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The Total Sorption Solution

DVS
VACUUM

The Vacuum Sorption Solution

www.TheSorptionSolution.com

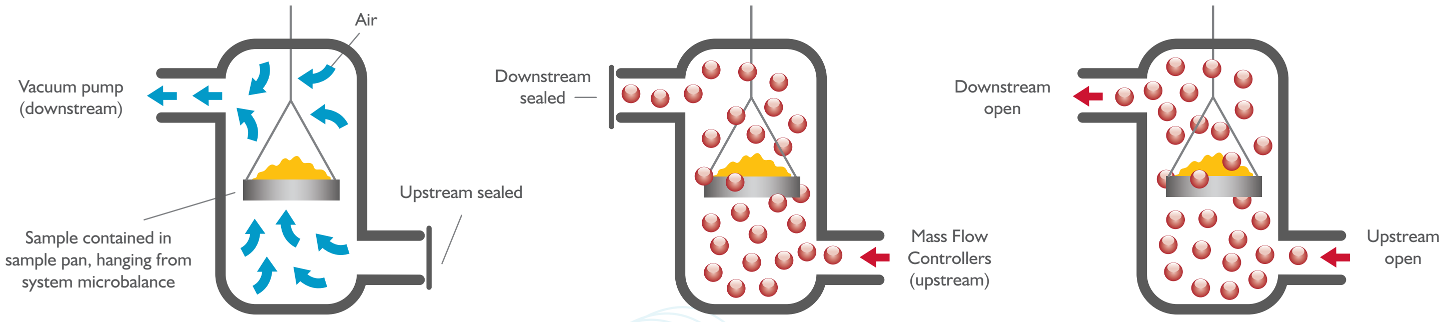
About the Technique

DVS Vacuum - the only gravimetric system that supports static *and* dynamic experiments

The DVS Vacuum offers dual sorption solutions, performing *static* experiments for higher sorbate pressures and *dynamic* experiments for lower sorbate pressures including the Henry region.

The principles of dynamic vacuum

Controlling the sorbate's entry *and* exit flows in tandem achieves dynamic vacuum conditions:



Sample chamber evacuation (1) **Filling with sorbate** (2) **Steady-state** (3)

1. Evacuation of the sample chamber using the downstream vacuum pump.
2. Introduction of sorbate molecules to the chamber at a user-controlled flow rate - the sample chamber fills until reaching the desired sorbate concentration.
3. Downstream butterfly valve opens to create equal exit and entry sorbate flow rates. The system runs at steady-state, with constant sorbate concentration. Different sorbate concentrations are attained by adjustment of the upstream and downstream flows before returning to steady-state.

Advantages of using the Vacuum Sorption Solution

For experiments in the Henry region, which use low sorbate pressures, operating in dynamic mode delivers two crucial advantages:

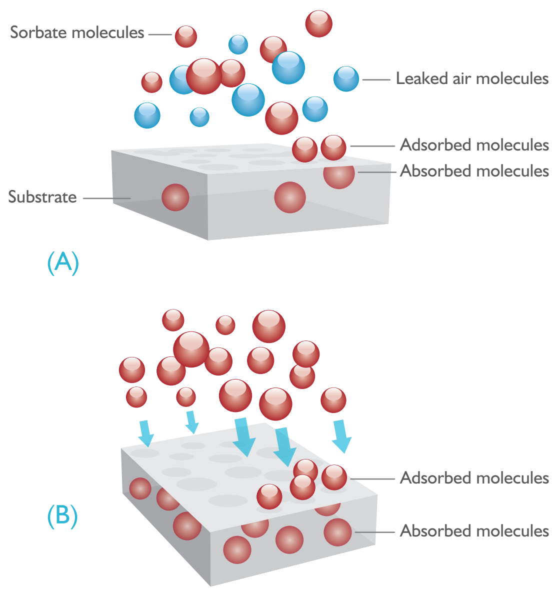
Sorbate compositional certainty

Static systems using low sorbate pressures can suffer small air leaks into the sample chamber which, over time, dilute the sorbate molecules. The total pressure in the chamber becomes the sum of contributions from the solvent and leaked air. Consequently, the actual sorbate concentration may be much lower than indicated by the system sensors, leading to experimental inaccuracies.

Using the DVS Vacuum in dynamic mode ensures the continuous removal of leaked air and its replacement with sorbate. Thus sorbate molecules constitute the total measured pressure in the sample chamber, providing certainty of the experimental concentration in the Henry region.

Residence time control

The DVS Vacuum reaches steady-state over a wide range of flow rates. This provides control over the sorbate residence time with the sample, delivering sufficient sorbate/substrate contact to accurately measure such data as pore size distributions and sorption kinetics.



Sorbate molecules and substrate under static conditions (A) and dynamic conditions (B). The measured sorbate pressure would be the same in each case.

Key Features & Selected Applications Data

Key Features

Multi-component / competitive sorption experiments

Perform multi-component experiments using two gasses, two vapors, or one gas and one vapor.

High-temperature sample pre-heater (Optional)

Pre-heat samples up to 400°C under high vacuum.

High vacuum

Experimental background pressures as low as 7×10^{-6}mbar.

Henry region (low sorbate concentration) experiments

Deliverable sorbate pressures as low as 0.1mbar (lower pressures available by consultation with SMS).

Using gasses and vapors

Typical gasses and vapors that can be used include:

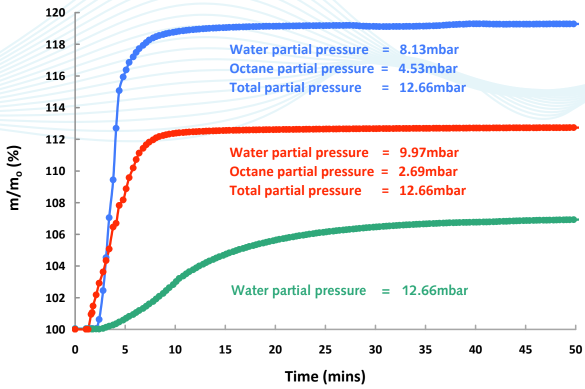
- CO_2
- SO_2
- H_2
- Ar
- Toluene
- Nitrogen
- Methane
- Water
- Cyclohexane
- Octane
- Ethanol

Selected Applications Data

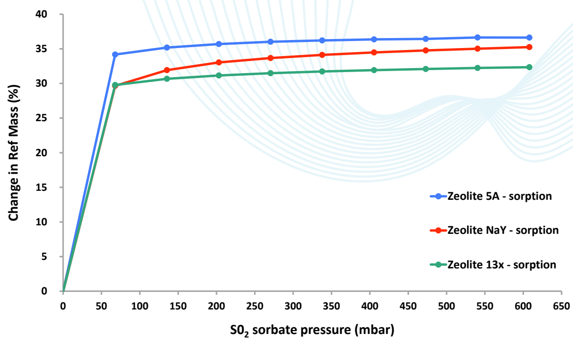
Competitive sorption and Zeolite studies: Compound separation for industrial and environmental applications

DVS Vacuum multi-component experiments simulate the industrial separation of compounds using Zeolite substrates. The Zeolite is activated using the DVS Vacuum's optional sample pre-heater.

Examples include the purification of organic solvents by their vapor-phase separation from water, and the removal of SO_2 gas – the major component of acid rain - from water vapor.



Octane/water competitive sorption on activated Carbon coated with Chitosan: The substrate shows a strong preference for sorption with Octane, making it a potentially useful agent for the separation of organics from water.

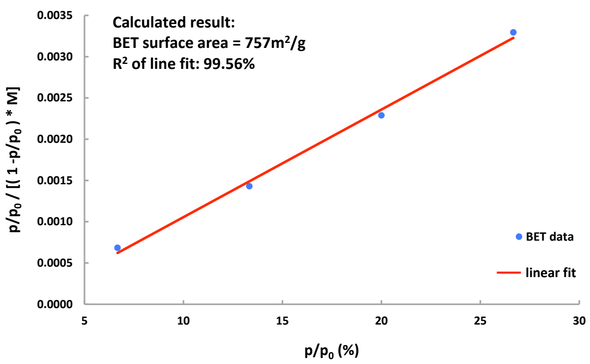


SO_2 gas sorption on three different Zeolites shows typical Type I isotherms. Sorption was found to be irreversible at 25°C - desorption could not be achieved by vacuum alone.

BET measurements at ambient temperatures

DVS Vacuum offers a number of benefits over traditional volumetric BET analyses, including the ability to use adsorbates with a range of molecular cross sections, sizes and shapes, and the ability to perform measurements at ambient temperatures rather than 77K (–196°C). This allows researchers to explore the more subtle physicochemical properties of complex microporous materials.

BET surface area of a 13x Zeolite measured by DVS Vacuum using SO_2 gas. The result correlates well with the established value of 750m²/g, measured using Nitrogen at –196°C.



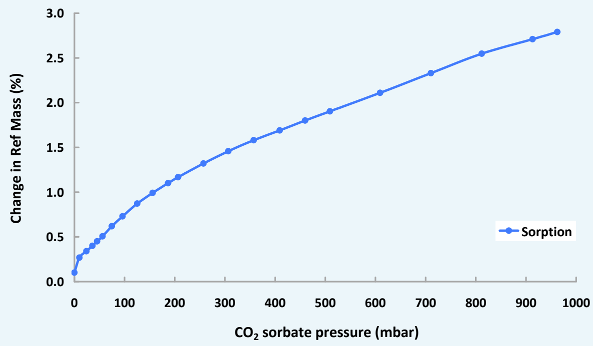
Selected Applications Data

MOF: Metal Organic Framework studies

MOFs are highly crystalline clusters of metal ions connected by organic linkers. Their nanometer-sized pores and large specific surface areas (up to 5,000m²/g) facilitate high-capacity storage of natural gasses and other compounds.

Consequently, MOFs are at the forefront of crucial environmental applications such as fuel storage for natural-gas powered vehicles and the capture of CO_2 emissions. Other applications include catalysis, drug delivery and nano-reactors.

The DVS Vacuum's low experimental pressures facilitate the activation of these highly nanoporous materials; in addition, the system can simulate 'real-life' MOF applications by using sorbates such as CH_4, H_2 and CO_2.



CO_2 sorption on MOF substrate: Isotherm measured at 25°C.

Measurement of vapor pressure and heat of sublimation of solids using a Knudsen Cell

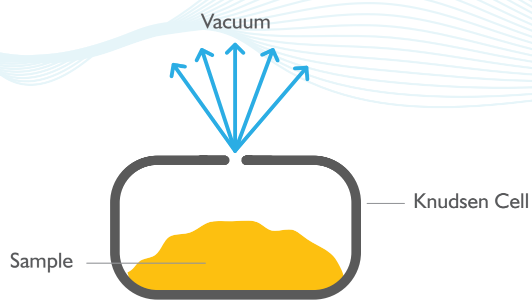
This robust method is included in the Organization for Economic Cooperation and Development (OECD) Guidelines for the determination of vapor pressure (OECD Guideline 104).

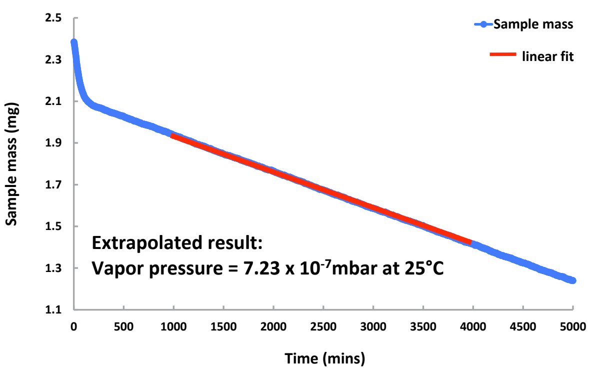
The sample is held in the Knudsen Cell - a crucible which has an orifice of known dimensions. After exposure to high vacuum, the material effuses through the hole via sublimation, resulting in mass loss - the vacuum prevents 'back scattering' by air molecules.

Through the measurement of the rate of mass loss and other experimental parameters, the Knudsen equation can be applied to calculate the sample's vapor pressure.

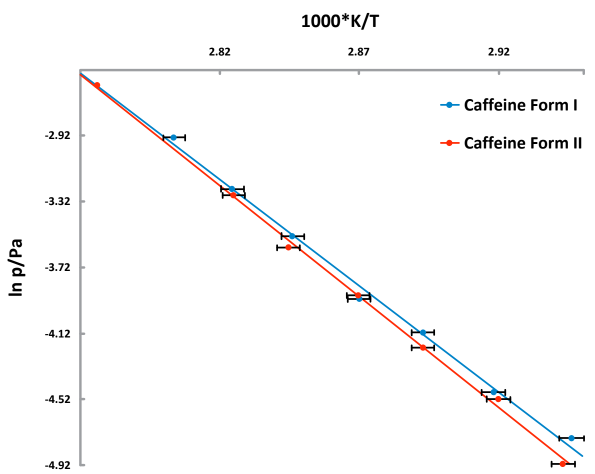
The determination of the vapor pressures of solids is relevant to several crucial applications. Examples include the storage of toxic chemicals, fragrance research into air fresheners and pharmaceutical studies on drug stability.

The Knudsen Cell



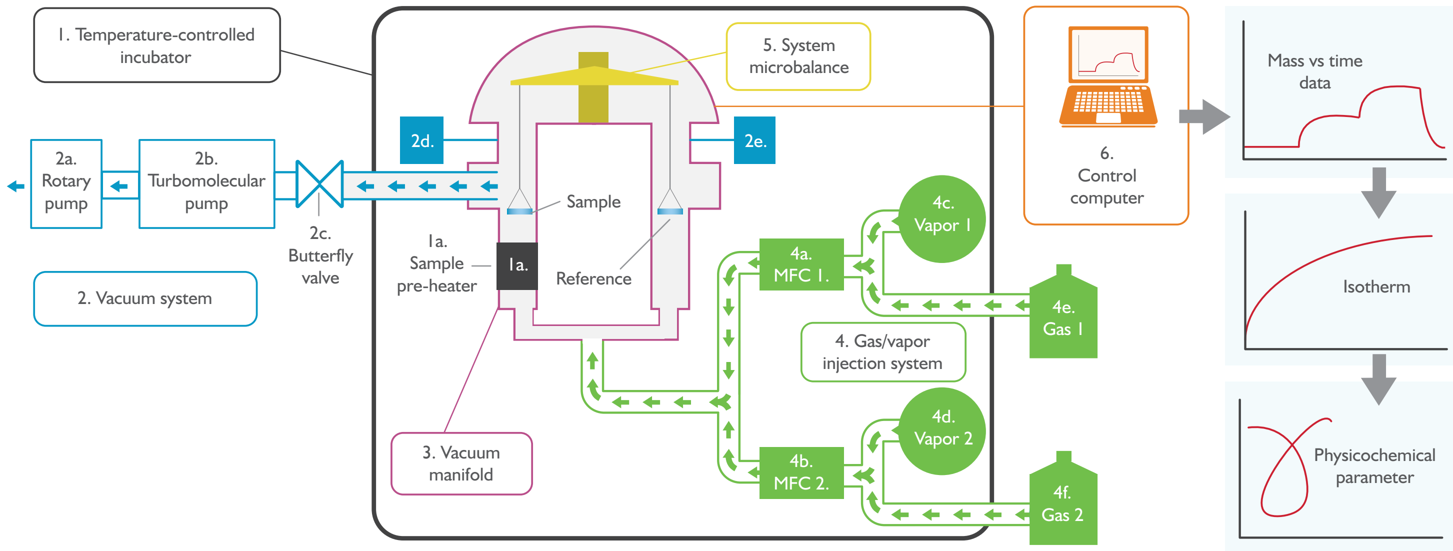


Vapor pressure of Bifenthrin is too low to be measured at 25°C, but can be extrapolated from the DVS Vacuum data recorded at 65°C.



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

Specifications and Schematic



1. Temperature-controlled incubator

Sets the experimental temperature within a range of 20°C to 85°C.

1a. High-temperature sample pre-heater (Optional)

Pre-heats the sample up to 400°C under high vacuum.

2. Vacuum system

2a. Rotary pump

Produces vacuum down to 1.3×10^{-3}mbar.

2b. Turbomolecular pump (Optional)

Provides further vacuum as low as 7×10^{-6}mbar (in combination with Rotary pump).

2c. Butterfly valve

Regulates the gas/vapor sorbate exit rate from the system, providing a point of pressure control downstream from the sample chamber.

2d & 2e. Gas/vapor pressure transducers

Measure and feed back gas/vapor sorbate pressure, ranging from 1013mbar to 0.1mbar.

3. Vacuum manifold

Constructed primarily of 316 stainless steel for chemical inertness.

4. Gas/vapor injection system

The DVS Vacuum can use one gas or vapor, two gasses or two vapors, or one gas and one vapor in combination, delivering a wide range of concentrations for water, organic vapors and gasses.

4a - 4f. Upstream sorbate pressure control

The sorbate entry is regulated by the thermal conductivity Mass Flow Controllers (MFCs - 4a and 4b).

Downstream sorbate pressure control

The sorbate exit rate is regulated by the downstream butterfly valve (2c).

5. System microbalance

Records the sample mass in real time as sorption and desorption of the sorbate occurs.

- Sample mass: up to 1.0g
- Resolution: 0.1 μg
- Mass change: up to ±150mg

6. Control PC and software

Controls experimental parameters and continuously records and saves data for subsequent analysis.

Software

SMS continues to be the world leader in stable, flexible and intuitive system software. Designed entirely in-house with extensive customer participation and feedback, SMS software provides easy-to-use sorption solutions.

DVS Vacuum control software:
PIRANI CONTROL

Pirani Control makes experimental sophistication easy. Its intuitive, windows-based, user-friendly interface allows you to run a sequence of different DVS Vacuum experiments all from the control computer.

Special features include:

- Real-time display of experiment progress
- Vapor 1 flow control
- Purge gas flow control or Vapor 2 flow control
- Butterfly valve control
- Turbo pump control
- Balance tare and calibration wizards
- Each method stage may be fixed in time or determined by a user-defined dm/dt

DVS Vacuum data analysis software:
PIRANI ANALYSIS

Pirani Analysis harnesses the experimental flexibility of the DVS Vacuum, delivering extensive and user-friendly data analysis alongside one-click report generation.

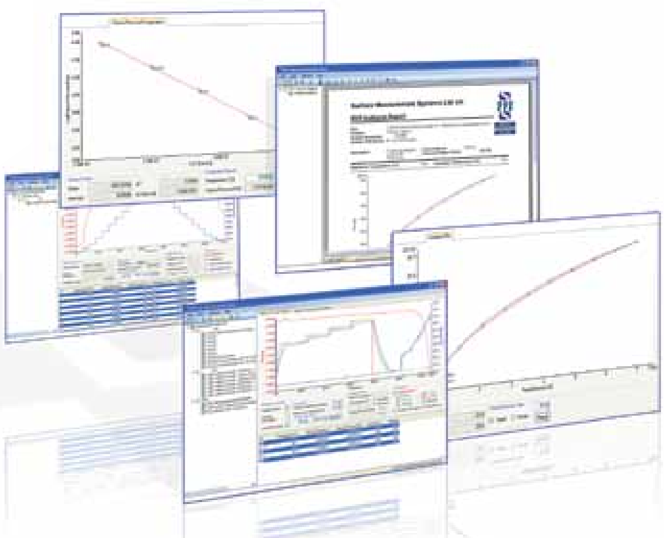
Special features include:

- Flexible data-range selection
- Easy importing of results from different methods for simultaneous analysis
- Superposition of multiple data graphs
- Isotherm generation
- dm/dt and Knudsen vapor pressure analysis

DVS analysis suites:
ADVANCED and ISOTHERM

Use the DVS *Advanced* and *Isotherm* analysis suites to elucidate key physicochemical parameters including:

- BET surface area
- Heat of sorption analysis
- Diffusion analysis
- Activation energy analysis
- Pore size distributions



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