# Sorption technology for pharmaceutical applications

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Surface Measurement Systems

# Overview

- Surface Measurement Systems - Introduction
- Dynamic Vapour Sorption (DVS)
- Inverse gas chromatography (iGC)

# Surface Measurement Systems

**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, building materials and food industries, and are used by hundreds of leading laboratories and universities throughout the world.

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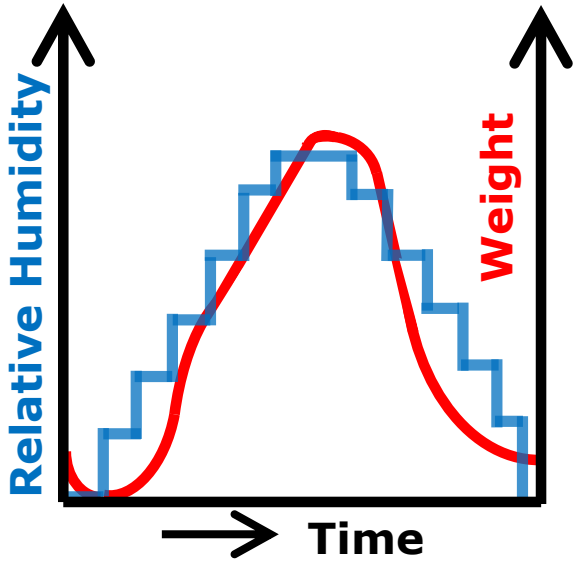
# Dynamic Vapour Sorption

## What is DVS?

DVS is a gravimetric technique that measures how quickly and how much of a solvent is absorbed by a sample.

- Sample is exposed to a series of step changes in relative humidity and the mass change is measured as a function of time.



# What can the DVS do for me?

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

# Comparison to traditional jar method

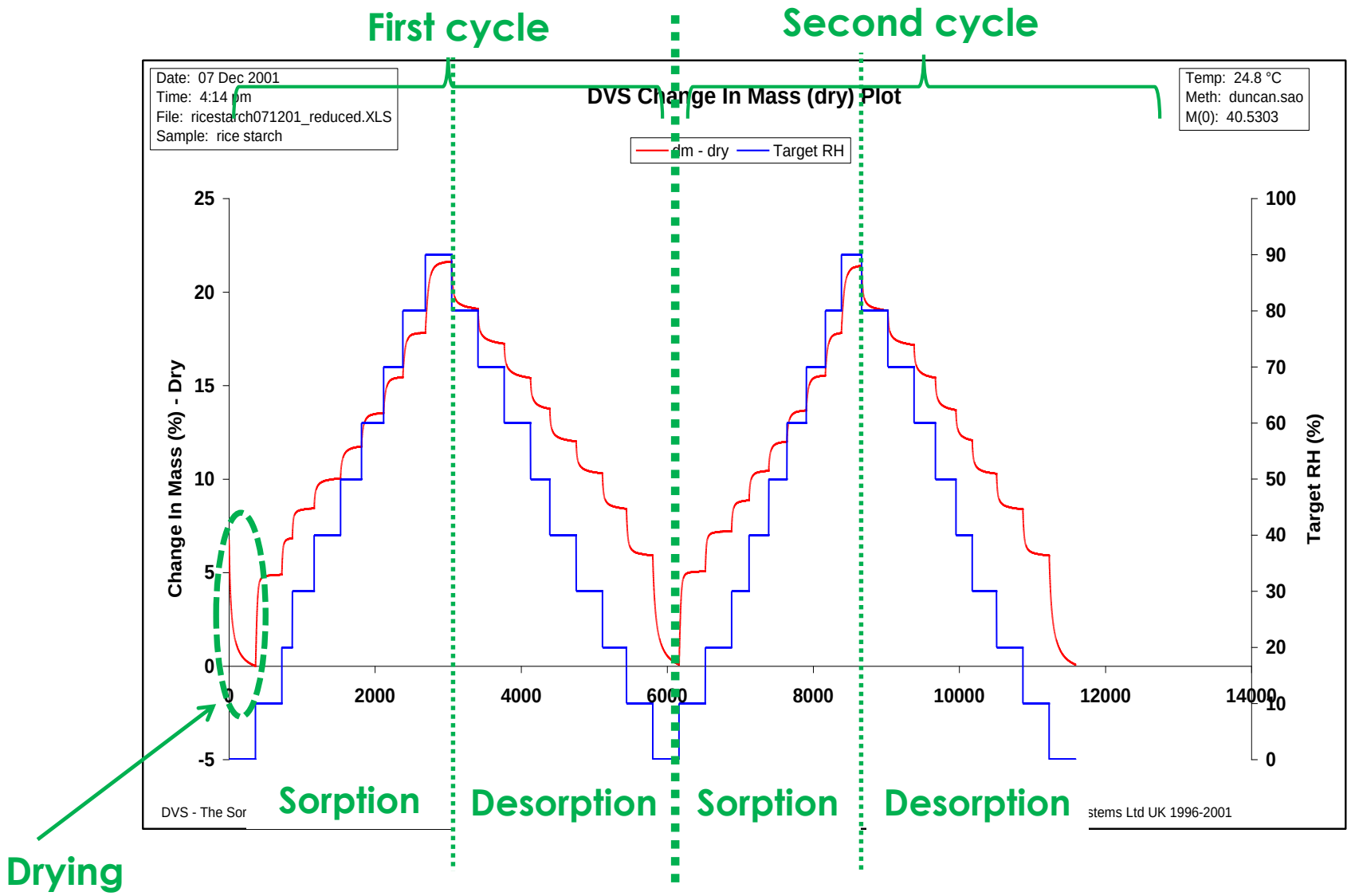
## Jar Method (static sorption)

- Static system - Very slow achievement of equilibrium
- Longer experiment time
- Large amounts of sample required (1 to 10 g)
- Risk of contamination or sample loss due to manual weighing

## DVS (dynamic sorption)

- Dynamic Flowing Gas System - Faster Equilibrium
- High sensitivity Ultra-Microbalance (0.1 µg resolution)
- Allows small sample to be used (1 to 10 mg)
- No risk of contamination or sample loss
- Both sorption and desorption measured

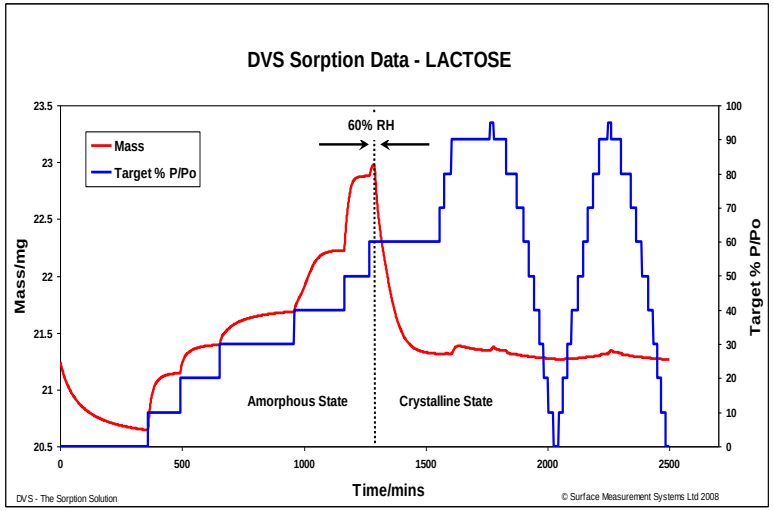
# Typical DVS data



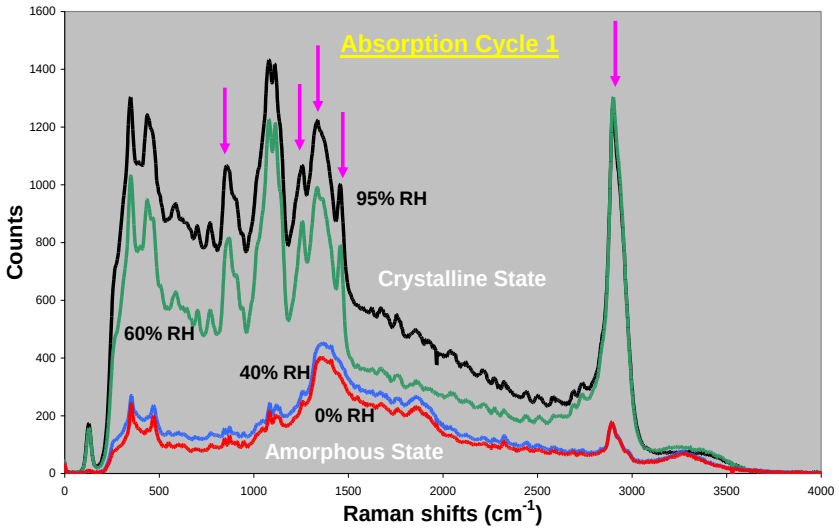
# Applications of DVS - Examples

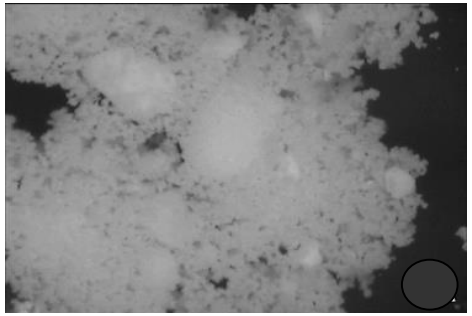
## Phase transitions - Amorphous Lactose

DVS Data



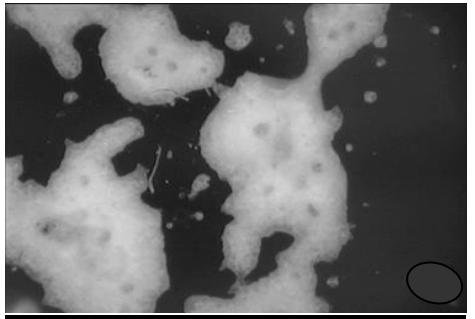

Raman Data



Amorphous State

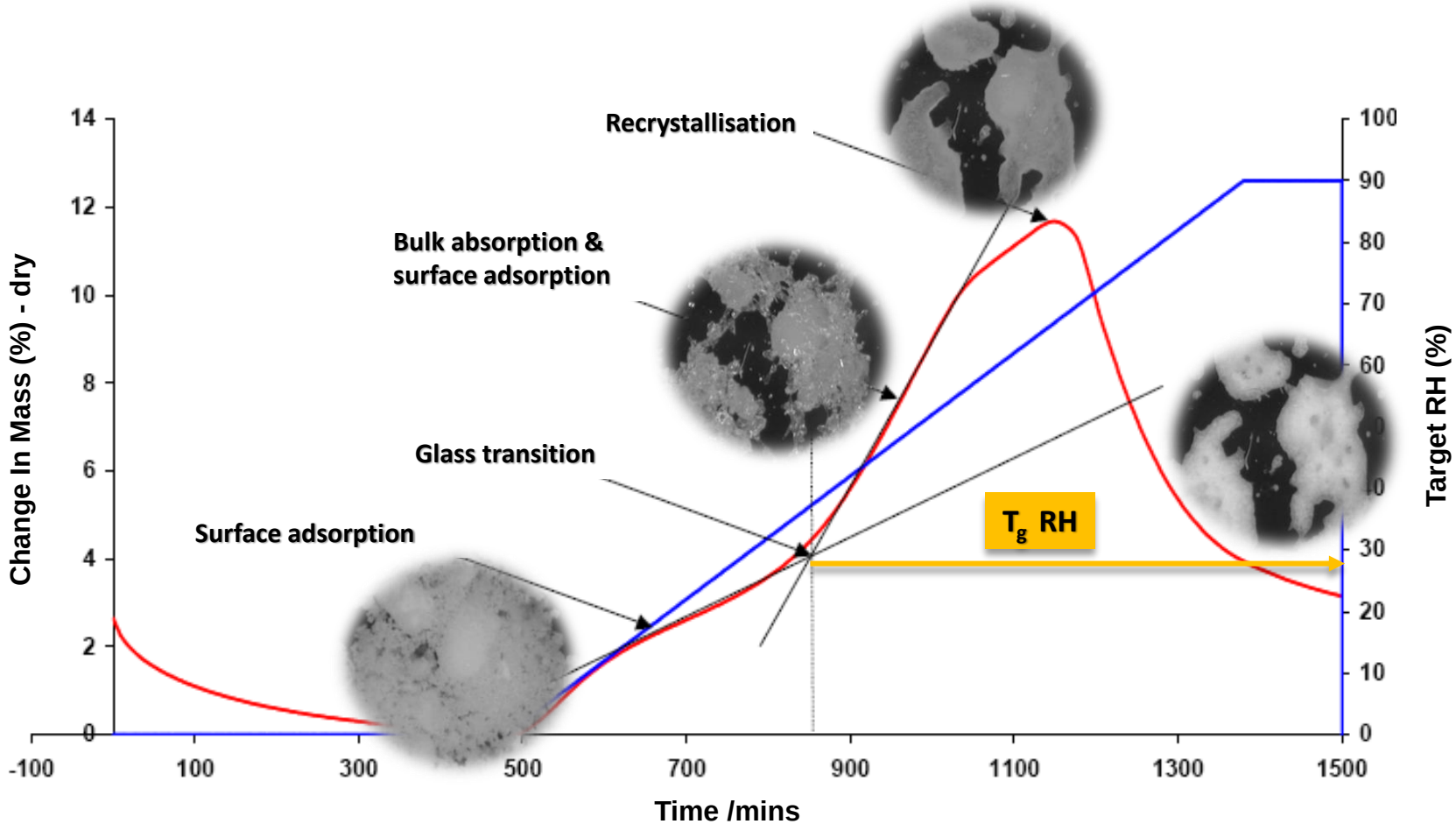
Video Images



Crystalline State

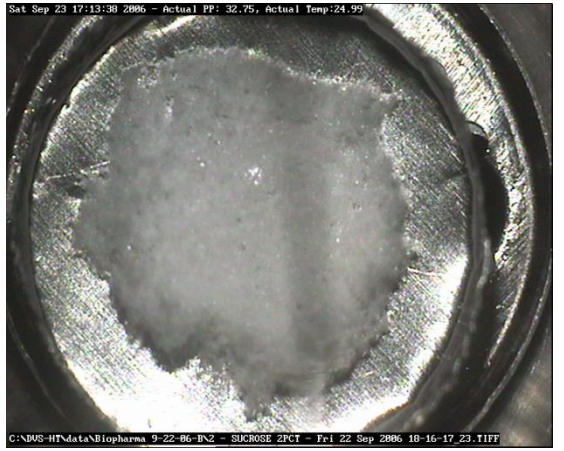
# Applications of DVS - Examples

## Glass Transition and Crystallisation



# Pure Sucrose Images


- *0% RH*




- *32.7% RH Glass Transition*


- *80.9% RH Crystallized*


- *92.6% RH Deliquesced*

# Properties measured by DVS

**DVS is a tool for thermodynamic and kinetic studies of surface and bulk properties**

- Sorption Isotherms
- BET Specific Surface Area
- Phase Transitions
- Permeability and Diffusion
- Competitive (Multicomponent) Adsorption
- Kinetics information
- Heat of Sorption
- Tg RH determination
- Amorphous content determination
- Camera and Raman capability

# DVS Family of Products

## DVS
### INTRINSIC PLUS

Water vapor only
20-40 °C
Small footprint

## DVS
### VACUUM

$10^{-6}$ Torr to Atmospheric
20-70 °C
**Water + Organic Vapors & Gases**
Competitive sorption
Sample preheat to 400 °C

## DVS
### ADVENTURE

Water only
**5-85 °C**
Temp stability +/- 0.1°C
Camera and Raman
Sample preheat up to 300 °C

## DVS
### RESOLUTION

**Water + Organic Vapors & Gases**
**5–85 °C**
**Speed of Sound Sensor**
Temp stability +/- 0.1°C
Camera and Raman
Sample preheat up to 300°C
Has 2 configurations:
<u>Standard and Advanced</u>

## DVS
### ENDEAVOUR

**5 experiments in parallel**

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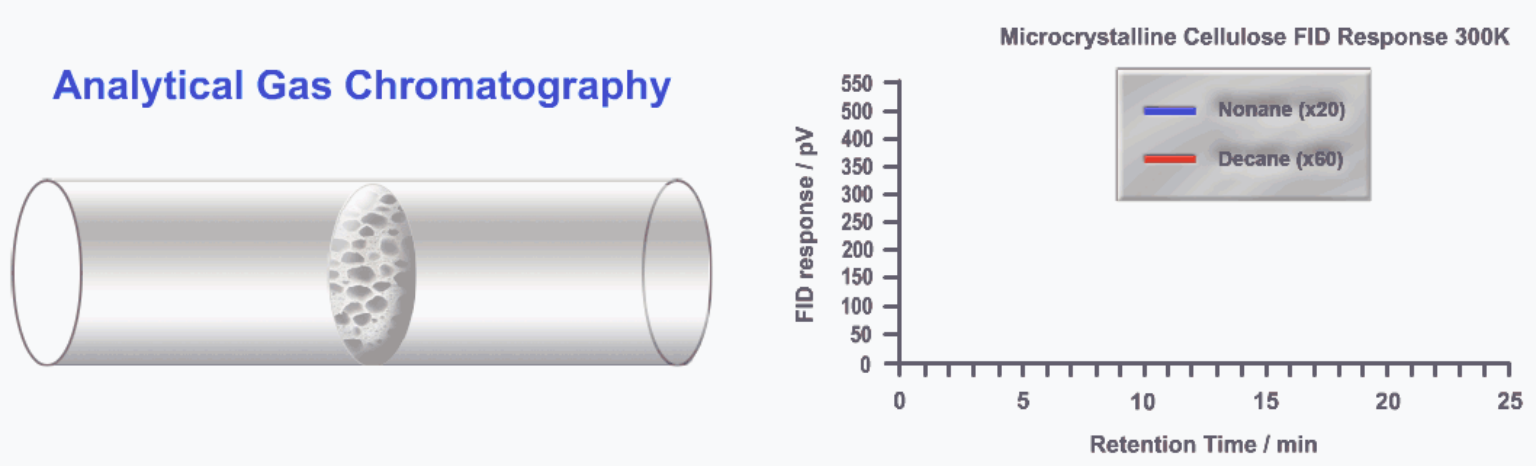
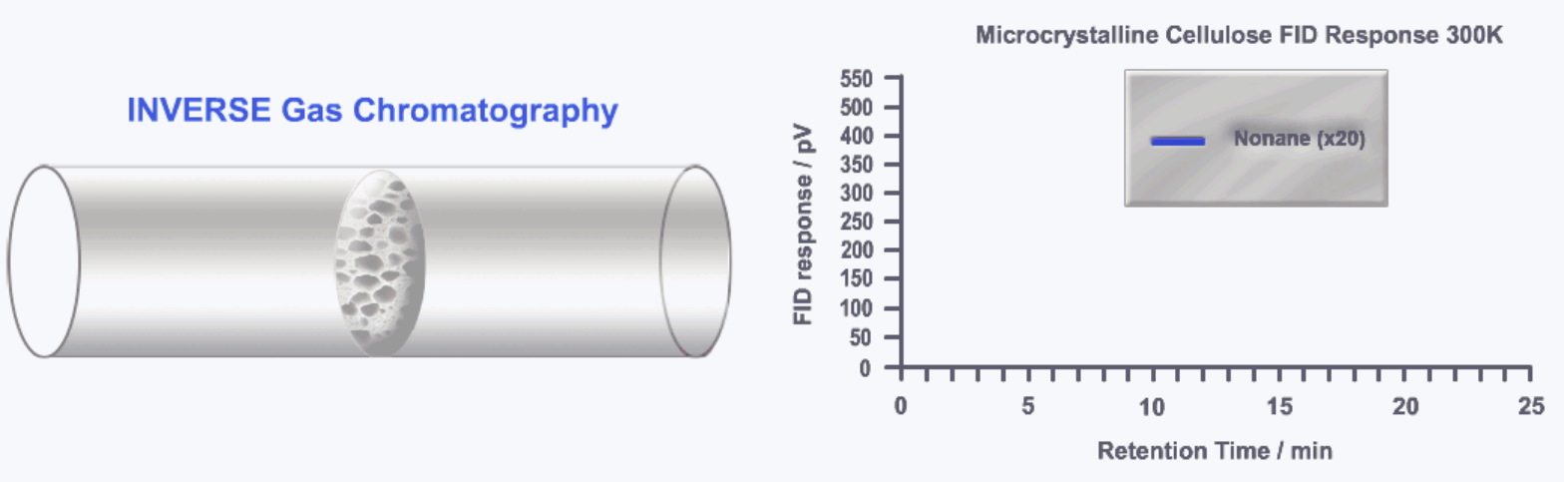
# Inverse Gas Chromatography

- Gas phase sorption technique.
- Focus on physicochemical studies - kinetic information and thermodynamic quantities from sorption equilibria.
- Earlier work on catalytic materials, e.g. activated carbon, alumina and silica.
- Powerful physico-chemical characterization tool for **powders, fibres, films, particulates, semi-solids**.
- Surface Energy is the most common measured property by iGC.

** Schultz, J., Lavielle, L., and Martin, C., The role of the interface in carbon fibre epoxy composites. J. of Adhesion, 1987. 23(1): p45-60.

# IGC Principles




Animation by L. Teng, Surface Measurement Systems

# iGC-SEA Introduction

- **Gas phase injection** (like Headspace) - 12 vapor reservoirs (50 ml)
- **Carrier gas** is helium/nitrogen
- 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**

# Properties measured by IGC-SEA

The IGC-SEA provides unique access to the following physico-chemical properties of a wide range of solid materials in a controlled humidity environment:

- Dispersive and Polar **Surface Energies**
- Heats and Entropies of Adsorption
- **Acid/Base** Interactions
- **BET** Specific Surface Area
- Phase Transitions
- Sorption **Isotherms**
- Permeability, Solubility and Diffusion
- Competitive (Multicomponent) Adsorption
- Thermodynamic Work of **Cohesion** and **Adhesion**
- Surface Energy **heterogeneity mapping**
- **Constantly extend the applications – future applications e.g. Chemisorption**

# Thank you!

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