or multiple components
 - Cycling or complex programs are easily created
 - Concentration and temperature set in steps or ramps
- **Concentration & temperature ranges for CCUS applications**
 - Enclosure and *in situ* heating/activation - from 5 °C up to 300 °C
 - High (%) or low range (ppm) CO₂ can be accurately controlled thanks to integrated sensors.
 - High range of humidity – up to 98% to 85 °C
 - Atmospheric operation
- **Options for extending functionality to suit your needs**
 - Hyphenation options – Raman, NIR, camera, outlet sensors
 - Expanded mixing and accuracy range, as DVS Carbon Advanced
 - High throughput option – 5 simultaneous measurements

DVS Carbon Examples

- Single component water uptakes
- Single component CO₂ uptakes
- 400 ppm CO₂ with varying humidity
- Independent concentration control
 - 30 vol% CO₂ with varying humidity
 - 30 RH% with varying CO₂ concentration

Summary

A powerful gravimetric instrument, ideally adapted to screening CO₂ capture materials both in PCC and DAC conditions.

BTA Frontier

Self-contained competitive sorption
breakthrough analyzer



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Key Features of the BTA Frontier



- Packed bed breakthrough instrument
 - Flow system – uses an inert carrier gas
- Determination of true multicomponent sorption
 - Generates and measures complex mixtures
 - Dedicated sensors for CO₂, H₂O and organics + TCD
- Optimized for small sample amounts 10s of mg to below 1 g
- In situ activation and regeneration
 - Under inert or process gas
 - Local oven for high temperatures
- Automatic dead volume determination
 - Helium injection during experiments
 - Column bypass system
- Complex methods available: controlling concentration flowrate, temperature

Summary

A batteries-included BTA instrument, capable of recording true multicomponent data and kinetic information with CO₂, H₂O, VOCs and more.

Thank you for Listening!

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