

New Dynamic Vapor Sorption Innovation in Sorption Science

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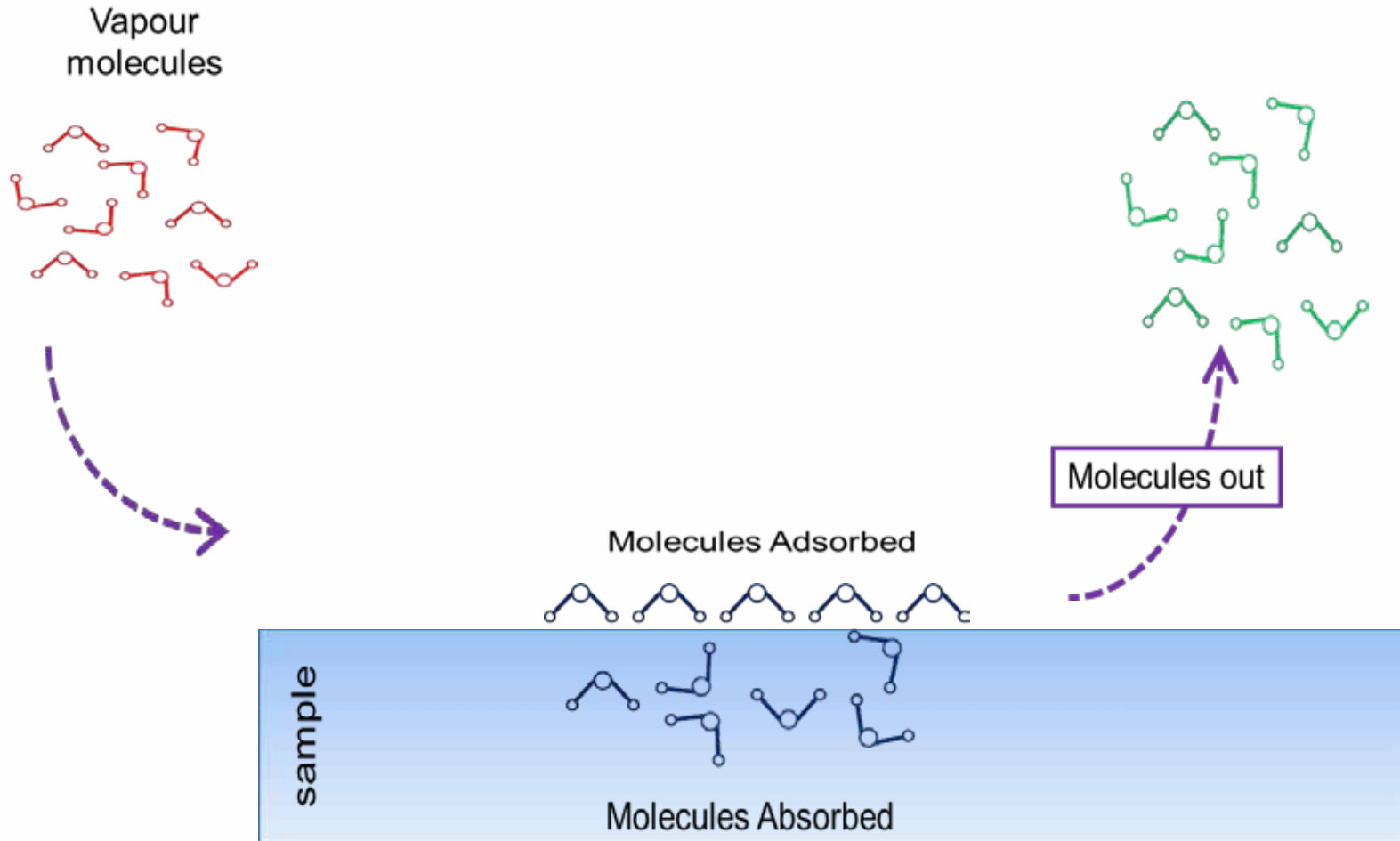


Surface Measurement Systems develops and engineers techniques and instrumentation for physico-chemical characterisation of complex solids. We are the world leaders in Dynamic Vapor Sorption technology and Inverse Gas chromatography instrumentation, providing professional world-class scientific and technical support for our international customers.

Our range of characterization instruments continues to help solve difficult problems in the pharmaceuticals, biomaterials, polymers catalysts, chemical, cosmetics and food industries, and are used by hundreds of leading laboratories and universities throughout the world.

1. How does my material interact with moisture or solvents and temperature in the vapour phase?
2. Stability, Performance and Processing issues: Reversible and Irreversible effects of moisture
3. Create Moisture Isotherms – i.e. Equilibrium moisture content as a function of %RH
4. Heterogeneity? – Identify the Heterogeneity of a sample batch
5. Homogeneity? – Identify variance within one sample
6. Kinetics – Moisture transport properties, how fast or slow?
7. Energy – How strongly is the moisture bound to the material, surface or bulk?
8. Identify & Characterise Phase Transition/Changes, e.g. polymorphs, amorphous stoichiometry
9. Hydration and Solvate Formation
10. Drying Analysis
11. Diffusion and Activation Energy
12. Heat of Sorption
13. Moisture Uptake/Content? i.e. how much moisture/vapour is taken up or release

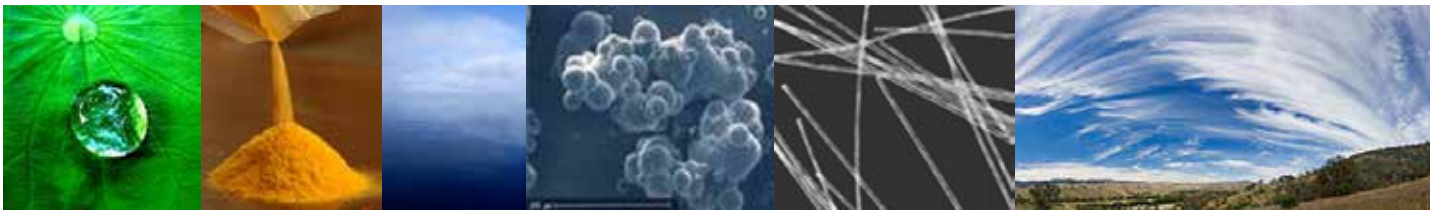
Molecules as a Probe



Water-solid interactions important for wide range of industries:

- Food
- pharma
- proteins
- fuel cells

Accurately determining water sorption isotherms critical for proper development and storage of these materials



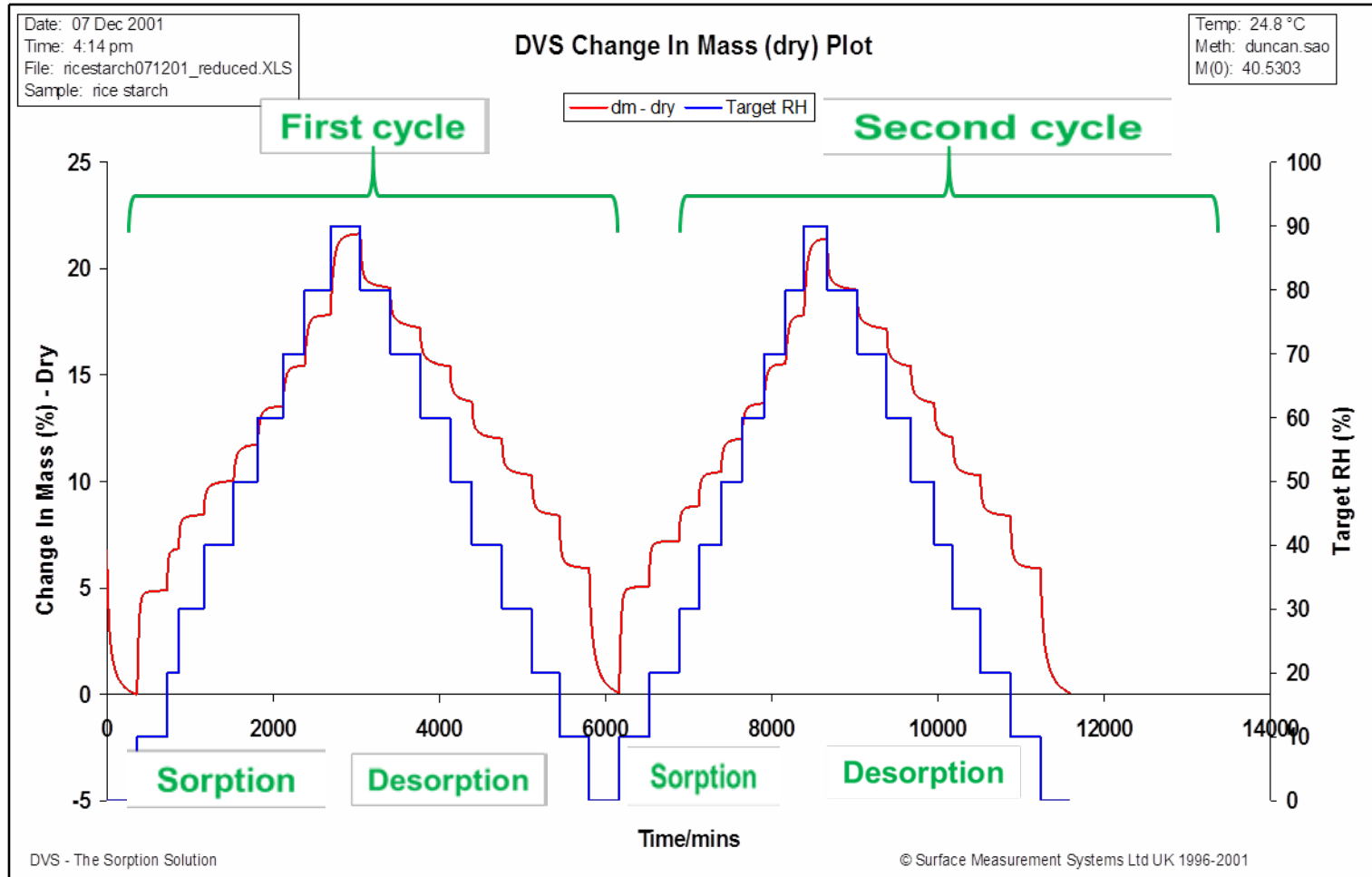
Where can Vapour Sorption occur?

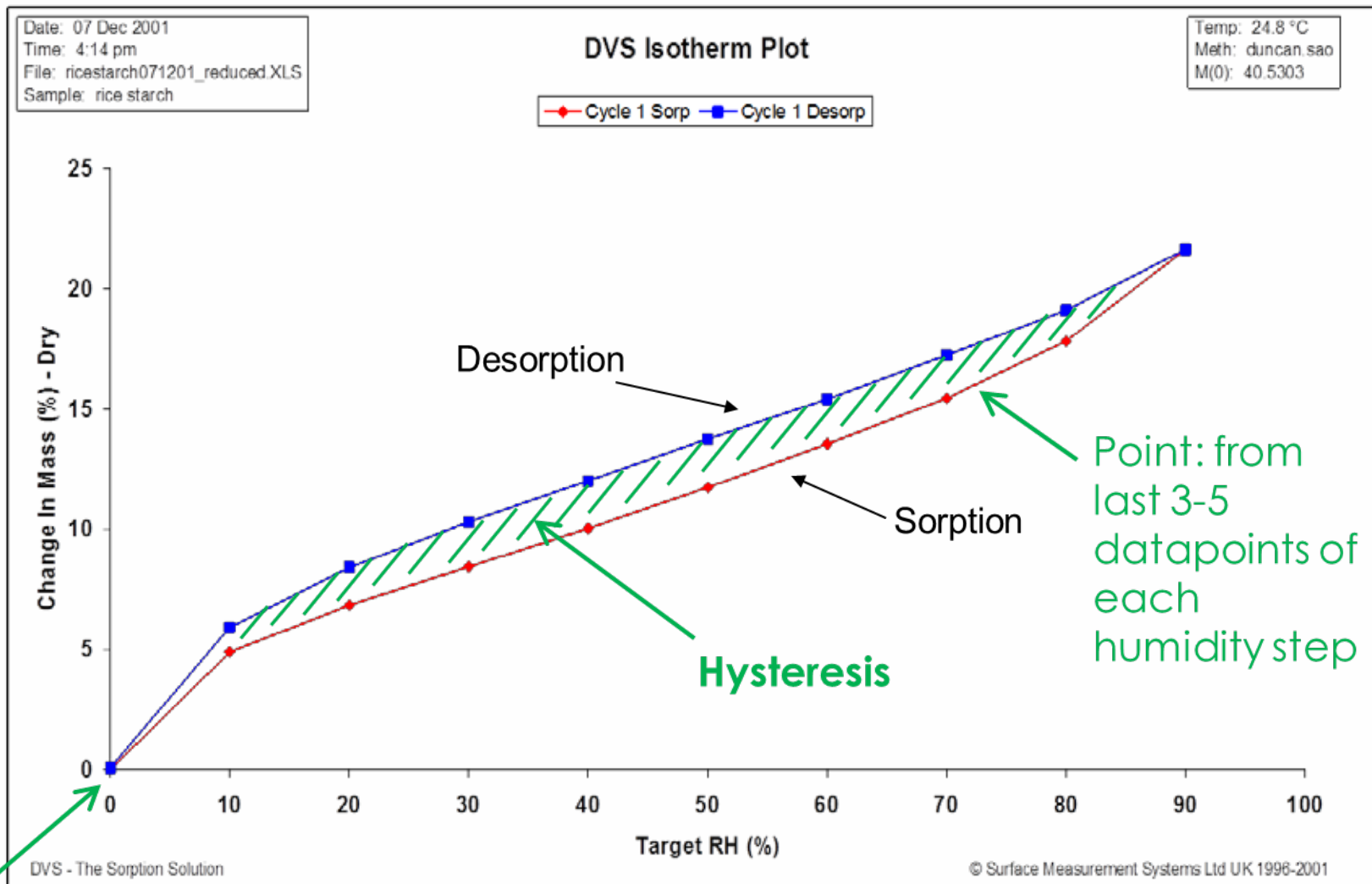
- On the surface
- In pores – micro/meso?
- Between the particles (condensation?)
- Sorbed into the bulk
- Chemically reacted (hydrate formation)?

What can vapour sorption tell me?

- The stability of materials at different vapour concentrations.
- Accurately determining water sorption isotherms is critical for proper development and storage of these materials

DVS – Kinetics of Moisture Sorption of Rice Starch



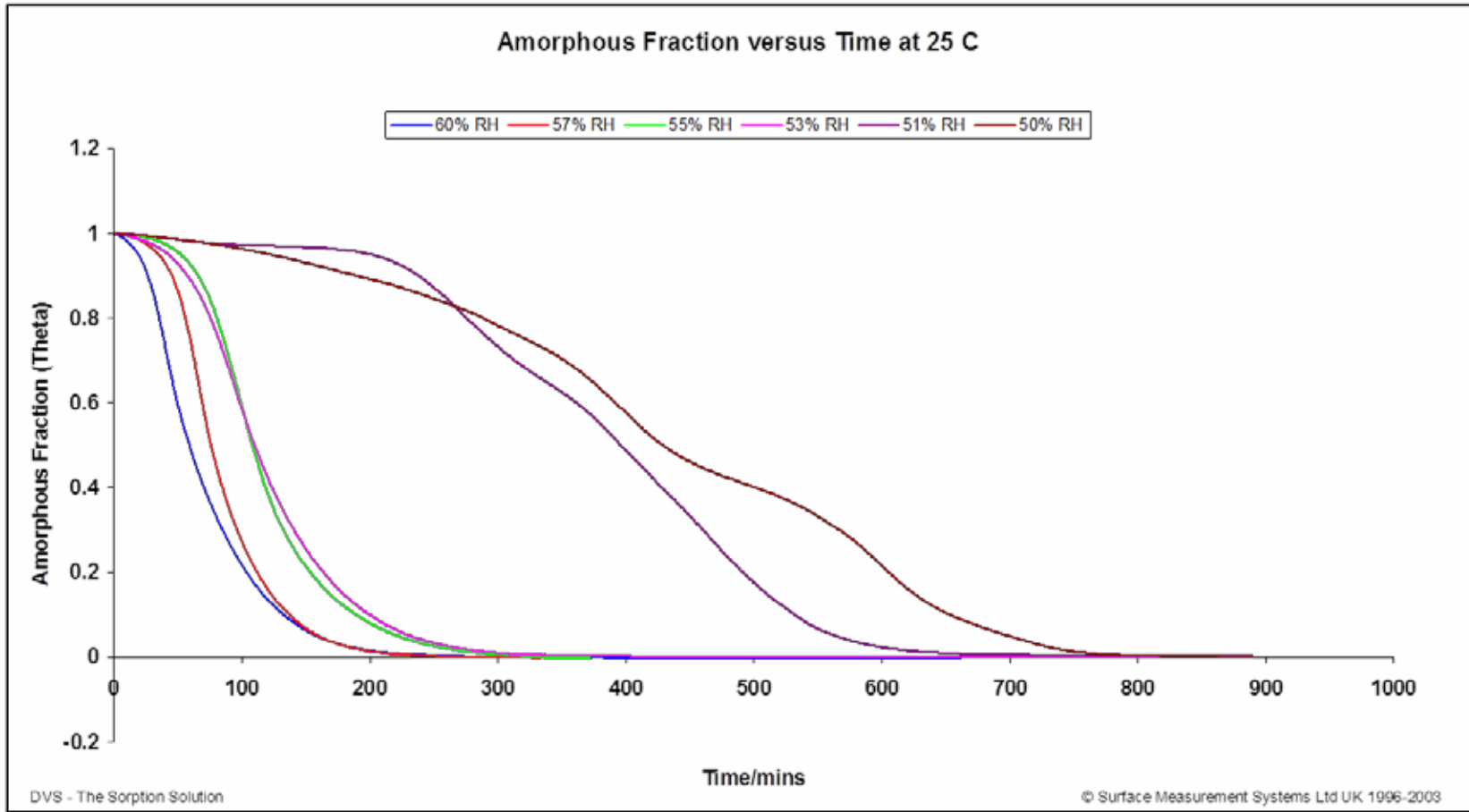


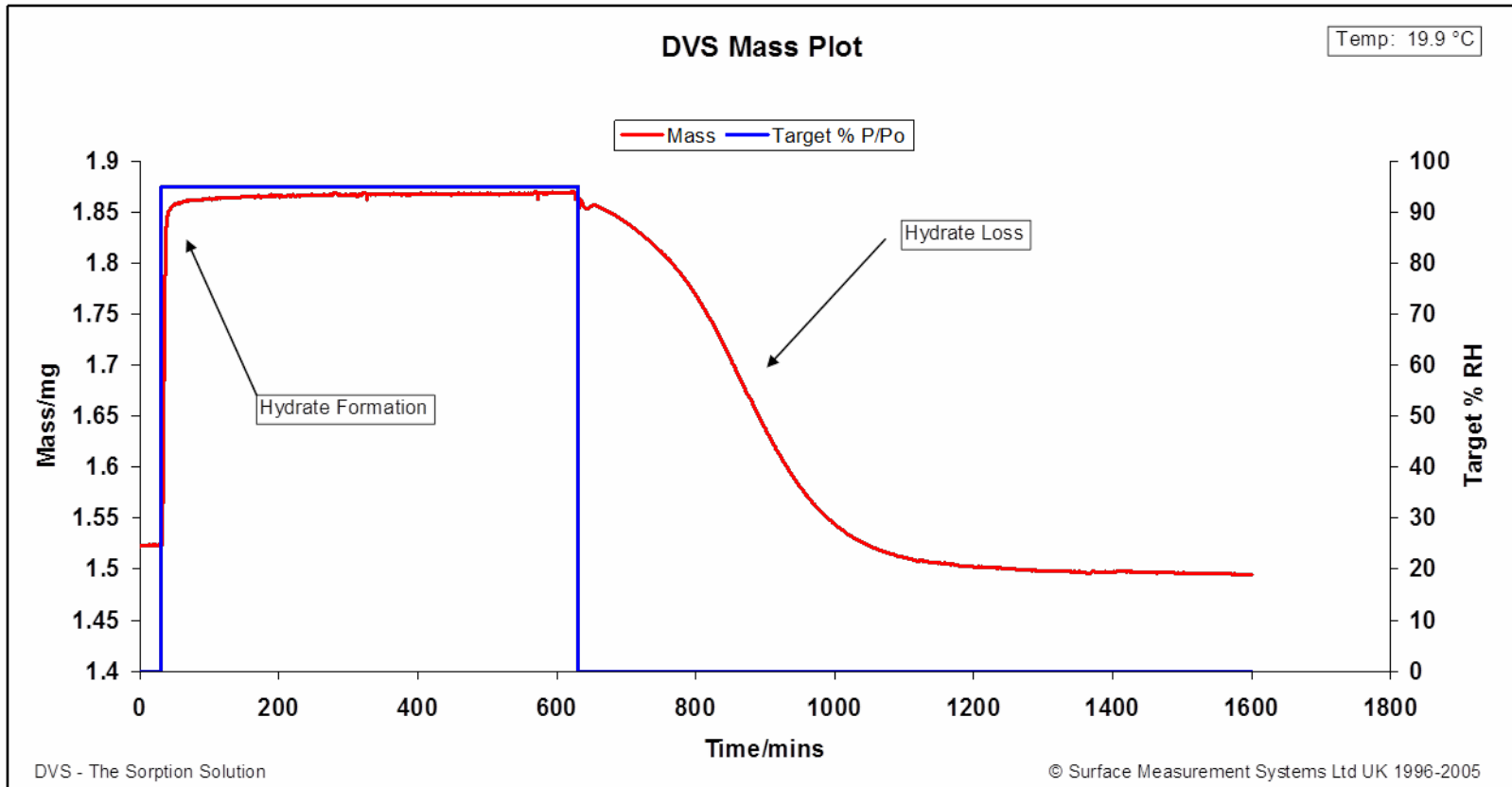
Back to 0 → reversible

Glass transition for spray-dried lactose at 25 °C can be found at 30 % RH.

Crystallisation for spray-dried lactose at 25 °C can be found at 58 % RH.

If temperature *and* humidity is kept constant (between 30% and 58% RH at 25 °C) crystallisation is kinetically controlled and can be followed by loss of weight due to water desorption.





NEW DVS Products



DVS
ADVENTURE



DVS
RESOLUTION



DVS
ENDEAVOUR



DVS
INTRINSIC

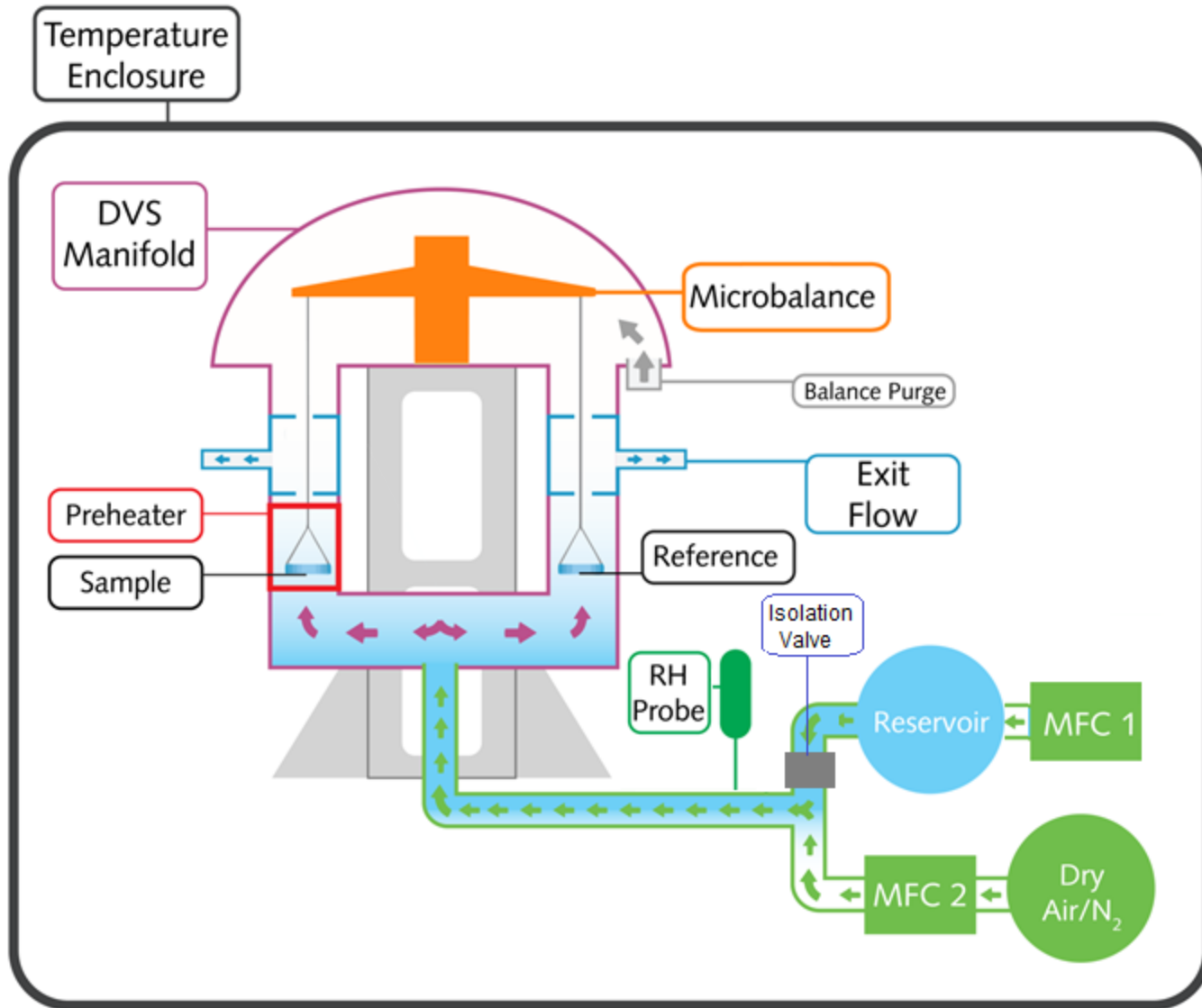


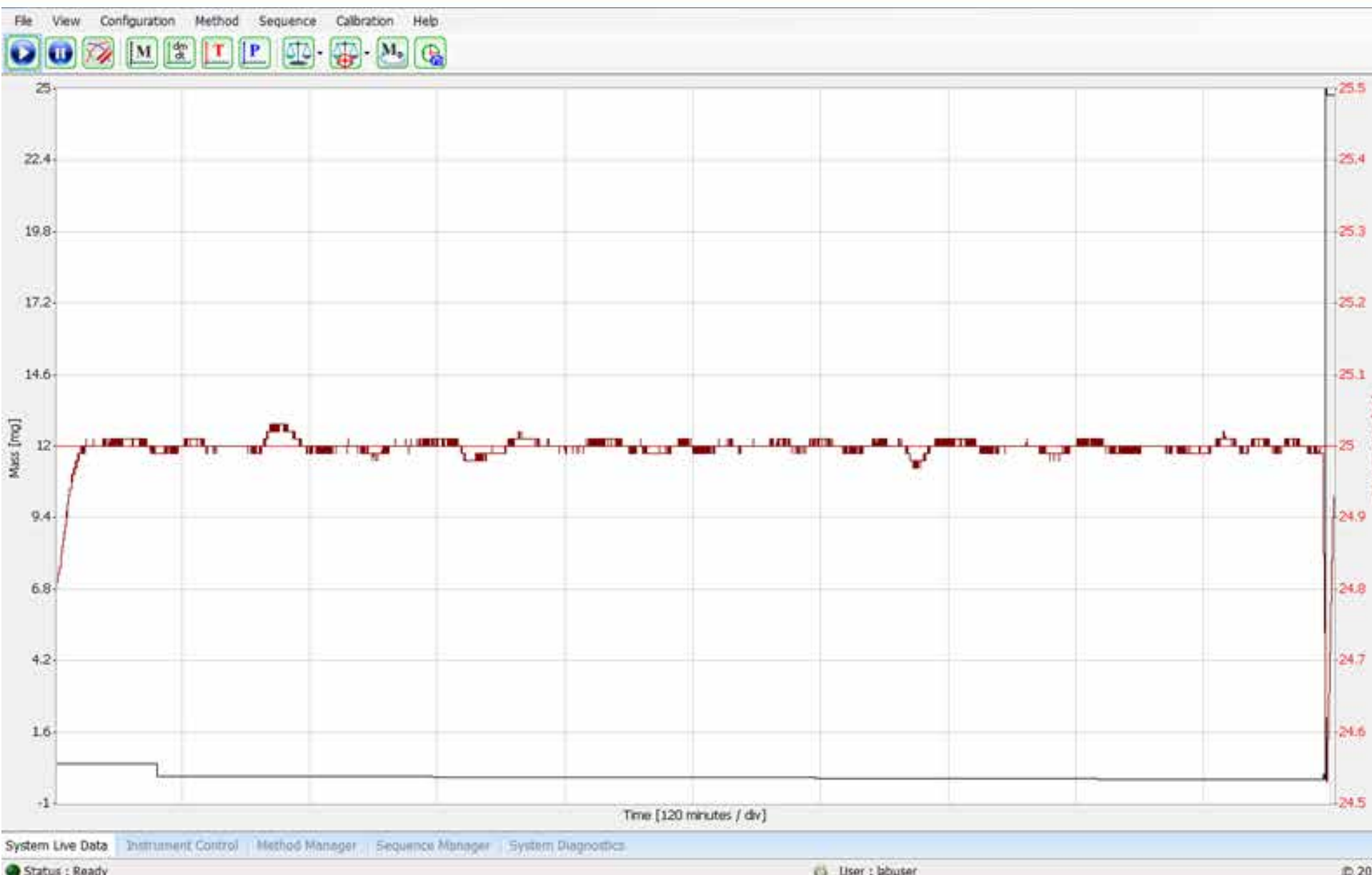
DVS
VACUUM

- § Most advanced water sorption analyser in the market
- § Temperature range: 5°C to 85°C
- § Temperature stability: $\pm 0.1^\circ\text{C}$
- § Optional preheater for sample drying to 300C
- § Wide humidity range typically 0-98% RH
- § Humidity stability $\pm 0.3\%RH$
- § True0™ drying at 0.0% RH
- § Active PID control of relative humidity
- § Automated isotherms and isohumes
- § Preheater, Raman, NIR and video options
- § Upgradeable to other DVS's in family
- § Small laboratory bench footprint (18")



Schematic of DVS Adventure





SMS UltraBalance1™

Sample mass: between 1 and
1500mg

Resolution (precision): 0.1 μg

Peak to peak noise: $\leq 0.2 \mu\text{g}$

Mass change: $\pm 150\text{mg}$

Stability (drift): $\leq 5\mu\text{g}$

(24 hrs @ 25°C and 0% RH)

SMS UltraBalance2™

Sample mass: between 10 and
5000mg

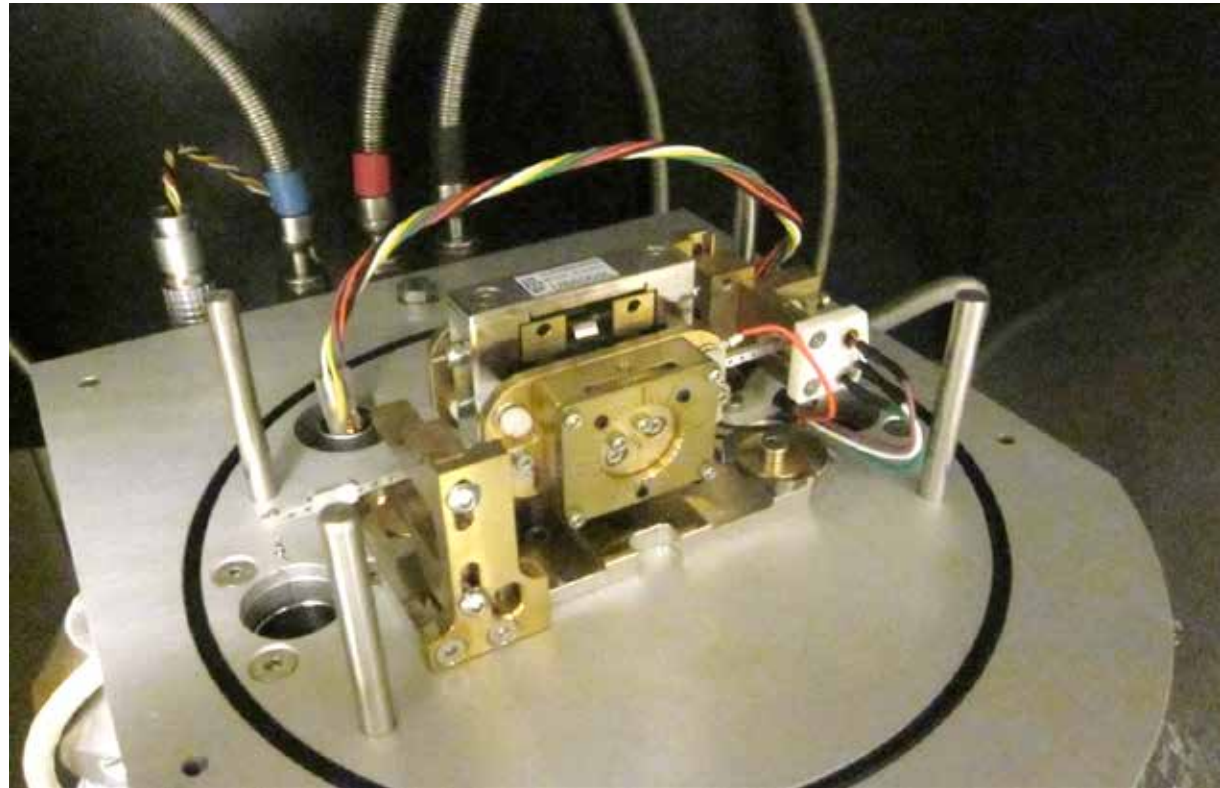
Resolution (precision): 1.0 μg

Peak to peak noise: $\leq 2.0 \mu\text{g}$

Mass change: $\pm 1000 \text{ mg}$

Stability (drift): $\leq 50 \mu\text{g}$

(24 hrs @ 25°C and 0% RH)



SMS Ultra-Balance Performance

The DVS Adventure is capable of measuring mass changes at a resolution of 0.1mg with peak to peak noise of less than or equal to 0.2 µg. The Surface Measurement Systems Ultra-Balance™ is unrivaled in its precision and accuracy.

Company Confidential

Integrated 200x Microscopy/ Raman or NIR Hyphenation

IsoTherm Segment Editor

Method Stage Type

Step Time [min.]: 2

Step dm/dt [%/min.]: 0.002000

Incubator Temp. [°C]: 25.0

Options

Take Video Image

Use Raman

Cycle

Half Cycle Cycles: 1

Full Cycle # Stages: 1

Multiple Cycles

Preheater Temp. [°C]: 0.0

Active Reservoir: Reservoir A

Total Gas Flow [sccm]

(A): 200

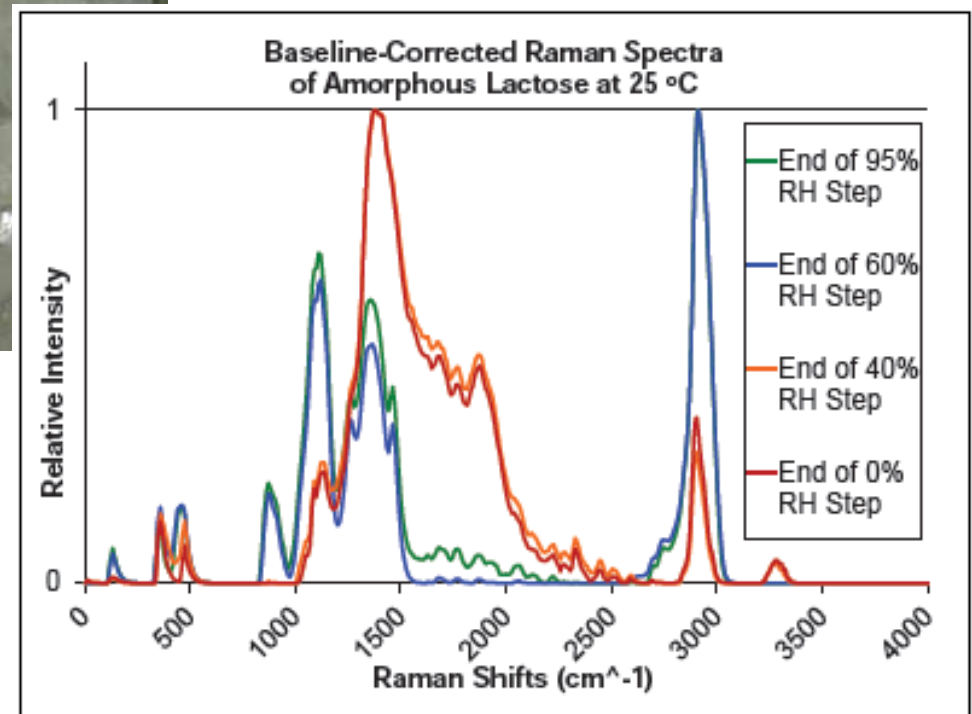
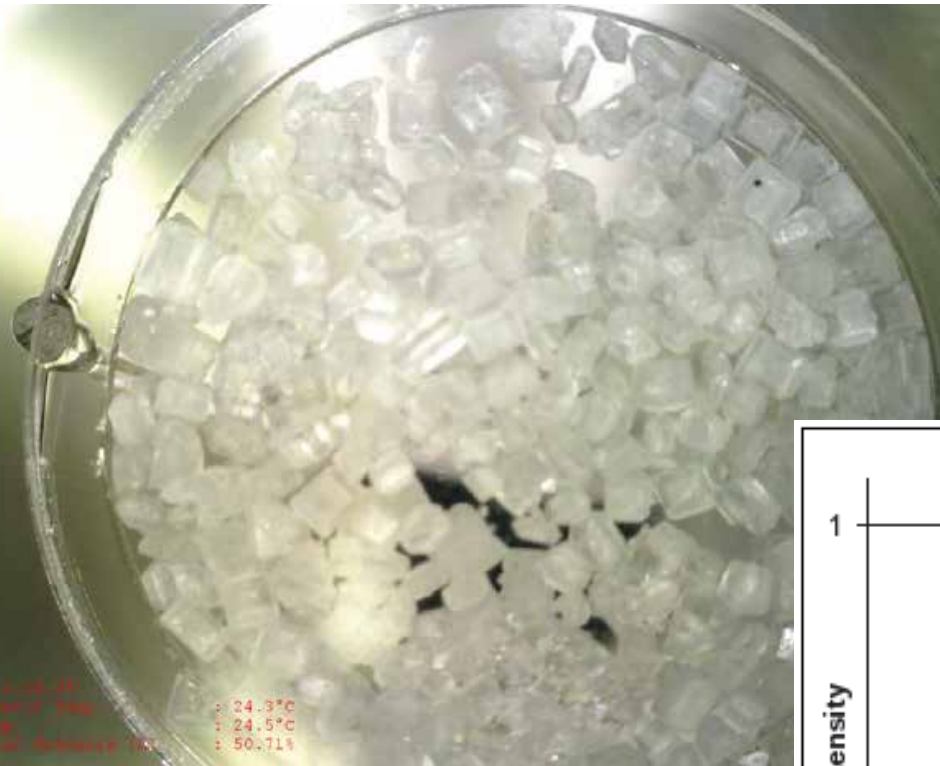
(B): 200

Half Cycle Definition

Seg. #	Start Partial Pressure (A) [%]	Stop Partial Pressure (A) [%]	Start Partial Pressure (B) [%]	Stop Partial Pressure (B) [%]	Step Size Partial Pressure (A) [%]	Step Size Partial Pressure (B) [%]
1	0.00	0.00	0.00	0.00	5.00	5.00

OK Cancel

Integrated 200x Microscopy/ Raman or NIR Hyphenation



DVS-Resolution

Dual Vapor Sorption Analyzer

Capabilities:

- Competitive sorption of two vapors
- Organic and Water vapor sorption kinetics
- Organic vapor sorption in a fixed RH background
- Real time partial pressure measurement and control
- Water vapor sorption isotherms from 5 to 85 °C
- Organic vapor sorption isotherms from 5 to 50 °C
- In-situ drying of samples to 300 °C
- Optional Fiber Optic Raman
- Optional Color Video Microscopy
- True0™ drying at 0.0% RH



Schematic of DVS Resolution

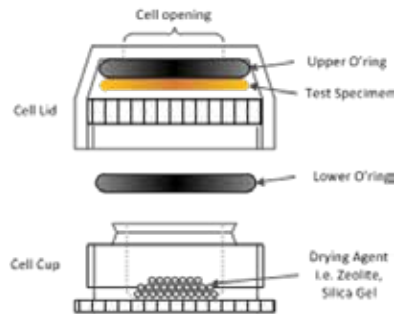
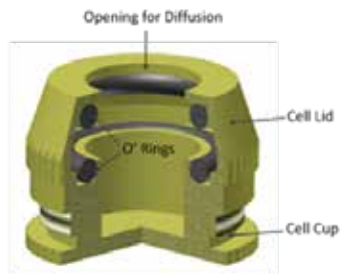
Pre-Heater to 300 °C



300C Preheater

Payne Cell for Diffusion and Permeability

- **Payne Cell** for WVTR measurements, water activity, diffusion and permeability of films, and other applications



**Payne Cell for use with DVS Ultrabalance1
(Part # P21MA031)**



The opening diameter is 6 mm.
Mass empty, with O-rings: 400 mg
Mass with 200+ mg Zeolite or Water: approx. 600 mg.

**Large Payne Cell
for use with DVS Ultrabalance2
(Part # P21MA052)**



The larger opening diameter is 12 mm.
Mass empty, with O-rings: 1.75 g
Mass with 200+ mg Zeolite or Water: approx. 1.95 g

DVS Consumables

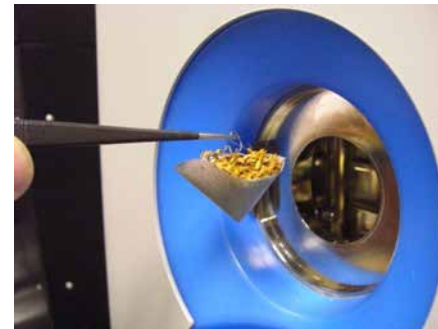
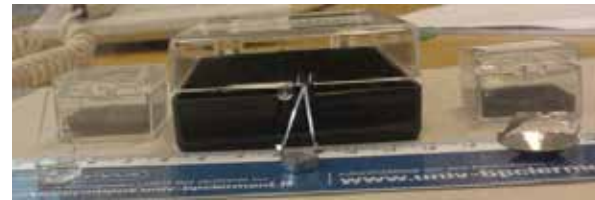
- **Sample pans** (different sizes and versions to accommodate all sample types)



Glass pans
13 mm quartz pan (part #QP13NH) (Left TOP)
13mm video quartz pan (part #QP13VID) (Left BOTTOM)
13mm Stainless Steel mesh (BELOW)



(a) Stainless Steel Pans (a) and Aluminium inserts (b).



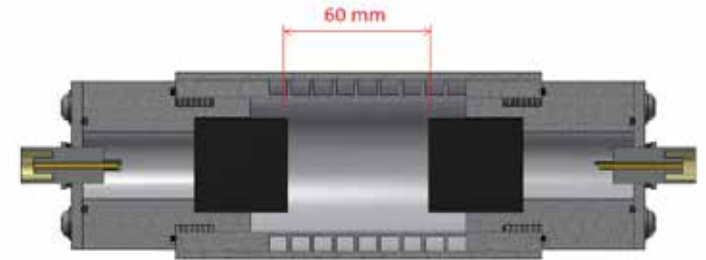
Speed of Sound - Methodology

Configuration

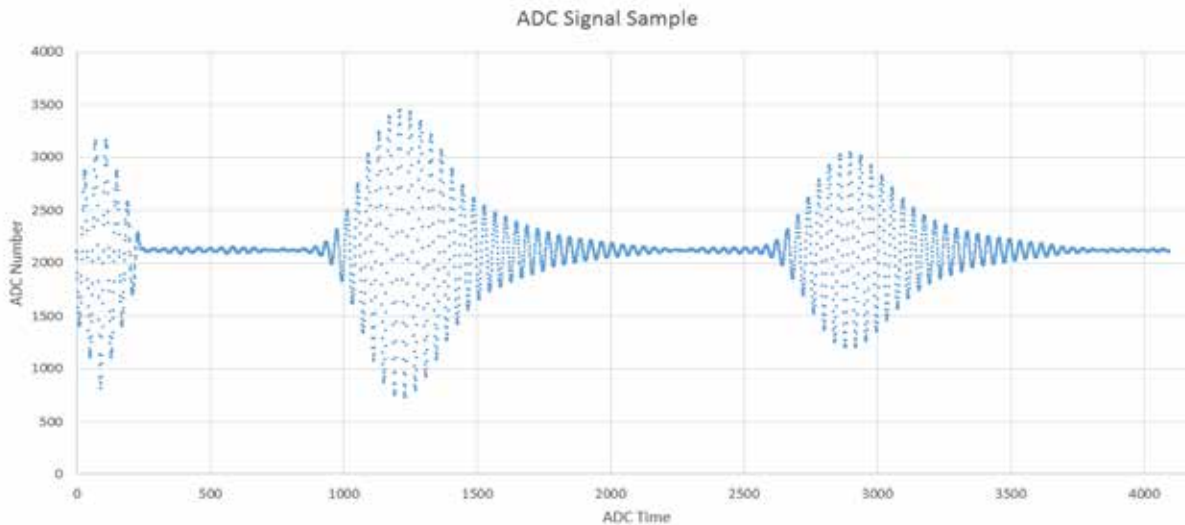
- Pitch-Catch

Algorithm

- Parabola curve-fitting & Zero-crossings



Sectioned View



Speed of Sound

The SMS Ultrasonic Sensor is a novel method of measuring the composition of gases and vapours. The speed of sound through a medium is a fundamental property of the medium and is dependent on composition, temperature and pressure. The SMS Ultrasonic Sensor measures the transit time of ultrasonic pulses through a sample of a gas/vapour mixture before the mixture interacts with the sample. By measuring the speed of sound through the medium the exact relative proportions of the composition can be determined. The advantage of the Ultrasonic sensor over traditional measurements, such as chilled mirror devices (DPAs), is that the Ultrasonic sensor is non-interacting (does not condense vapours) and can work on a wide range of solvents (high degree of solvent compatibility).

SOS Sensors: Calibration

The following solvents are calibrated in air from typically 15C to 50C:

Acetone	Hexane
Chloroform	Isopropanol
Cyclohexane	Methanol
<u>Decane</u>	<u>Methyl Ether Ketone</u>
<u>1,2 Dichloroethane</u>	<u>Nonane</u>
<u>DiChloromethane</u>	Octane
Ethanol	Toluene
Ethyl acetate	Water
Ethyl benzene	<u>m-Xylene</u>
Heptane	<u>p-Xylene</u>

The following solvents are calibrated in nitrogen at 50C:

<u>m-Xylene</u>	<u>p-Xylene</u>
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The following solvents are calibrated in air at 25C*:

<u>Pinene</u>	Limonene
<u>Hexanal</u>	Butyl amine

The following gases are calibrated in from 10C to 50C*:

CO ₂ in air	CH ₄ in air
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DVS-Endeavour

- 5 High precision Ultrabalances offer true high parallel performance- 5 samples at once- under identical humidity and temperature conditions.
- All 5 samples zone can be accessed individually
- Can run 1 to 5 samples as required by user
- All balances can be tared and calibrated simultaneously
- 10 to 70C temperature range
- Factory calibrated capacitance humidity probe for
 - 25C , 35C, 45C
 - At 0% to 90% relative humidities in 10% steps
- SOS Sensor for organic vapours and gas mixtures
- Laboratory bench footprint 450mm (18") plus PC



Schematic of DVS Endeavor

DVS-Endeavour



Common Software Platform

- § Common software platform used for all new DVS models
- § Easy to use wizards for balance calibration
- § Live view of all data
- § Isotherm, Isobar, and Ramping Experiments
- § Quick start method development
- § Run methods and sequences
- § Integrated fiber optic Raman spectroscopy capture
- § Integrated color video microscopy capture
- § Full system diagnostics

DVS
ADVENTURE

DVS
RESOLUTION

DVS
ENDEAVOUR

iGC-SEA Introduction

- **Gas phase injection** (like Headspace) - 12 vapor reservoirs (50 ml)
- **Carrier gas** is helium
- 2 column position oven design: **20 to 150 C**
- **Background Humidity Controller**
- Flame Ionization Detector (FID)
- **User Friendly** Control and Analysis **Software**



Carbon fiber cotton hair granules powder medical metal implants



Safety Features:
Hydrogen Leak & Organic Vapor Leak Detectors

Thank you!

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