

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

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

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

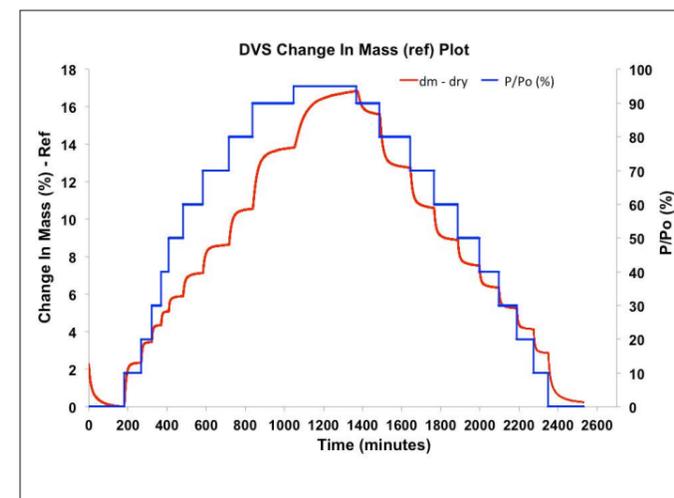


Figure 2. Water sorption kinetics of cellulose membrane at 25 °C

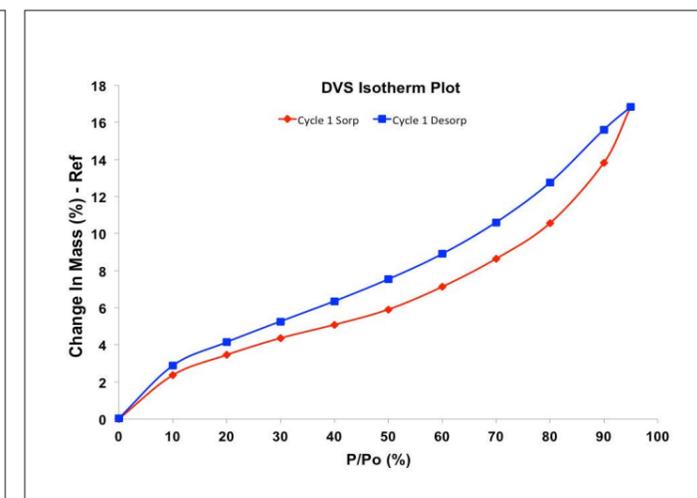
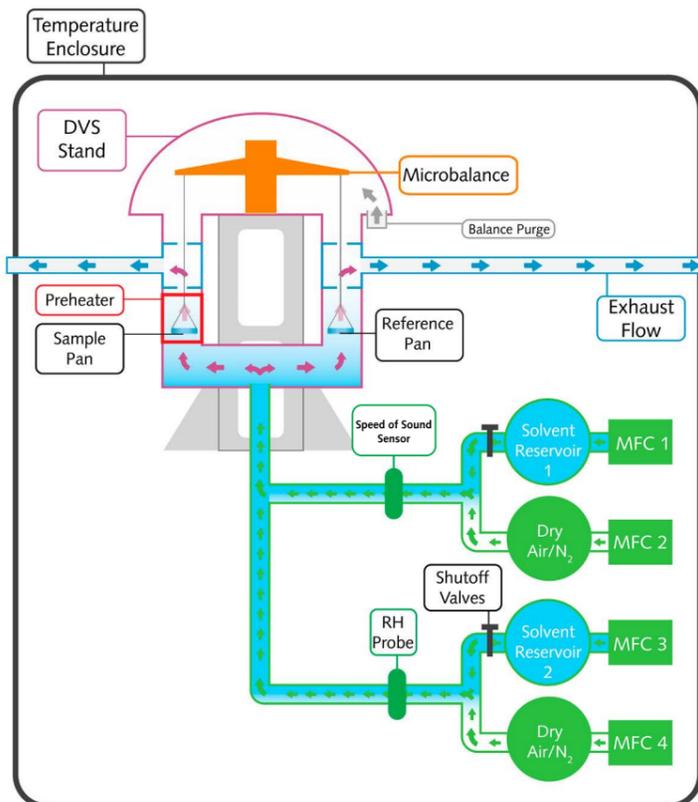


Figure 3. Water sorption isotherm of cellulose membrane at 25 °C



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

Outstanding Performance

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

Mass and Temperature Measurement

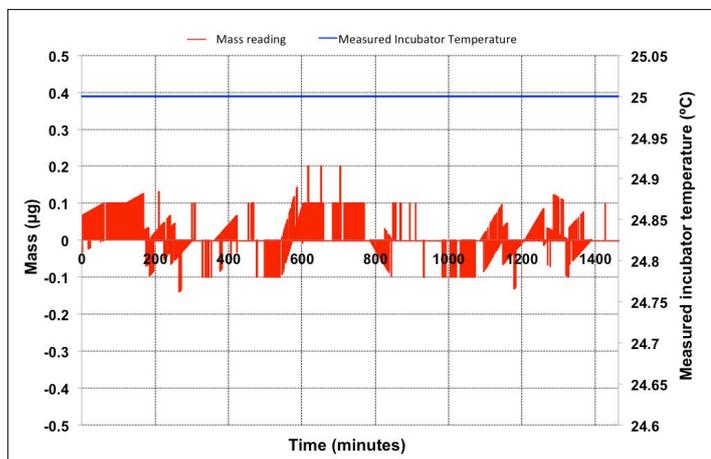


Figure 4. DVS Mass Baseline Stability Plot Over 24 Hours

True0™ RH

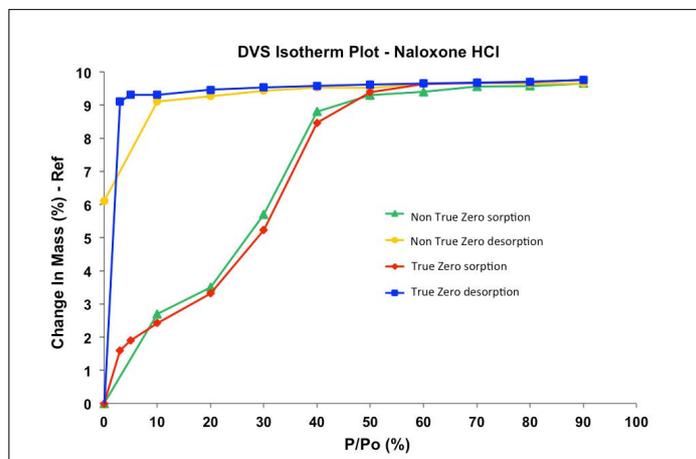


Figure 5. Comparison of Naloxene Hydrochloride Dihydrate Water Sorption Isotherms

- 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)

- 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

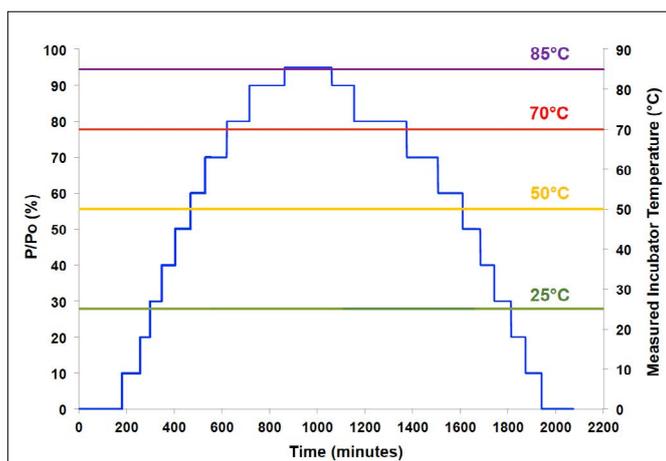


Figure 6. Kinetic plot of partial pressure (or relative humidity) at different temperatures*

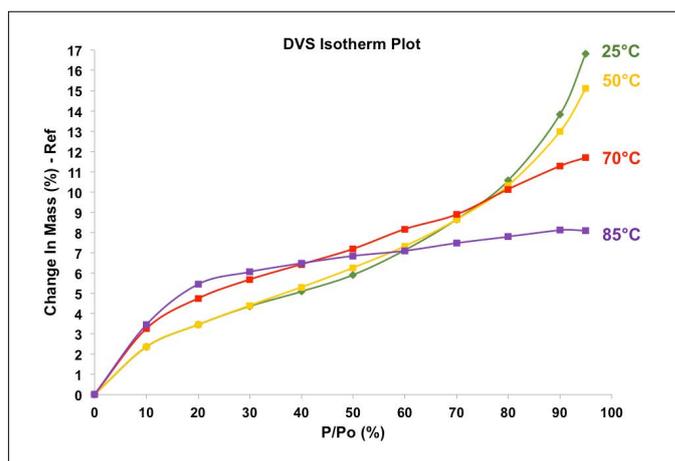


Figure 7. Water sorption isotherm of cellulose membrane at different temperatures

- 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:

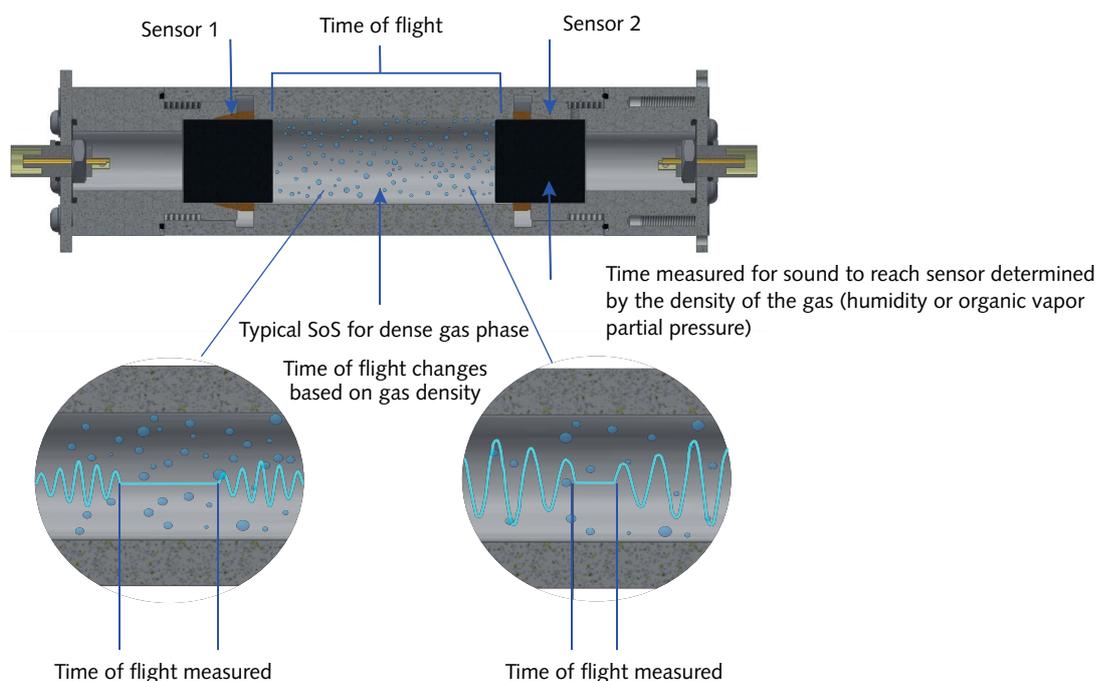
(1) Humidity with controlled generation using a humidity sensor (2) Organic vapors with controlled generation using a proprietary **Speed of Sound** sensor.

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 simultaneous 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 $\pm 0.1\%$ P/P_0 of target P/P_0

*patent pending

Partial Pressure Control

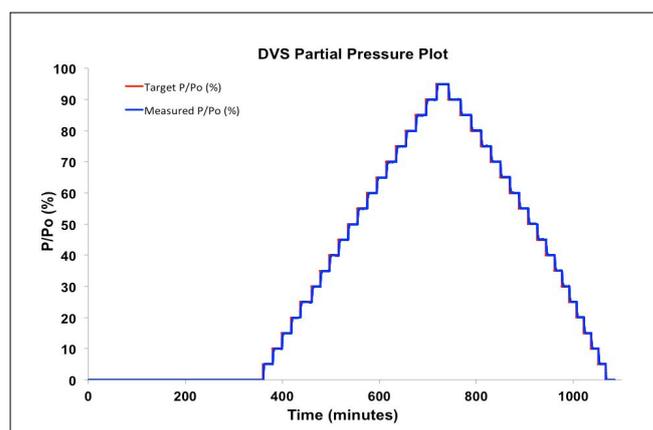


Figure 8. Kinetic of the partial pressure of acetone measured using the SoS Sensor during a DVS experiment

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

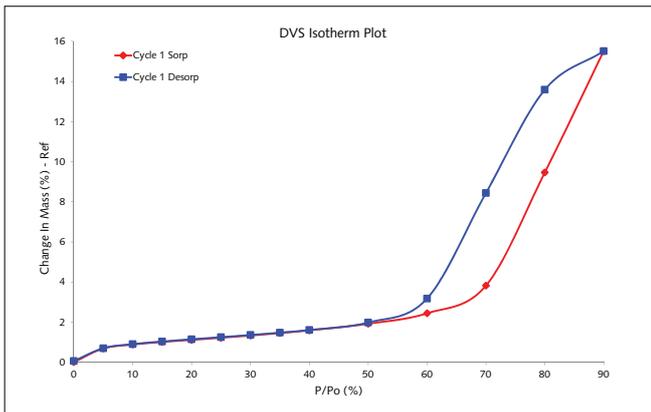


Figure 9. DVS Isotherm Plot

- Octane isotherm plot for ceramic powder at 25 °C

Surface Area Calculations

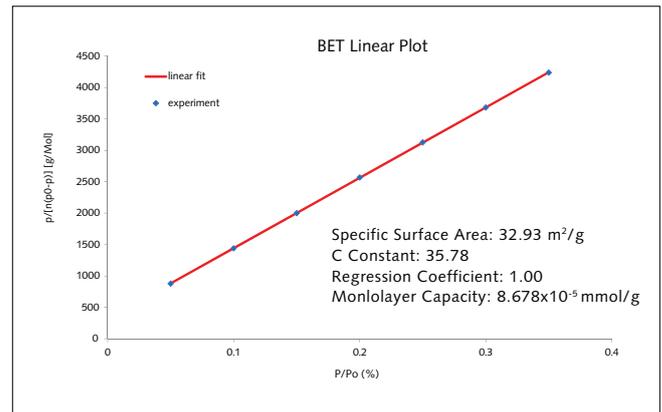


Figure 10. BET Linear Plot

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

Dual Solvent Experiments

Spray Dried Polymer Co-Adsorptions Isotherms

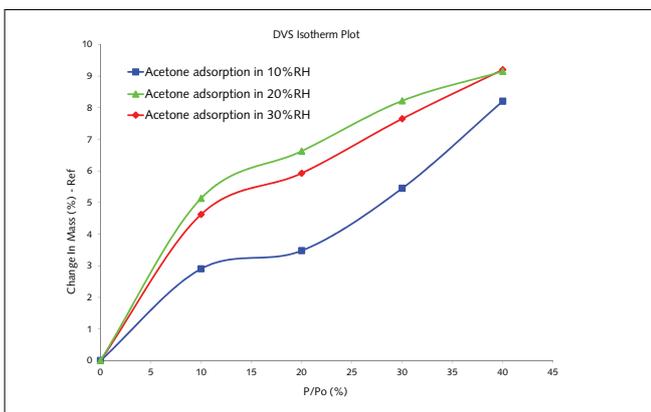


Figure 11. Acetone sorption in background humidity

- 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

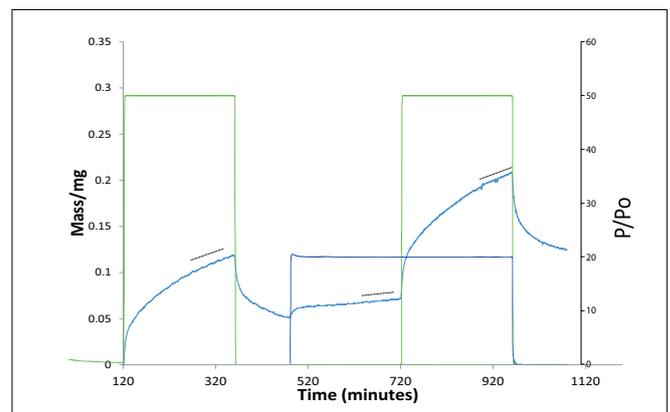


Figure 12. Kinetic plot of multi-component diffusion experiment performed on Kapton tape

- 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

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

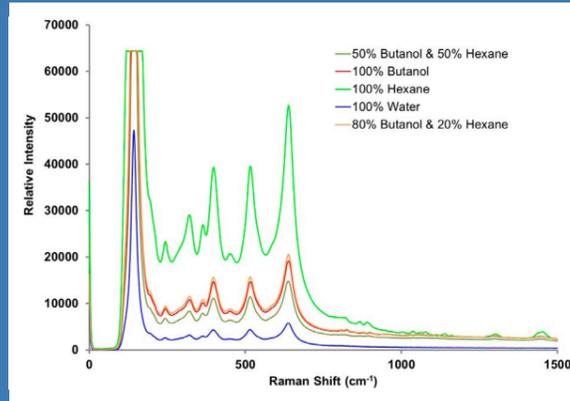


Figure 13. Raman spectra of single and dual-solvents sorption of Butanol and hexane for a titanium oxide sample at 25 °C

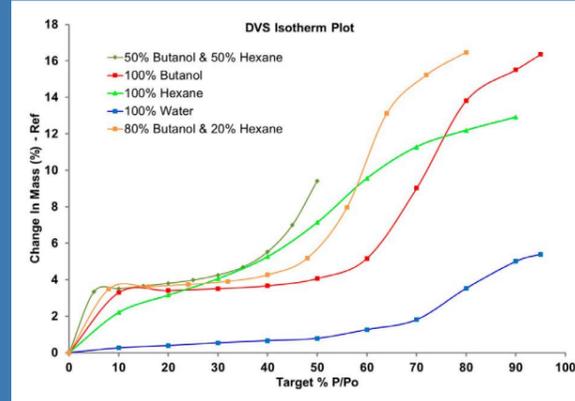


Figure 14. Single and dual-solvents isotherm of Butanol and hexane for a titanium oxide sample at 25 °C

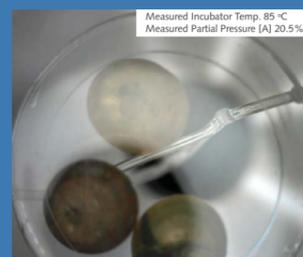


Microscopy and Video

- 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)

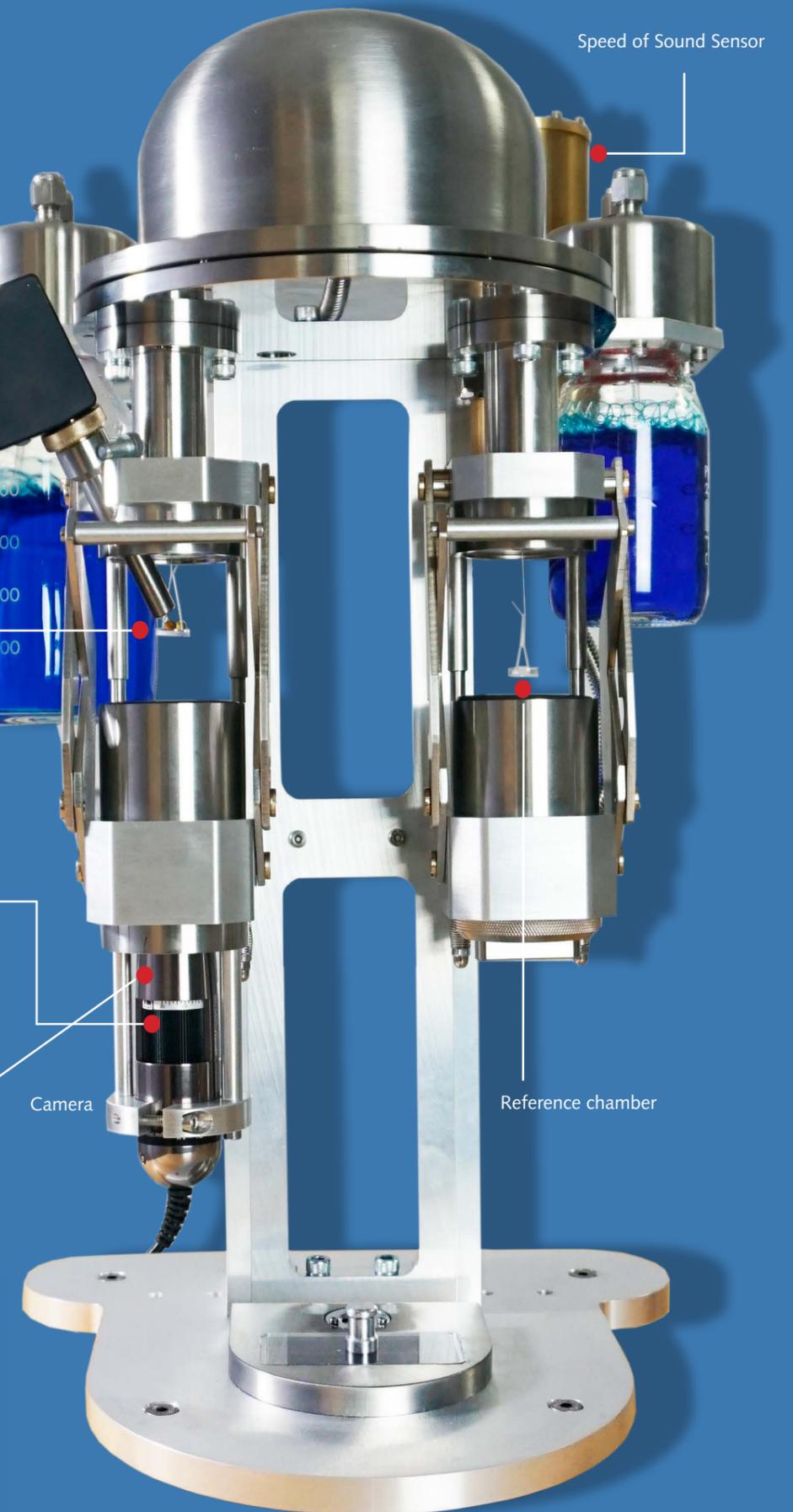
- *In-situ* degassing/activation of samples up to 200 °C
- The temperature is measured by a Pt-100 directly below the sample pan
- User programmable and controlled temperature ramps or steps

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



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/P₀ ⁵
P/P₀ resolution ± 0.1 %
P/P₀ accuracy from 5 - 85 °C ± 1 % P/P₀ ^{3,6}

Mass Measurement

Ultrabalance Low Mass

Maximum load: 1000 mg
Mass change: ± 150 mg
Resolution: 0.01 μ g
Balance noise: ≤ 0.3 μ g ⁷

Ultrabalance High Mass

Maximum load: 5000 mg
Mass change: ± 1000 mg
Resolution: 0.1 μ g
Balance noise: ≤ 3 μ g ⁷

Hardware Configuration

Standard: 1 humidity sensor plus 1 SoS sensor

Advanced: 1 humidity sensor plus 2 SoS sensors

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
- 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
- Dual vapor co-adsorption
- 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/P₀ calibration performance based on SMS

factory certified methods (Salt Calibration)

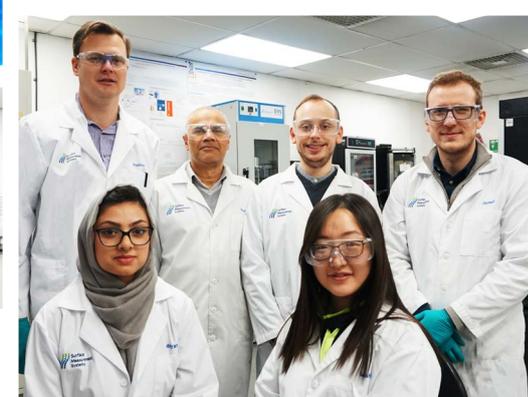
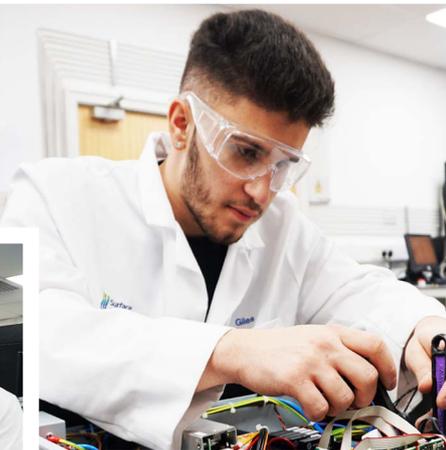
⁴ For an update of the list of solvent offered, please contact us directly

⁵ Depending on the solvent selected and experimental temperature

⁶ Depending on the solvent selected

⁷ Root mean square (averaged over 24 hour)

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



UK (European Office)

Unit 5 Wharfside, Rosemont Road
Alperton, London, HA0 4PE, UK
Phone: +44 (0) 208 795 9400

USA (North American Office)

2125 28th Street SW, Suite 1
Allentown, PA, 18103
Phone: +1 610 798 8299

SMS Instruments Private Ltd. (India Office)

1611-16/L/40, Teegal Guda Saleem Nagar,
Malakpet, Hyderabad, Telangana, India, 500036
Phone: +91 742 004 8972

version 1.0
15042020

