Vapor Pressure Analyzer

Knudsen Diffusion Method

Surface Measurement Systems
World Leader in Sorption Science

www.surfacemeasurementsystems.com

Vapor Pressure Analyzer

The VPA measures the vapor pressures of solids, liquids, and oils using the Knudsen effusion method.

The vapor pressure is an important physical property that defines the amount of vapor phase material that exists in equilibrium with the original material. All materials enter the vapor phase by sublimation (solid – gas) or evaporation (liquid – gas). The vapor pressure of a material at thermodynamic equilibrium is a fundamental property of the material and is only a function of temperature.

Knowledge of vapor pressure is highly desirable for many materials including pesticides and pharmaceutical samples in order to avoid the atmospheric accumulation of toxic compounds. Indeed, the vapor pressure is directly related to the Gibbs free energy of the original solid/liquid material.

The vapor pressure of material is required to be registered with the EPA (US Environmental Protection Agency) or the EC (European Community). The Knudsen effusion method used here is approved by the Organization for Economic Cooperation and Development and it is outlined in its Vapor Pressure OECD/OCDE Guideline 104.

Knudsen effusion method

The Knudsen effusion method is a dynamic gravimetric technique based on the rate of escape of vapor molecules through an orifice of known dimensions in a Knudsen cell into a vacuum at a known temperature. The rate of mass loss through the orifice is measured by the Surface Measurement Systems' UltraBalance within the VPA system. Sample masses from 1 to 100mg can be studied typically in the temperature range from 10°C to 400°C.

In a typical experiment, the sample is placed in a Knudsen cell made of titanium containing an orifice of known area and heated to experimental temperatures. The rate of mass loss is related to vapor pressure of condensed phase, P, by the Knudsen equation (Equation 1), where dm/dt is the rate of mass loss inside the Knudsen cell with time, M is the sample molar mass (mol/g), R is the universal gas constant, A is the area and T is the temperature.

$$P = \frac{\left(\frac{2\pi RT}{M}\right)^{1/2}\left(\frac{dm}{dt}\right)}{A} \qquad (1)$$

Dm/dt is the slope value from a least square regression fit to the experimental mass data over a certain period of time. The dm/dt is used to calculate the vapor pressure using Equation 1. A series of measured vapor pressures at differing temperatures are used to determine the constants A and B in the **Clausius–Clapeyron** equation (Equation 2) and thus calculate enthalpy of vaporization, ΔH, or heat of sublimation.

$$\log_{10}(p/Pa) = A - \frac{B}{T/K} \qquad (2)$$

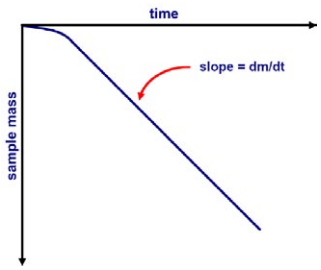
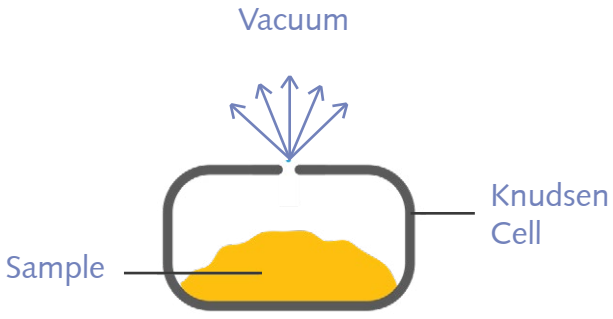


Figure 1 Mass versus time data from Knudsen cell

Knudsen cell



Experimental Data

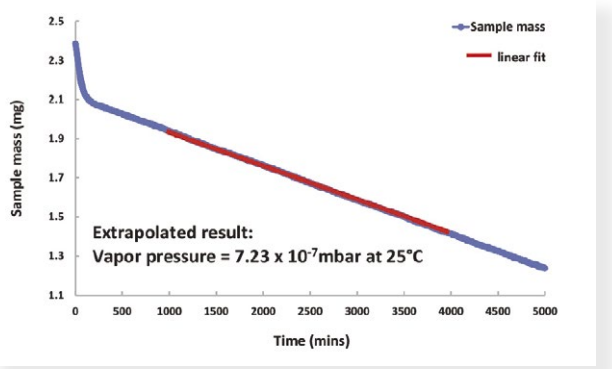


Figure 1. Vapor pressure of Bifenthrin is too low to be measured at 25°C, but can be extrapolated from the DVS Vacuum data recorded at 65°C.

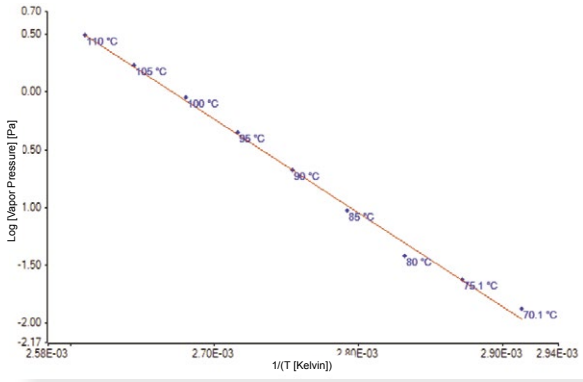


Figure 2. Plot of vapor pressure data for Bifenthrin.

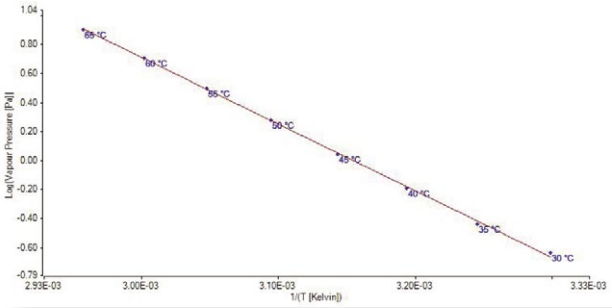


Figure 3. Plot of vapor pressure data for benzoic acid.

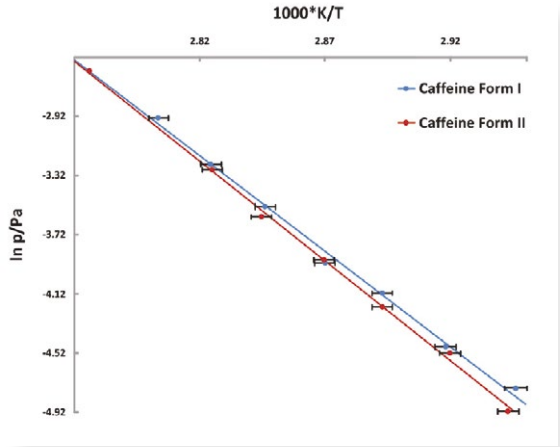


Figure 4. Vapor pressure of Caffeine vs temperature stability study. The lower vapor pressure of Form II shows it to be the more stable polymorph.

Software

VPA analysis software

includes the following features:

Windows®-based graphical interface

Knudsen vapor pressure analysis

One-click data analysis and report generation

Flexible data-range selection

Easy importing of results from different methods for simultaneous analysis

VPA control software

includes the following features:

Windows-based interface

Quick-set methods

Easy saving and restoring of methods

Real-time display of experiment progress

Data saved in a tab-separated values (TSV) file

Turbo pump control

Balance tare and calibration wizards

Incubator and pre-heater temperature control

Specifications and Schematic

Temperature

Temperature controlled enclosure
Control range: 10°C to 70°C
Temperature accuracy: ± 0.2°C

High-temperature pre-heater

400°C (maximum local temperature)
Temperature sensor: Pt-100

Vacuum generation

Roughing pump can produce minimum vacuum pressure of 1 x10^{-3}Torr.

Turbomolecular pump in combination with roughing pump provides vacuum pressure down to 1x 10^{-8}Torr.

Vacuum pressure measurement

Vacuum pressure transducer: full scale from 1x10^{-8}Torr up to atmospheric pressure.

Vacuum stand

Material: 316 stainless steel
Seals: Viton® and Kalrez®
KF flanges and VCR fittings

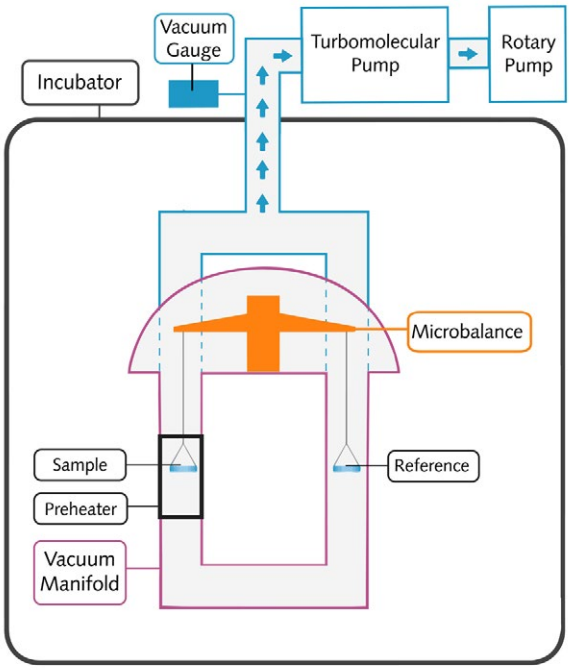
Mass Measurement

Ultrabalance Low Mass

Sample mass:	between 1 and 1000g
Mass change:	up to ±150 mg
Resolution (precision):	0.01 µg
Root mean square balance noise:	≤ 0.3 µg

Ultrabalance High Mass

Sample mass:	between 10 and 5000g
Mass change:	up to ±1000 mg
Resolution (precision):	0.1 µg
Root mean square balance noise:	≤ 3 µg



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