

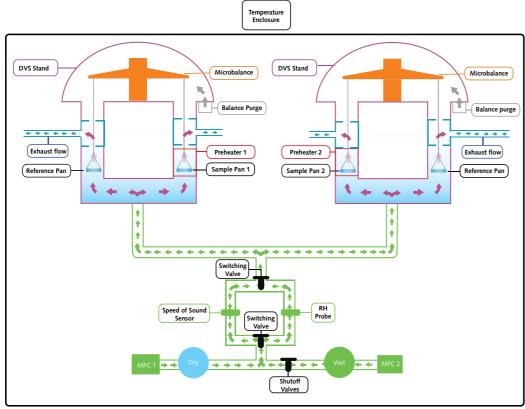




The World's Most
Advanced Dual Balance
Gravimetric Vapor
Sorption Instrument

- 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

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_a 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

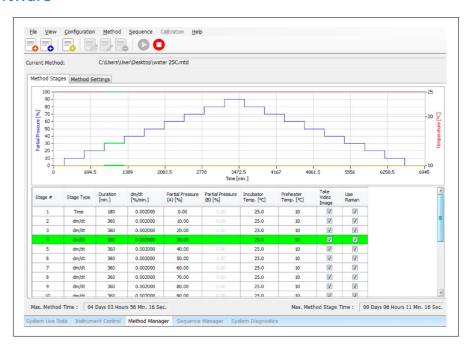
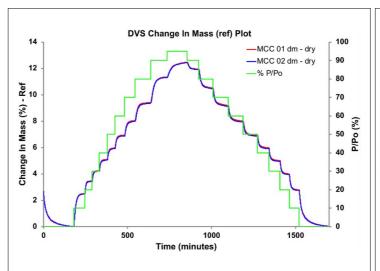


Figure 1. The above graph shows the method panel within the method manager. It displays numerically and graphically the current method for a water sorption experiment at 25 °C. The active stage of the ongoing experiment is highlighted in green. Figures 2 and 3 (below) are typical data generated by this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

Analysis Software

Water Sorption Data





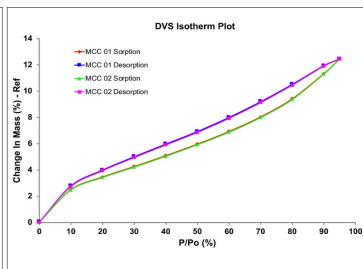


Figure 3. Water sorption isotherms of two samples of microcrystalline cellulose (MCC) at 25 $^{\circ}$ C

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

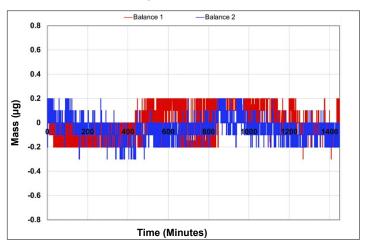


Figure 4. DVS Mass Baseline Stability Plot Over 24 Hours

- 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

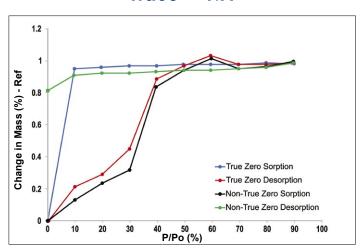


Figure 5. Comparison of Naloxone Hydrochloride Dihydrate Water Sorption Isotherms

- 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

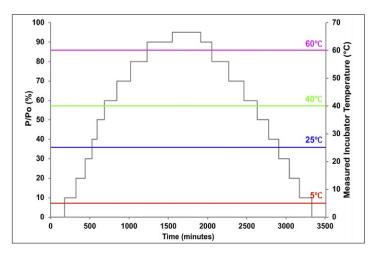


Figure 6. Partial pressure plots of Microcrystalline cellulose (MCC) at different temperatures

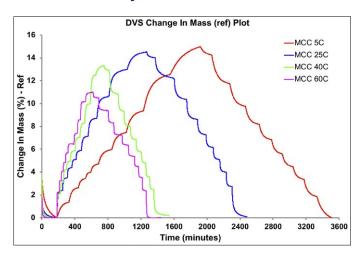


Figure 7. Sorption kinetic plots of Microcrystalline cellulose (MCC) at different temperatures

- 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:

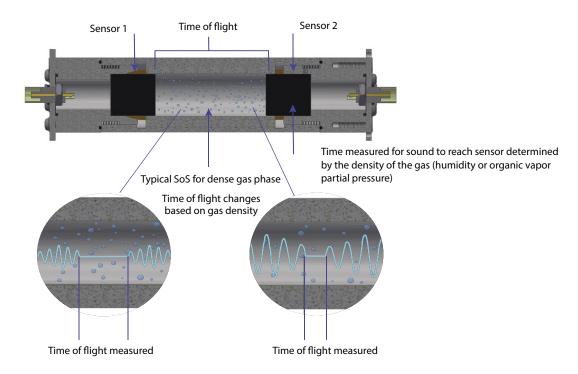
- (1) Humidity with controlled generation using a humidity sensor
- (2) Organic vapors with controlled generation using a proprietary Speed of Sound (SoS) sensor.

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/P₀ of target P/P₀

Partial Pressure Control

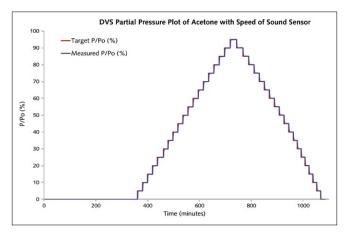


Figure 8. Acetone partial pressure Kinetic measured using the SoS Sensor during a DVS experiment

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

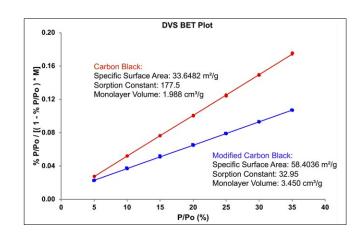


Figure 10. BET Linear Plots

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

Amorphous Content

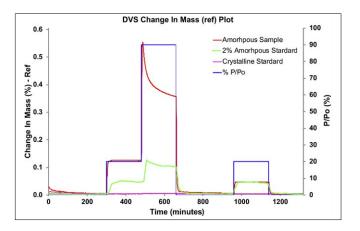


Figure 11. Amorphous content of Fluticasone Propionate

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

Diffusion Measurement

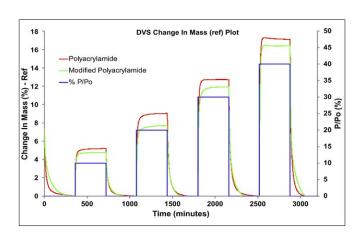


Figure 12. Kinetic plots of as-received and modified polyacrylamide samples at 25 °C

 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

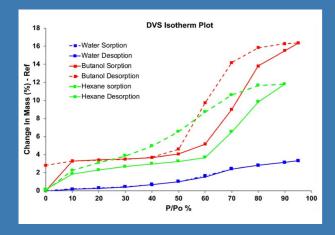


Figure 13. Isotherm plots of water, butanol and hexane sorption for titanuim oxide at 25 °C

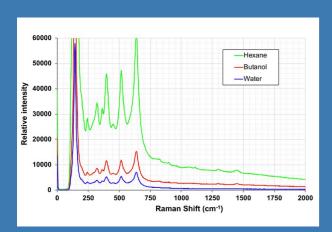


Figure 14. Raman Spectra of water, butanol and hexane sorption for titanuim oxide at 25 °C

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

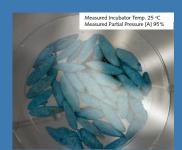


Cedar wood



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



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 $^{\circ}$ C 2

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

Relative humidity resolution $\pm 0.1\%$

Relative humidity stability ± 0.1% over 6 hours

RH range accuracy from 10 - 45 °C \pm 0.5%³

RH range accuracy from 45 - 70 °C \pm 1%³

Organic Vapor Generation and Measurement using Speed of Sound Sensor ⁴

Partial pressure range from 0 to 90% P/P₀ ⁵

P/P resolution ± 0.1%

P/P accuracy from 10 - 70 °C \pm 1% P/P $_{0}^{3,6}$

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

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.
- $^{\rm 2}$ System factory calibrated at 25 °C. Calibrations at other temperatures upon request.
- 3 SD (Standard Deviation) with % RH or P/P $_{
 m o}$ calibration performance based on SMS factory certified methods (Salt Calibration or others)

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 μ g
Balance noise: \leq 3 μ g

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
- WindowsTM 10

DVS Analysis Software

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

Software Options

Standard

- Control Software
- · Standard Analysis

Advanced

- Advanced Analysis Suite
- · Isotherm Analysis Suite

21CFR Part 11 software solution (optional)

- ⁴For 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







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