

# Clever Characterisation a Different Approach HTE

Dr Simon R Gibbon  
AkzoNobel Research, Development & Innovation  
[simon.gibbon@akzonobel.com](mailto:simon.gibbon@akzonobel.com)

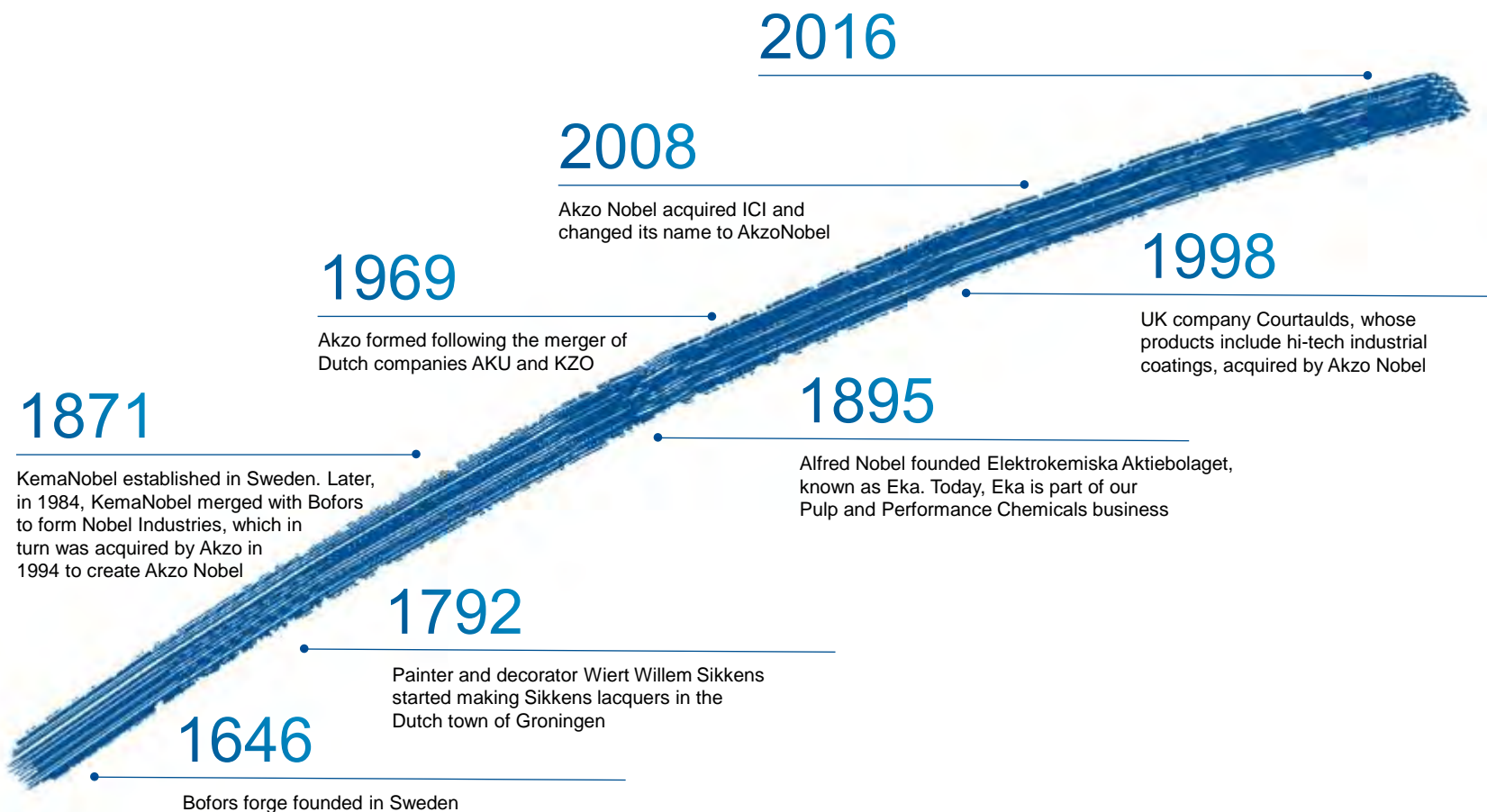


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- **Conclusions on Characterisation for HTE**

## More than 350 years of history and cutting-edge innovation



## The world of AkzoNobel\*

**€14.3**

billion in revenue

**47,200**

employees

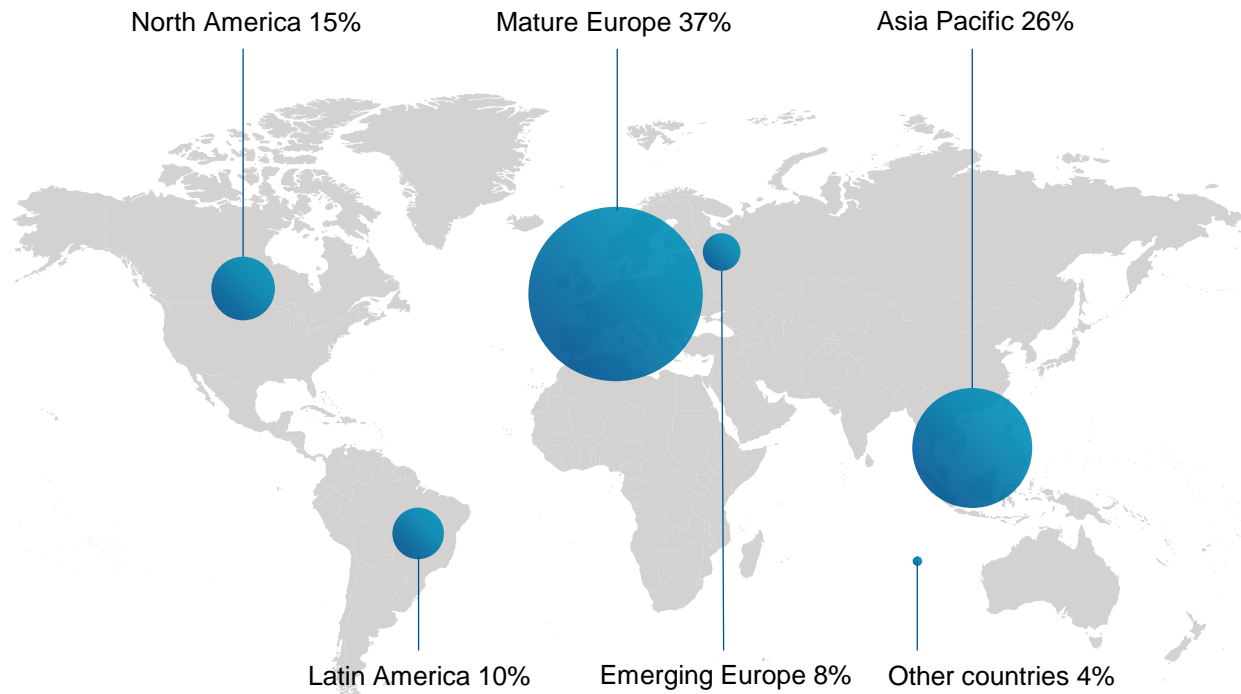
**200+**

production sites

**80+**

countries

### Key regions by revenue



### Revenue by end-user segment

Buildings and Infrastructure	42%
Transportation	16%
Consumer Goods	17%
Industrial	25%

### Revenue by Business Area

Decorative Paints	27%
Performance Coatings	39%
Specialty Chemicals	34%

\* All figures are based on year end 2014

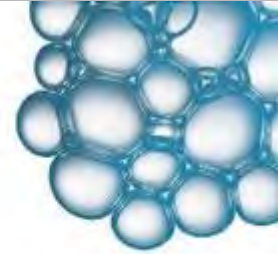
## Paints, coatings and specialty chemicals

Leading global paints and coatings company  
and a major producer of specialty chemicals

Consistently ranked as one of the leaders in  
the area of sustainability

Passionate about innovation, with 4,000  
scientists at 130 laboratories

Committed to society through our brands and  
hands-on community projects





Sustainability



Our commitment to  
doing more with less

# Human Cities

Making cities more human

AkzoNobel

Color

Heritage

Transport

Education

Sport and leisure

Sustainability

## AkzoNobel and Formulation

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### **We make ingredients**

#### **Speciality Chemicals**

- Functional Chemicals – chelates, .....
- Polymer Chemicals – anti-oxidants, crosslinking agents.....
- Surface Chemistry – surfactants, inhibitors, .....

### **We use ingredients**

#### **Performance Coatings**

- Marine Coatings – ships from oil tankers to ferrys
- Metal Coatings – buildings, cans
- Powder Coatings – furniture, cars, diggers, electrical cabinets, .....
- Protective Coatings – infrastructure, floating structures, .....
- Speciality Coatings – aerospace, yachts, mobile phones, .....

#### **Decorative Paints**

- Wall paints, wood paints, concrete paint, .....

### **Basically lots of formulation challenges**



## Reality of HTE

### Why HTE? Sample Space

**297 Identified Raw Materials**

**Blends of 2 gives 44000 combinations**

**x16 - 4 Concentrations of each component**

**x2 pHs (neutral)**

**x2 processing**

**x4 Ingredient v**

**x5 replicates**

**So, only**



**colour / flavour**

**nts to do**

**No HTE - 8 experiments a day = 20,000years**

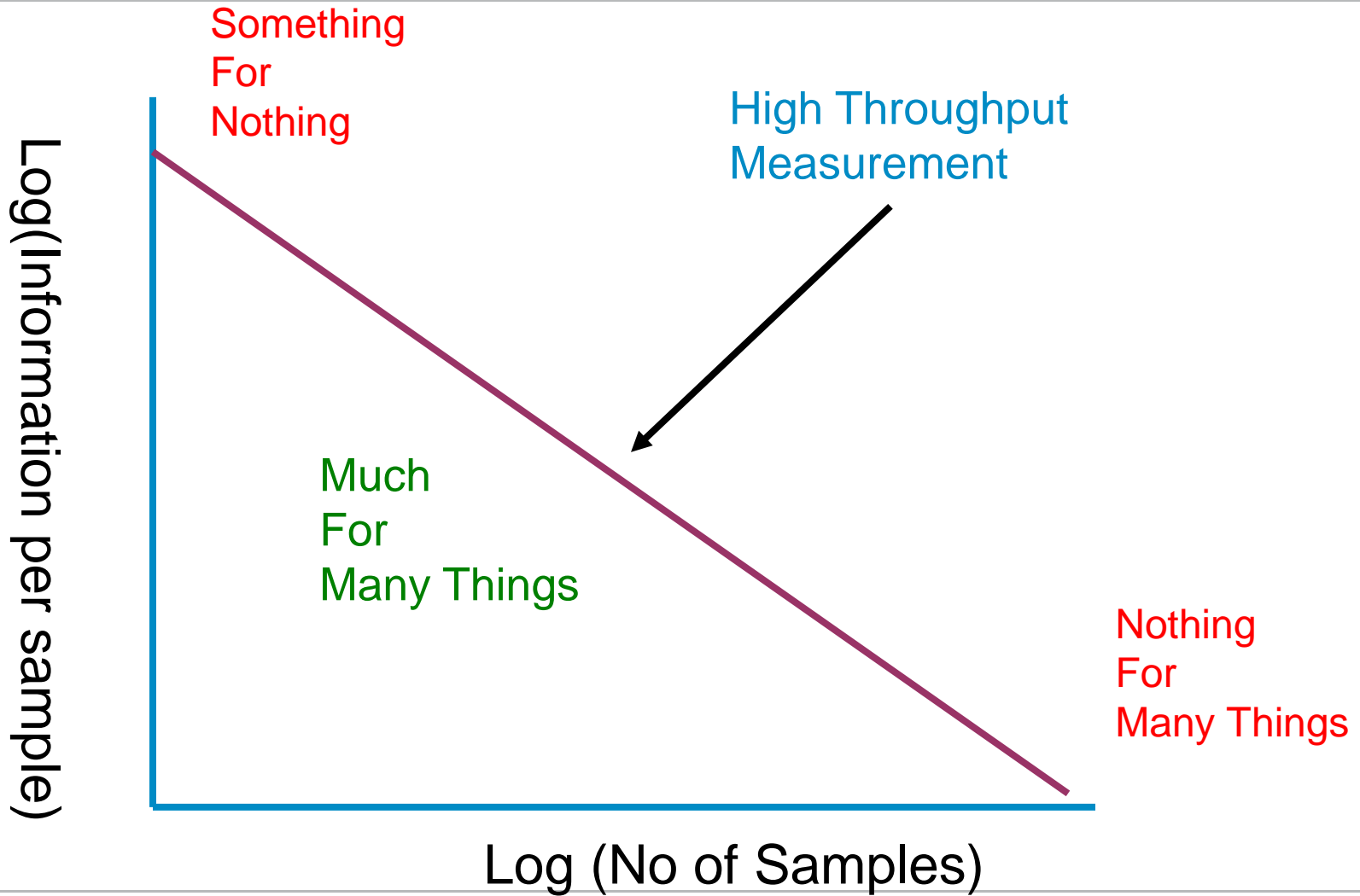
**HTE - 1 minute an experiment = 107years**

# 100 Years + 500,000 Years of Neglect – World Heritage Site



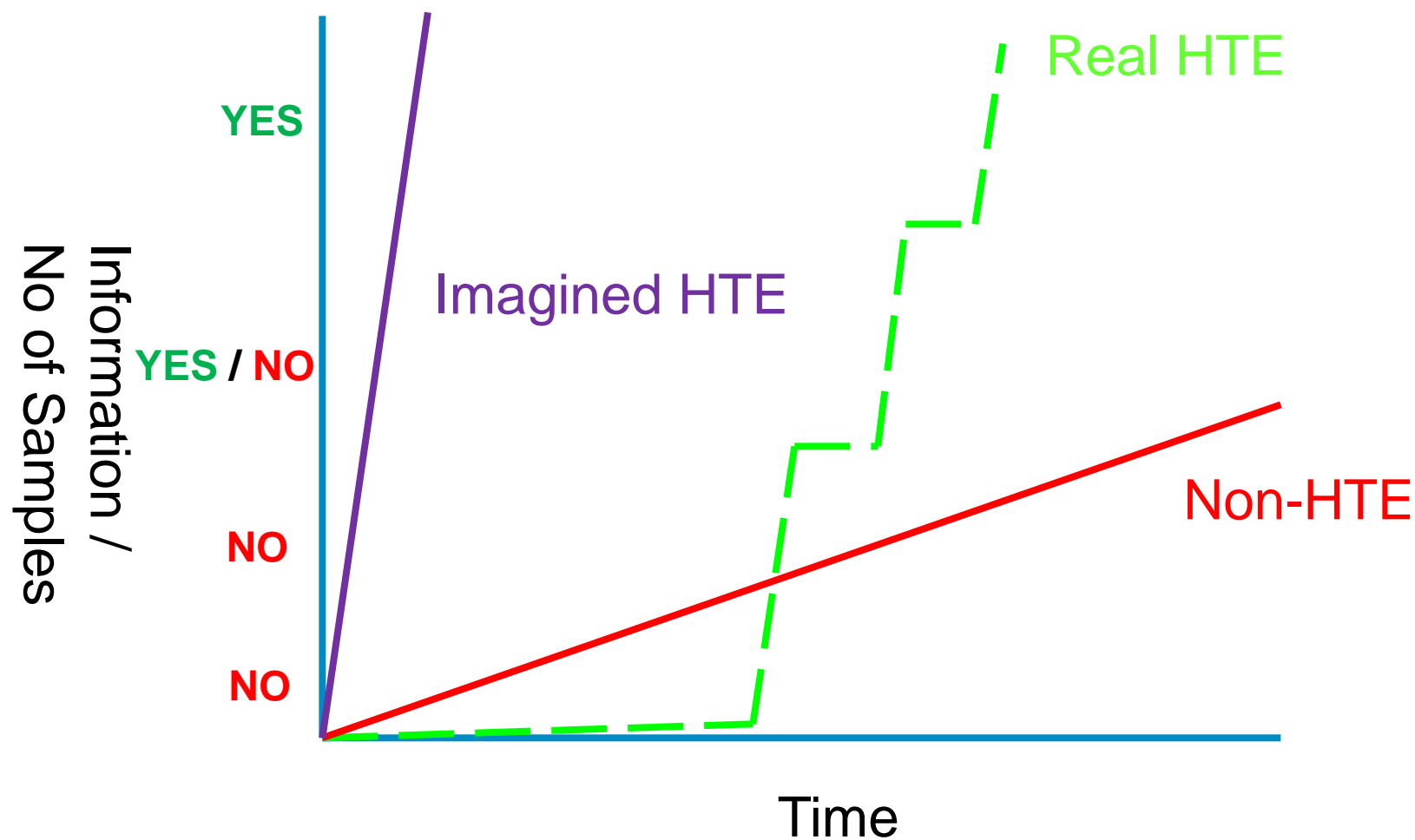
# Reality of HTE

## Choice of High Throughput Measurement



# Reality of HTE

## High Throughput Information Delivery



# Reality of HTE

## Smart HTE

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**HTE does not replace good experimental design**

**HTE needs modelling**

**HTE needs statistical design of experiments**

**HTE clever characterisation – measurement / screening**

**HTE needs validation at every step – sample preparation, screens, ....**

**HTE shouldn't just be about doing more, but doing it more repeatable**

**Select areas of composition space to work in – based on:**

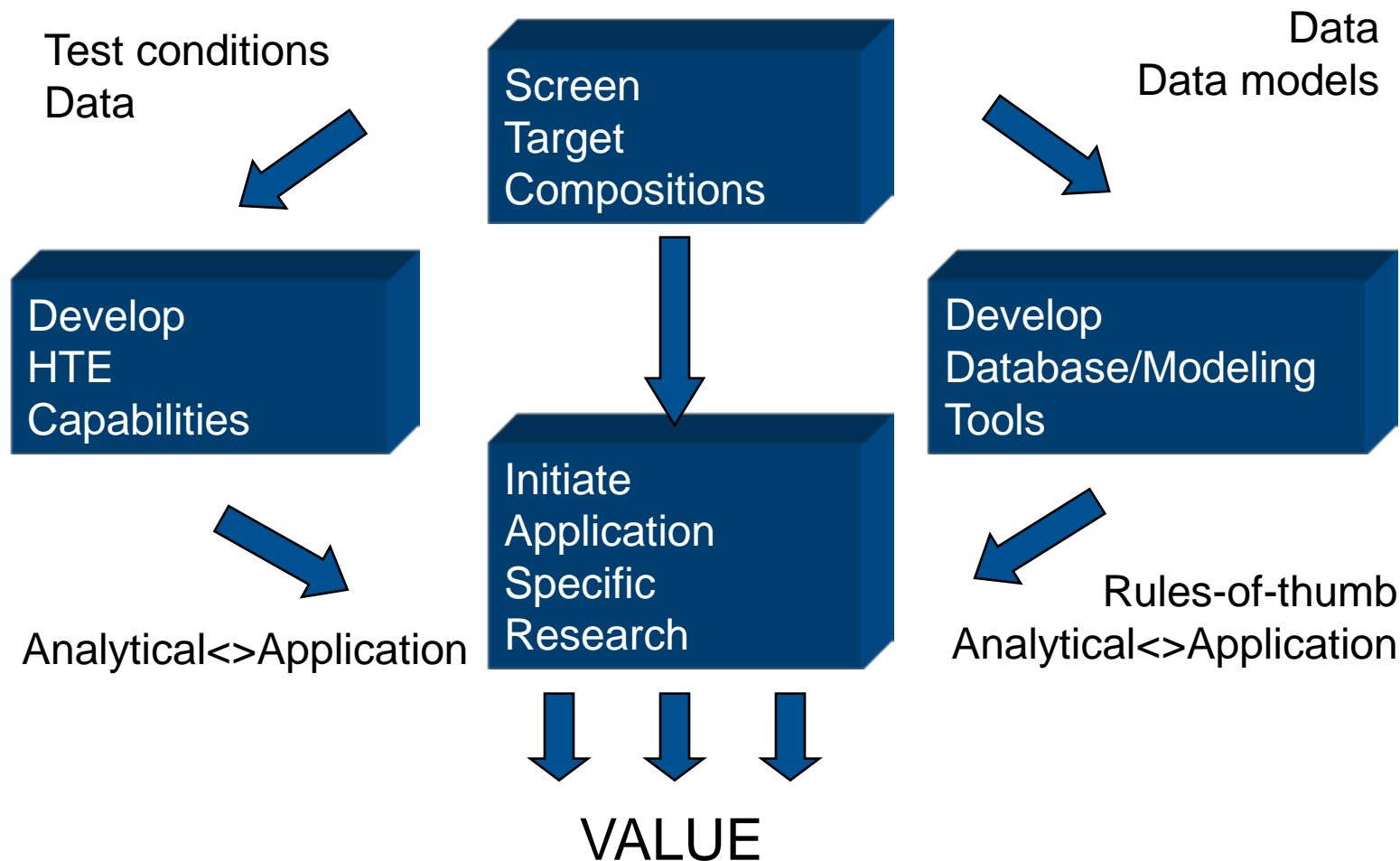
- Physical chemistry
- Understanding of raw materials
- Requirements of applications

**Plan campaigns to map these areas**

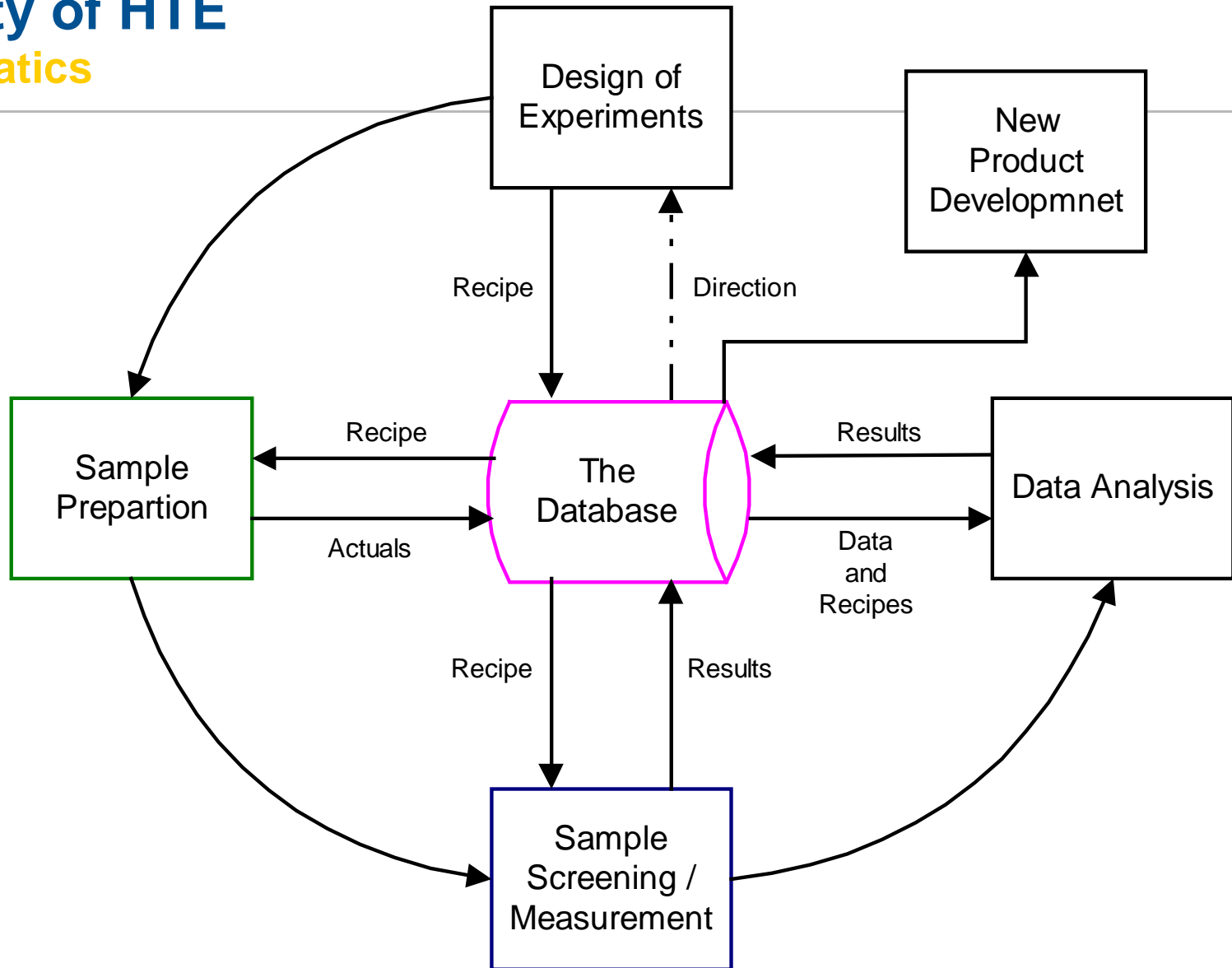


# Reality of HTE

## Overview of Development Programme



# Reality of HTE Informatics



## Reality of HTE Screening

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### 1st tier

- Rapid measurements
- Accept relative data
- Accept semi-quantitative
- Ensure no false negatives
- If necessary allow some false positives
- Pass / fail ok
- Sediment volume / rate, transmission, foam height, relative viscosity, .....

### 2nd tier

- Absolute data
- Quantitative results
- Statistically significant
- Modulus, complex viscosity, infra-red spectra, adhesion, .....

# Reality of HTE

## The Right Size For Processing and Measurement



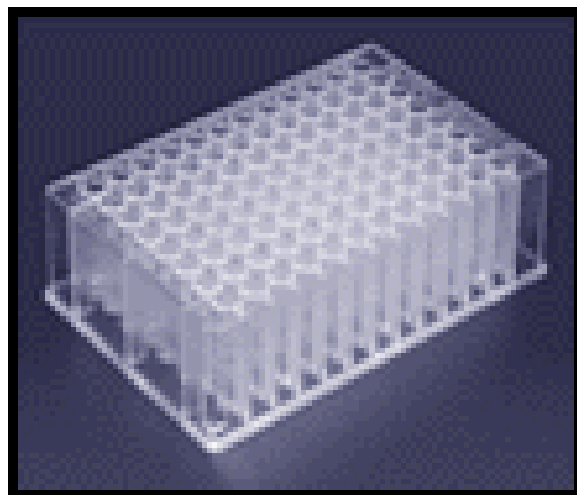
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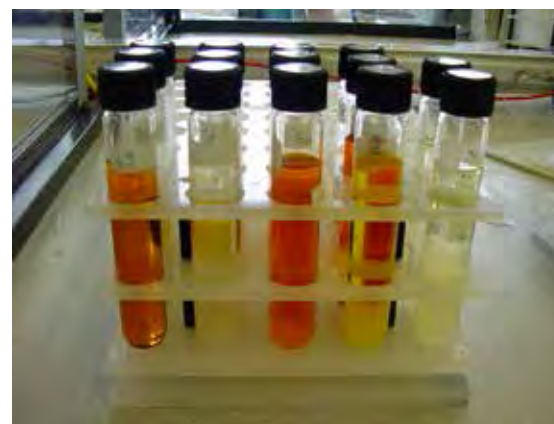
384



96



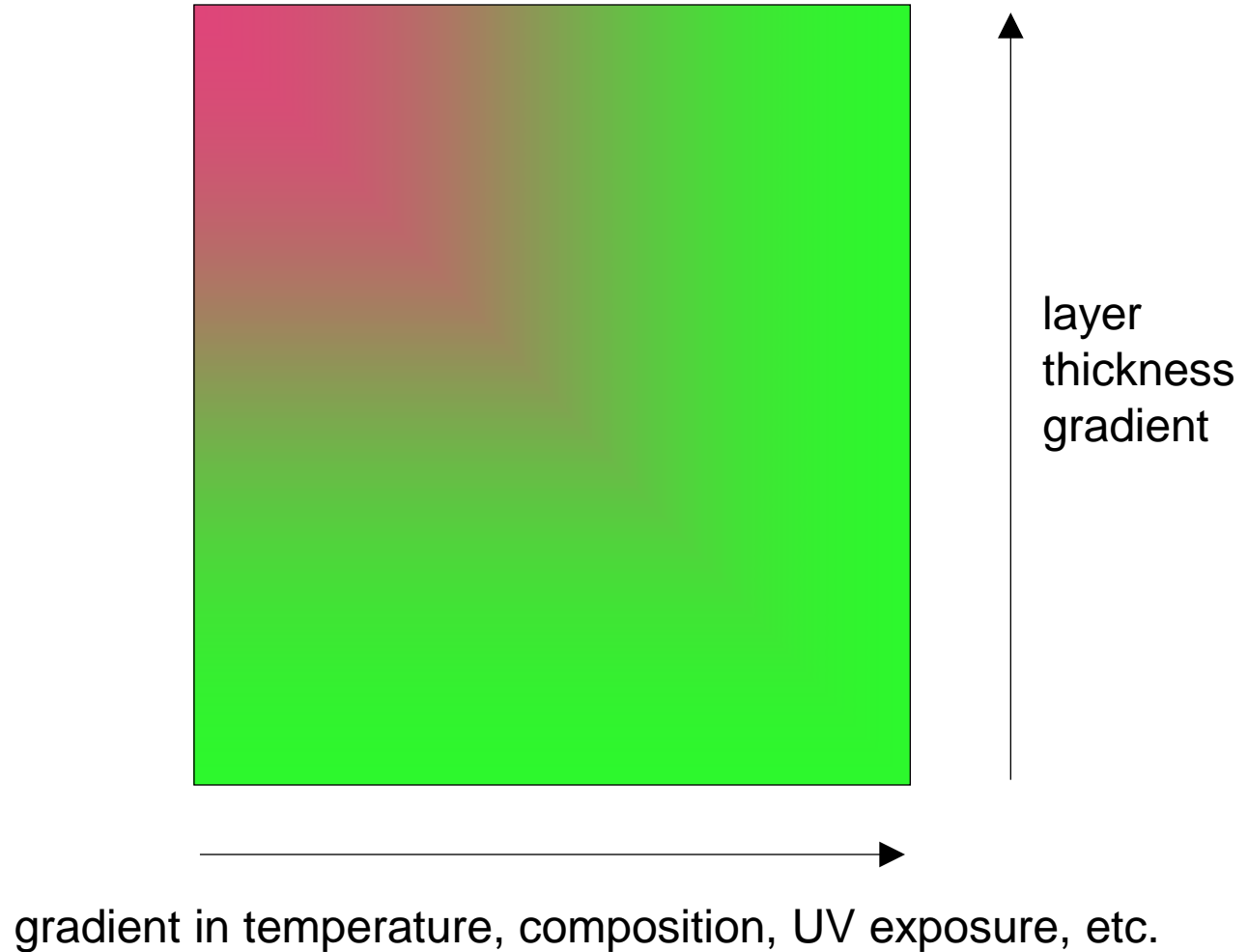
96 Deep well 1 - 2 ml



1-50 ml

# Reality of HTE

## Different Samples – NIST Gradient Approach





# Micro Capsule Formulation

## Background

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**Micro capsules are formed from multiple ingredients**

**Standard process adapted for HTE production**

**Ultimate application requires both efficient storage and rapid release of active**

**Size of capsule used a proxy for active storage measurement**

**Release in application occurs on shear**

**Sedimentation of capsules was used as a first tier screen to rapidly assess a wide formulation space**

**HTE effective as wide range of new actives continually become available**

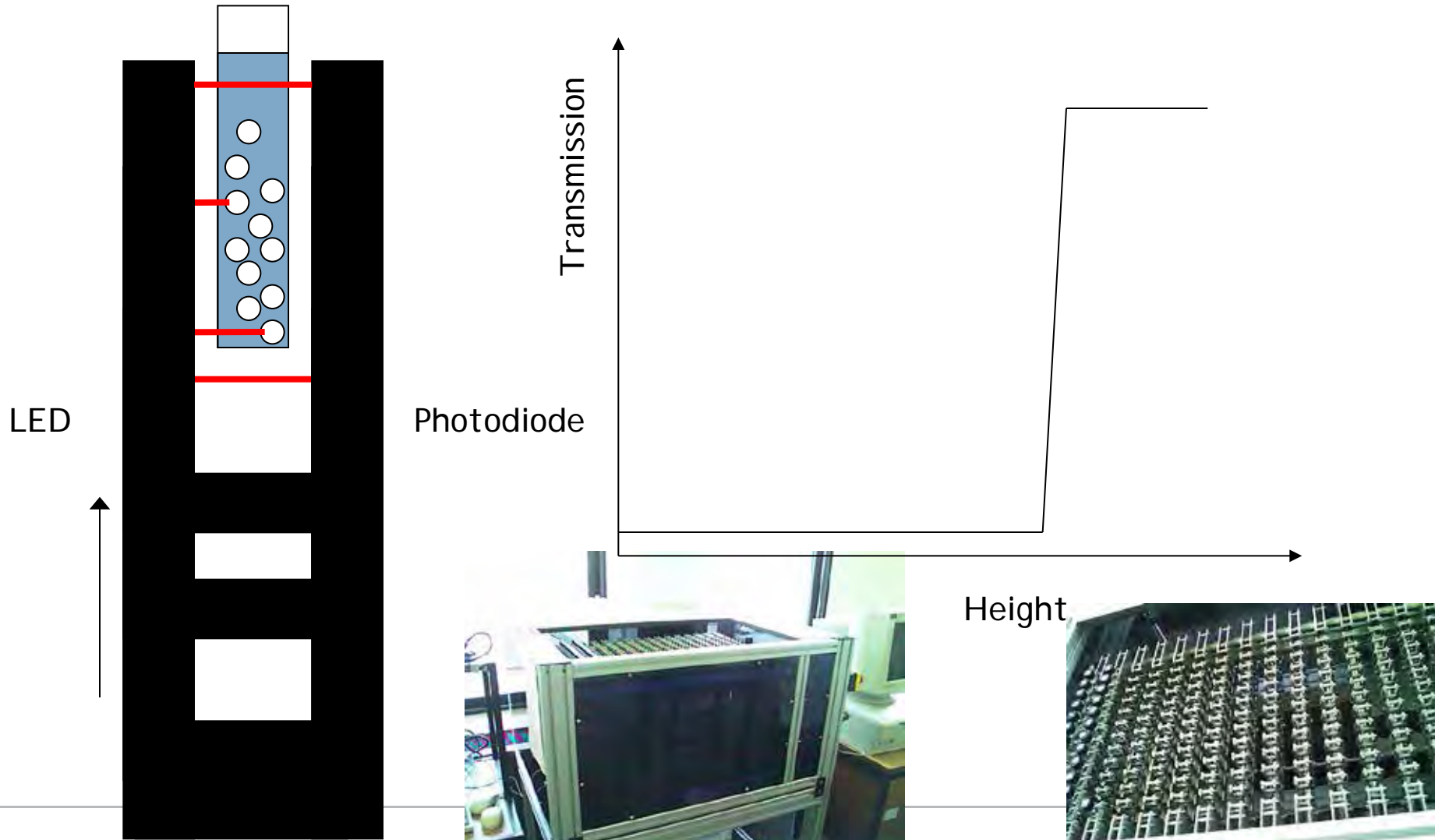
# Micro Capsule Formulation

## HTE Process

- **Dispense ingredients – powders / liquids**
- **Mix – violent shaking**
- **“Cook”**
- **Load active**
- **Process – shear**
- **Sample – avoid if possible**
- **Condition**
- **Application**
- **Measure**
- **Store Data**

# Micro Capsule Formulation Challenge

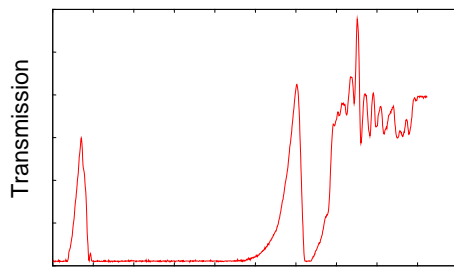
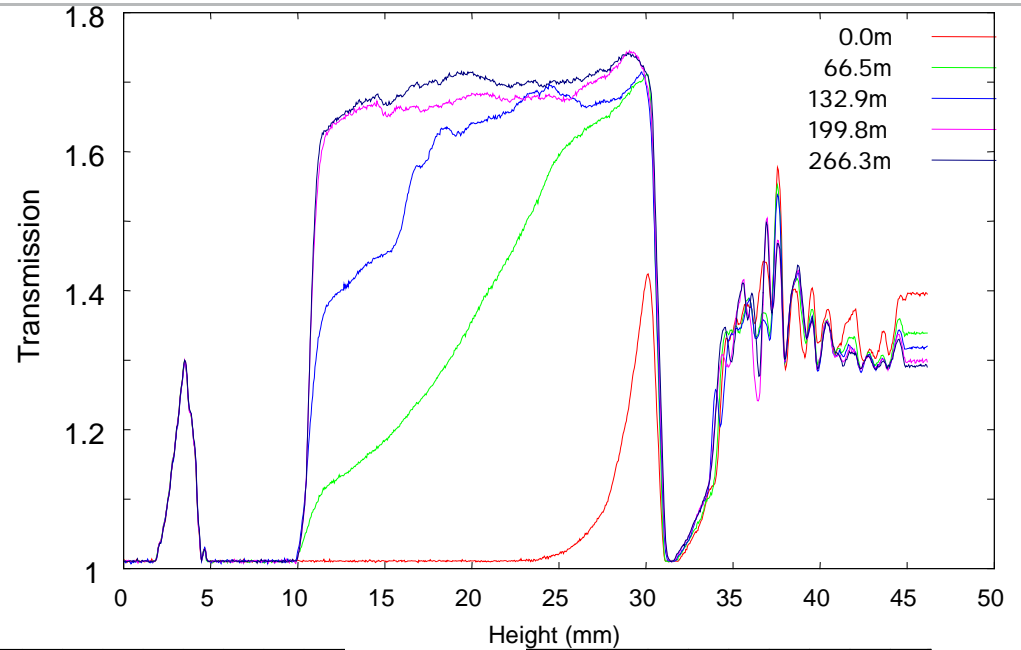
## Transmission Profile



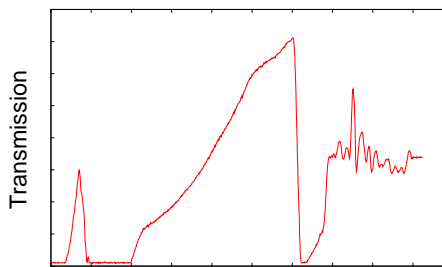
# Micro Capsule Formulation

## Transmission Profile

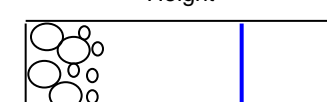
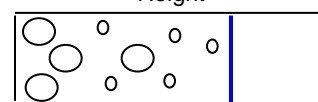
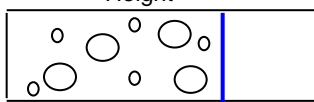
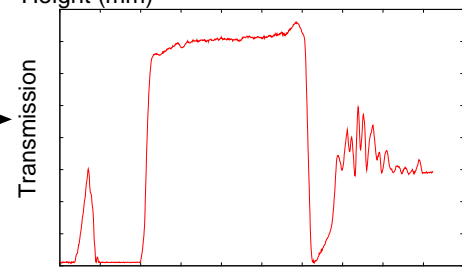
Transmission of Light through Settling Capsules



Time

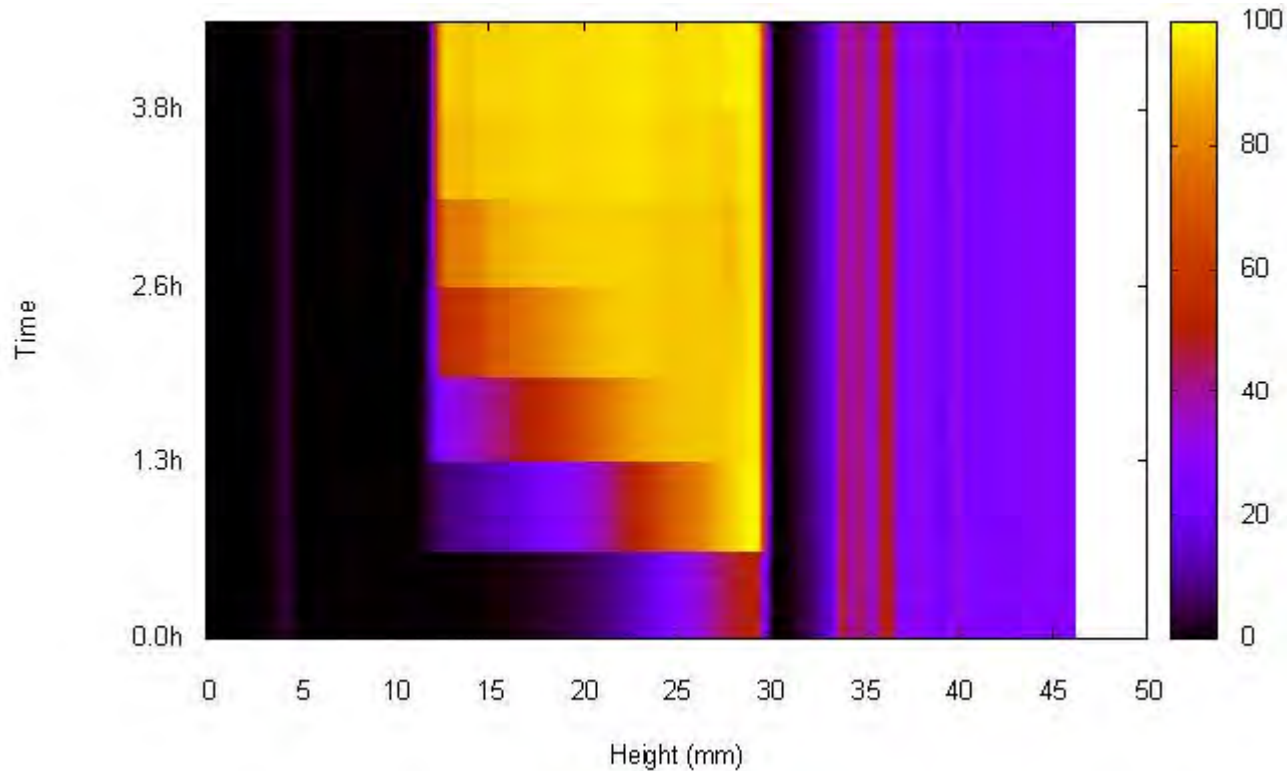


Time



# Micro Capsule Formulation

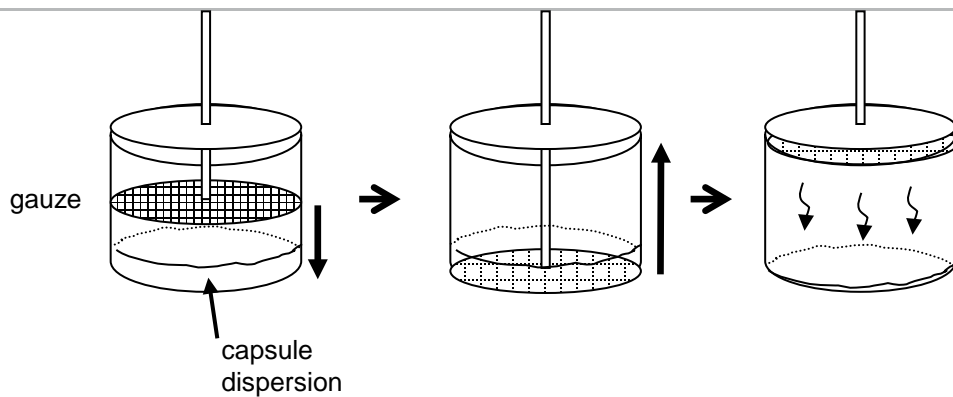
## Sedimentation “Heat Map”





## Micro Capsule Formulation

### Shear

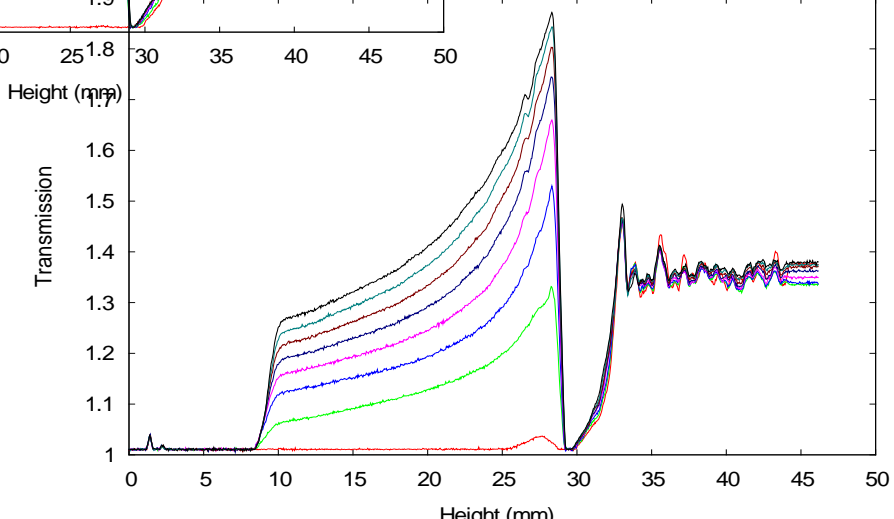
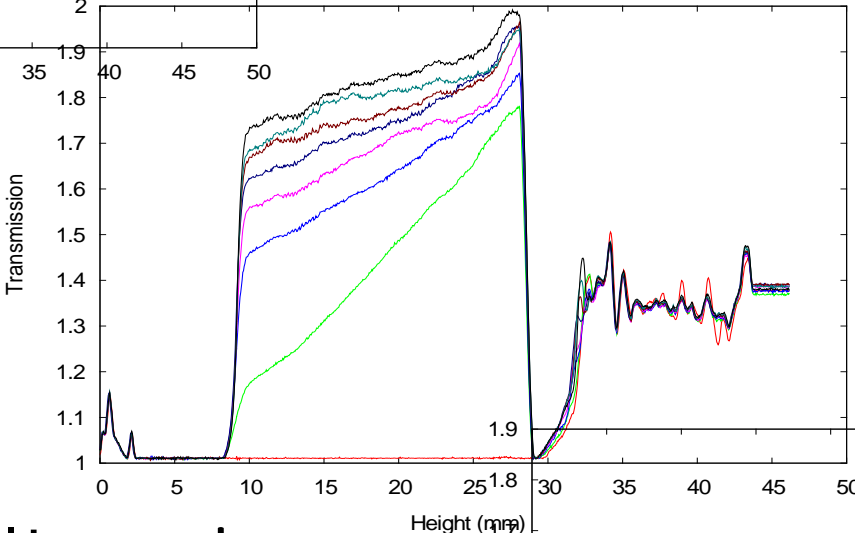
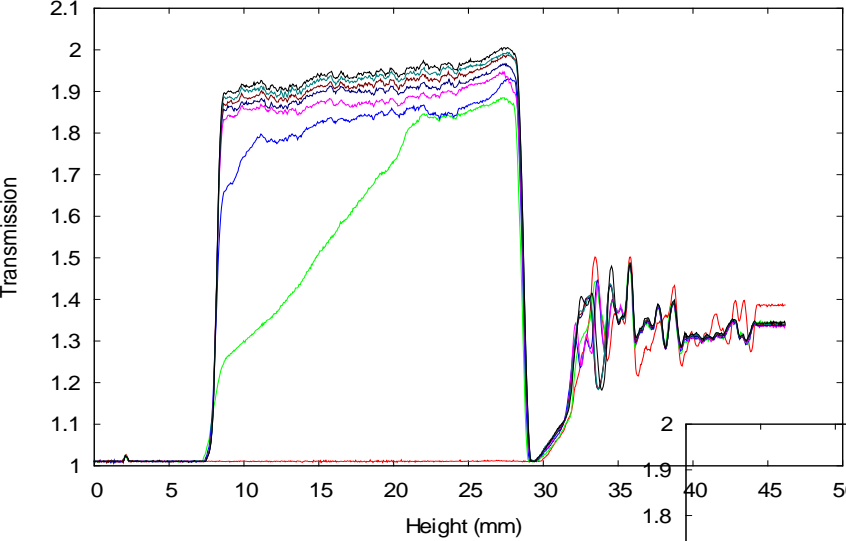


# 200g of 5% capsules sonicated using probe

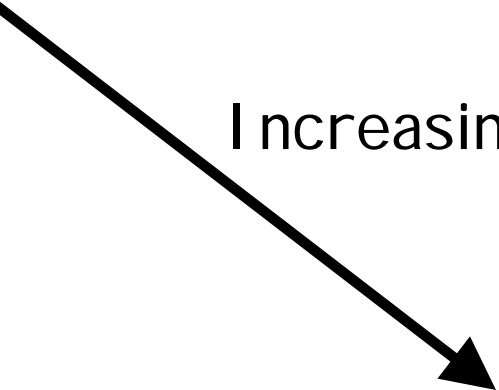


Time	3s	8s	13s	23s
Swell vol. (ml/g)	20	22	27	22

# Effect of Shear



Increasing Ultrasonics



# **Polymer Solubility Formulation**

## **Background**

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### **Simple process**

- **Mixed solvent of controlled formulation produced**
- **Polymer dissolved under pre-defined conditions**

### **Rapidly changing regulations:**

- **Acceptable solvents for different uses**
- **Volatile organic compound limits**

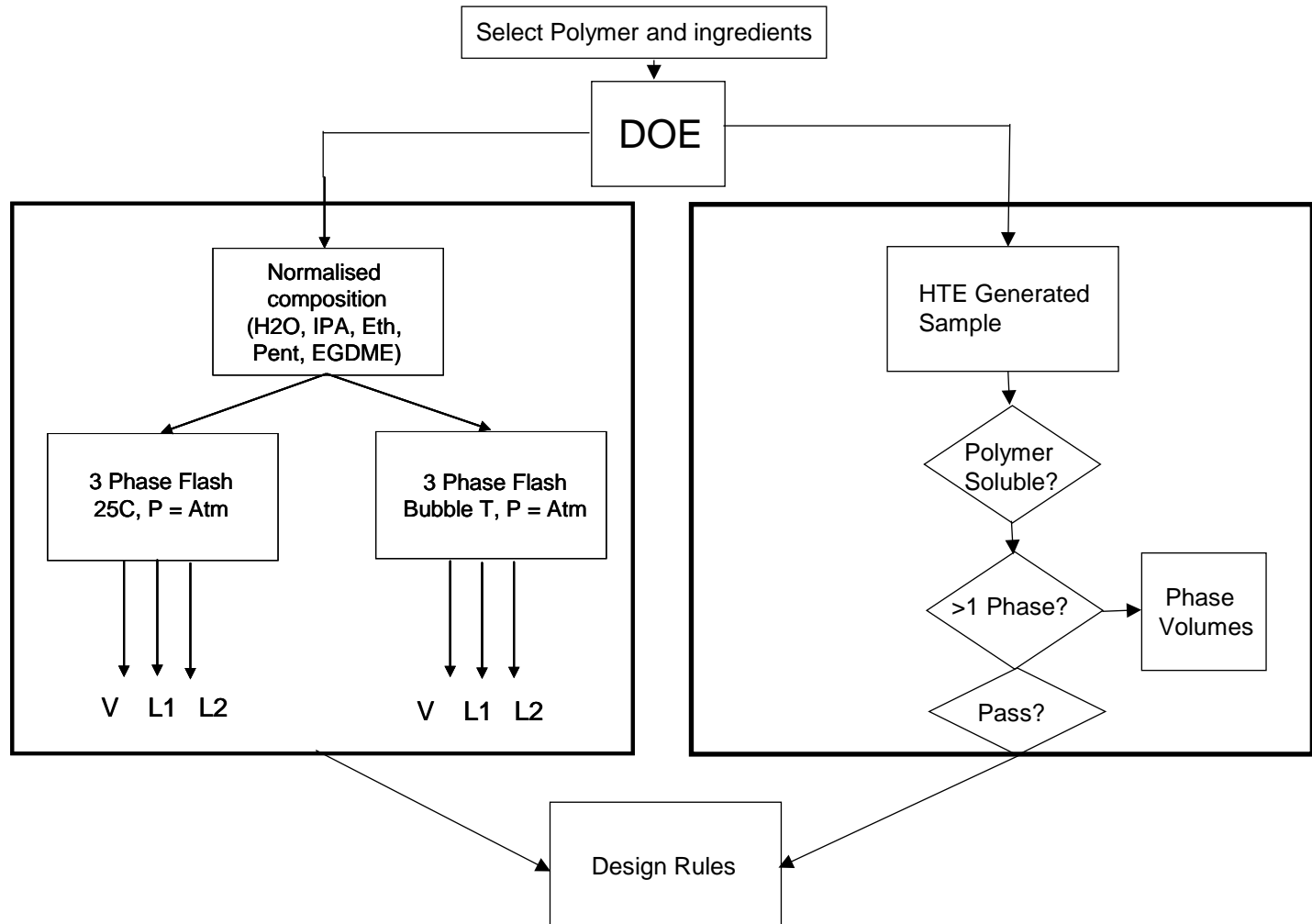
### **Increasing use of bio-derived materials:**

- **Solvents from bio sources**
- **Polymers from bio sources**

### **HTE effective as on-going requirement**

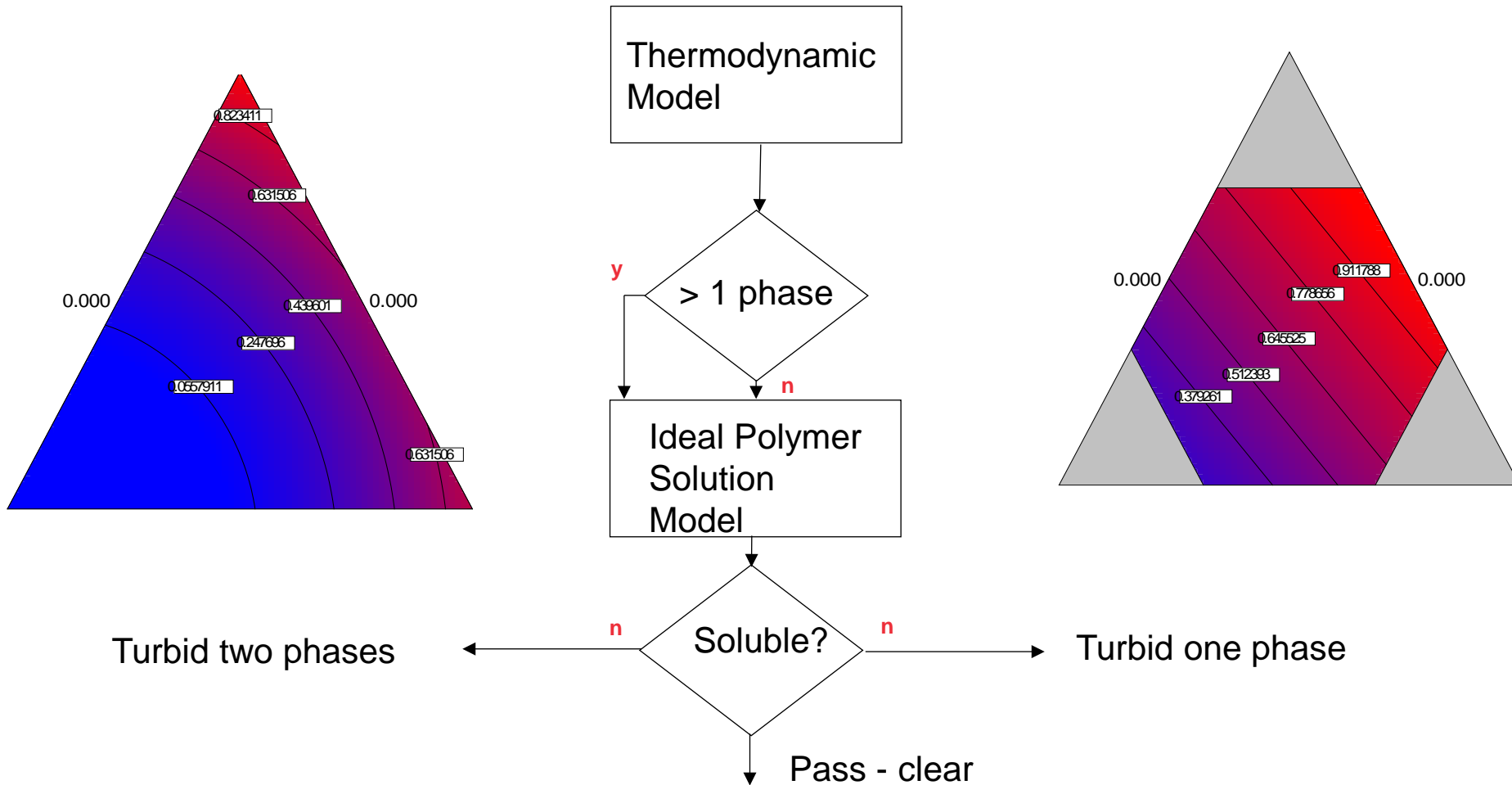
# Polymer Solubility Formulation

## Seeking Design Rules from models and data Thermodynamic Model



# Polymer Solubility Formulation

## Design Rules



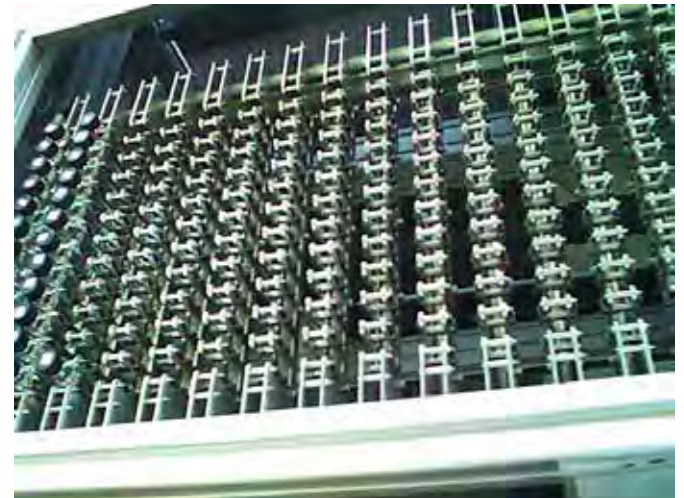
# Polymer Solubility Formulation

## Measurement Workflow



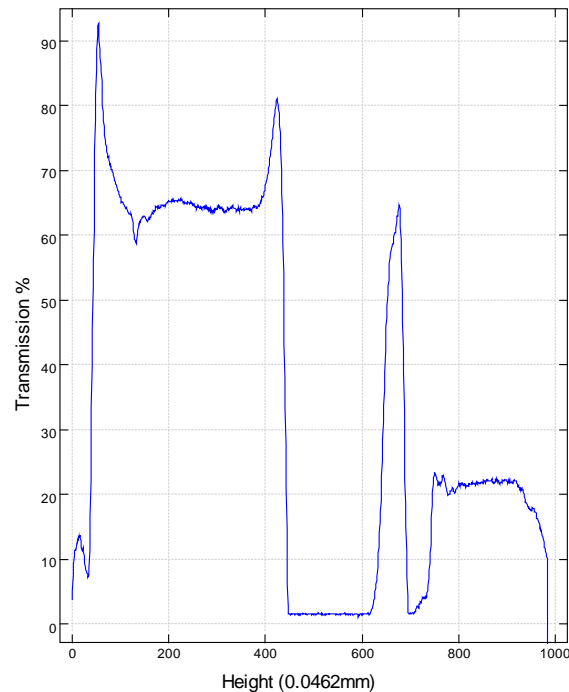
### ICIA – Transmission scanner

- suitable for glass vials
- LED / photodiode pair records transmission profiles
- 192 unique sample positions
  - Unique number of scans
  - Unique interval between scans
  - Unique no of measurements per scan



# Polymer Solubility Formulation

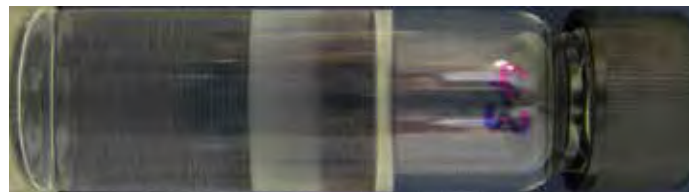
## Measurement Technique



**Sample placed in holder**

**Whole sample scanned  
Once / hour**

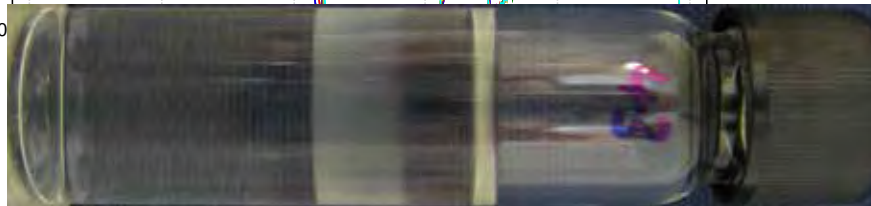
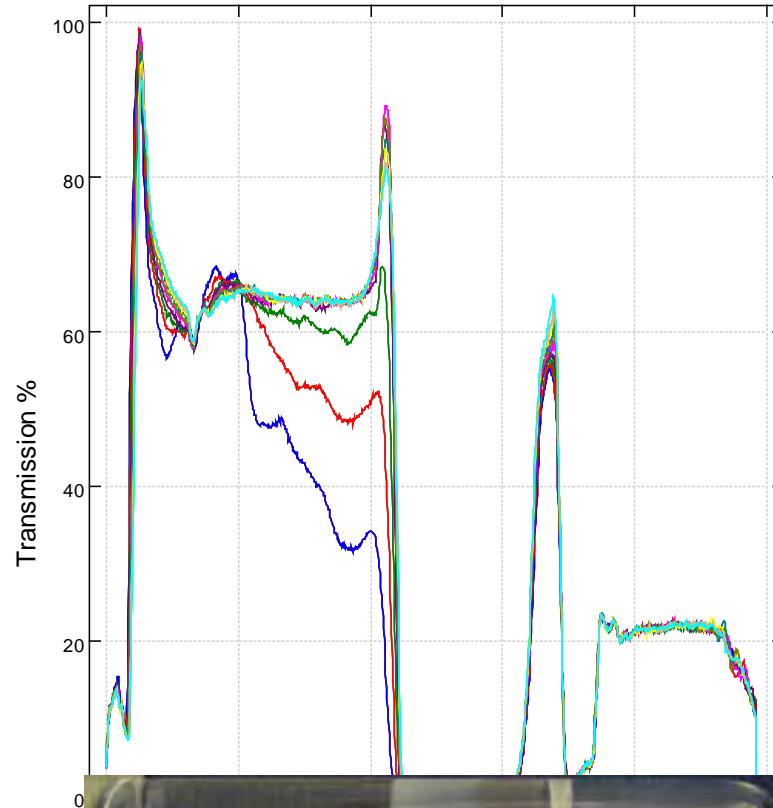
**Transmission scan produced  
which is representative of  
sample**





# Polymer Solubility Formulation

## Time lapse



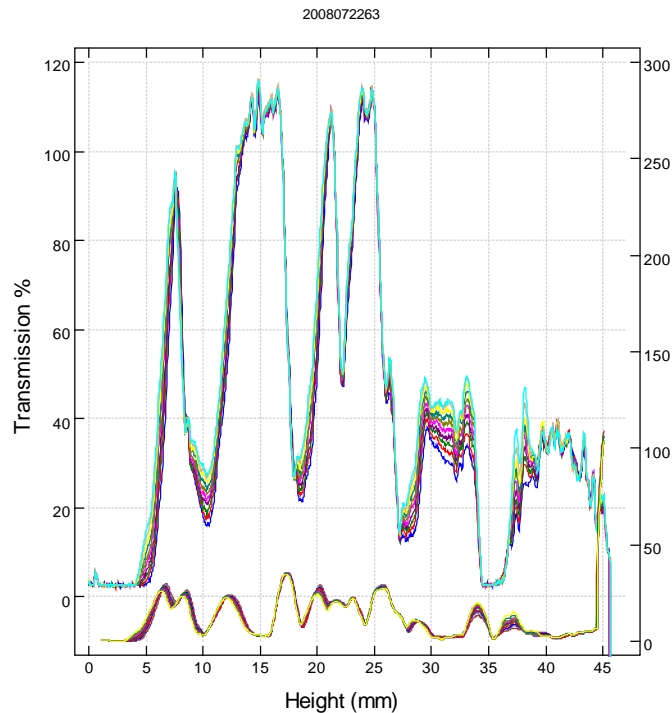
**All data stored in local database**

**Multiple scans can be overlaid**

**Monitor change in phase behaviour as system achieves equilibrium**

# Polymer Solubility Formulation

## Powerful Analysis Technique



**Significant fluctuations in transmission along the sample tube**

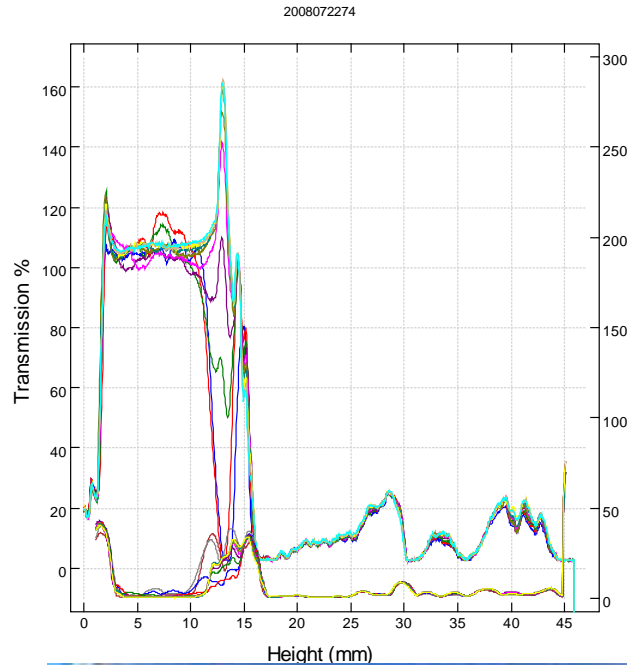
**This is due to unmixed polymer adhered to side of tube**

**Also, unmixed sample at bottom of tube**

**Sample is OK, but classified as fail.**

# Polymer Solubility Formulation

## Curious Samples



**2 area identified by scans and visual observations**

**Not actually 2 phases, the turbidity is caused by undissolved polymer on glass**

**Sample is OK, but classified as fail.**

# Sealant Elasticity Formulation

## Background

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**Additive produced for sealant required to produce a set mechanical elasticity**

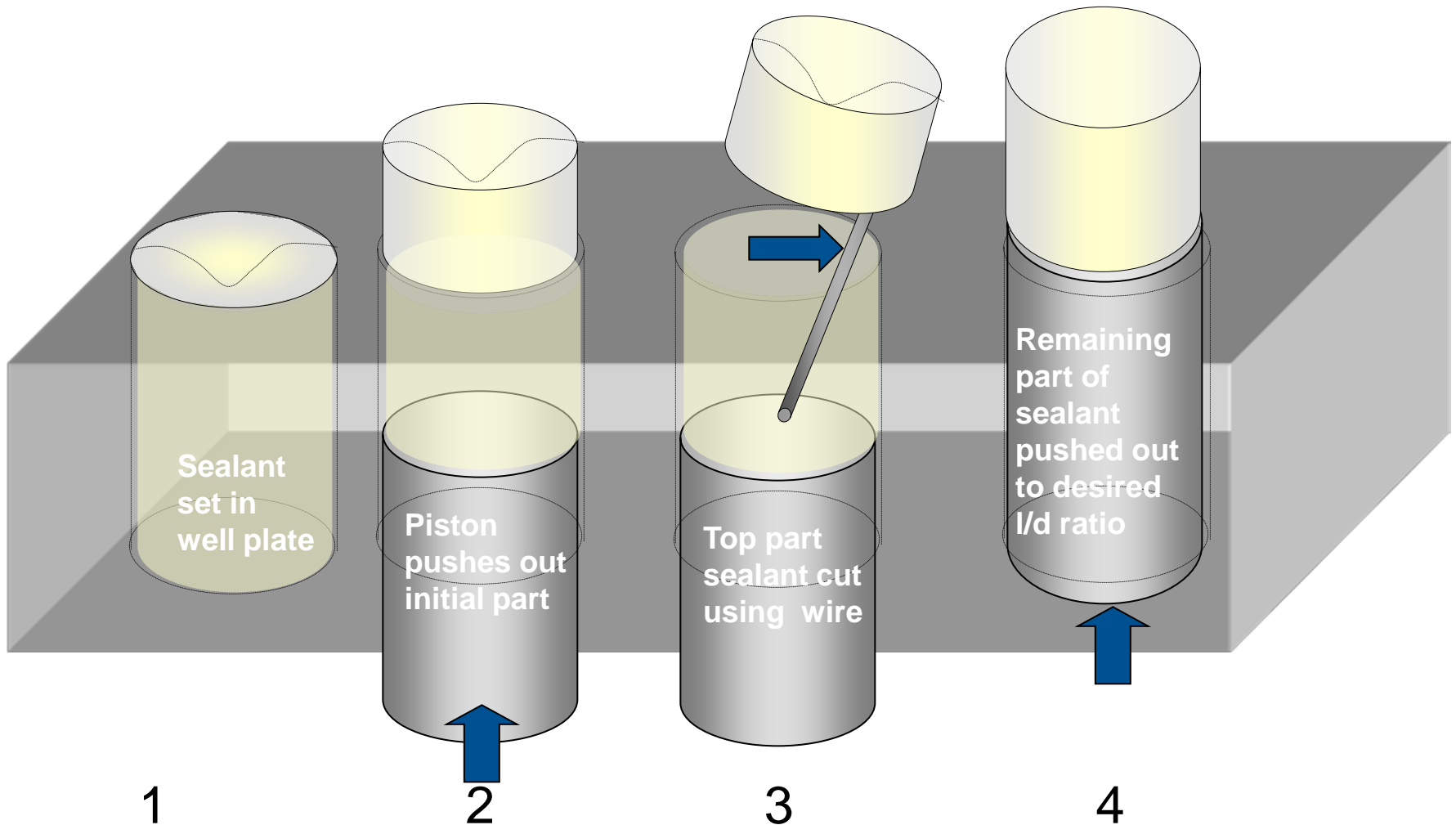
**Mixing process possible by in-line mixing at small scale so HTE friendly**

**Mini-instron measurement gives full mechanical properties on all samples**

**Direct formulation optimisation**

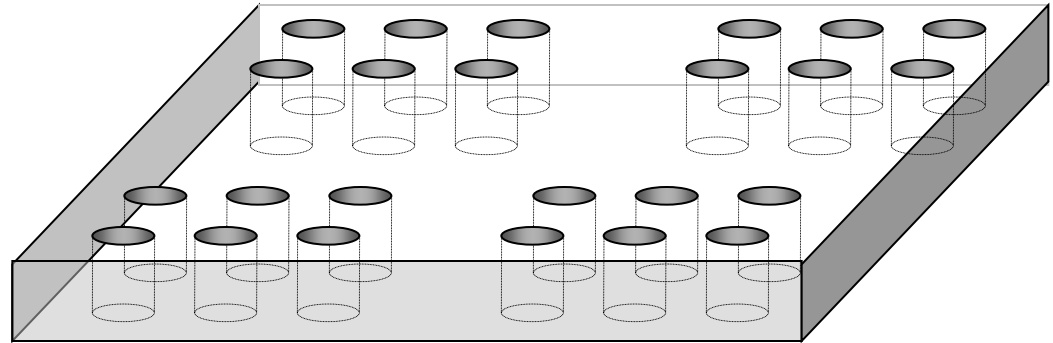
# Sealant Elasticity Formulation

## Sealant well plate preparation

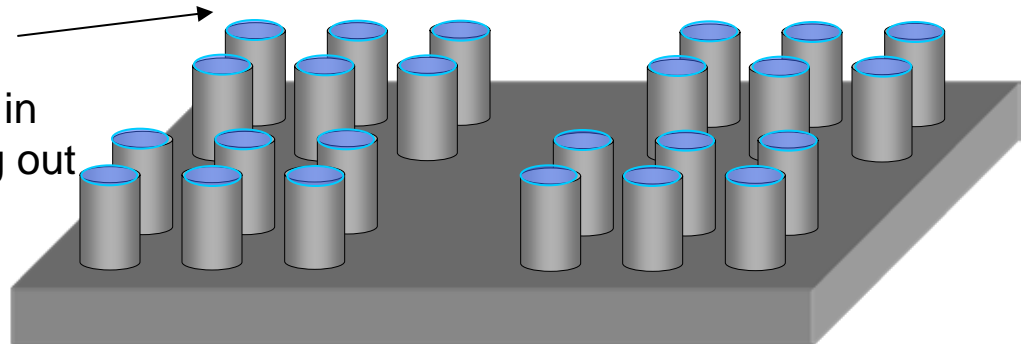


# Sealant Elasticity Formulation

## Testing procedure with lubricant - underside

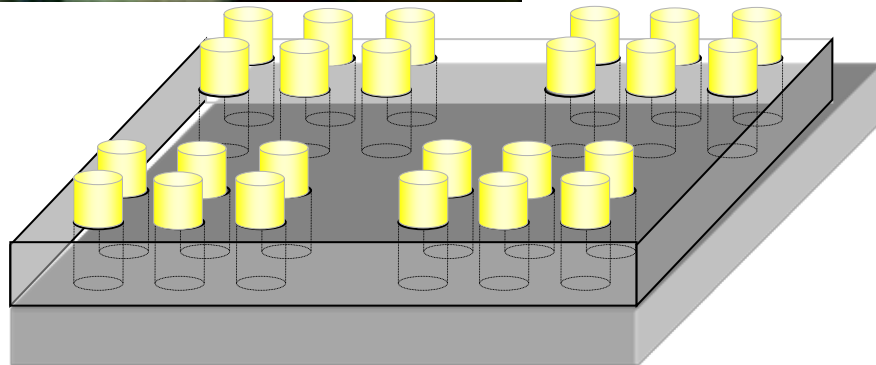


Piston rig coated in lubricant before well plate positioned in place before pushing out and cutting to length

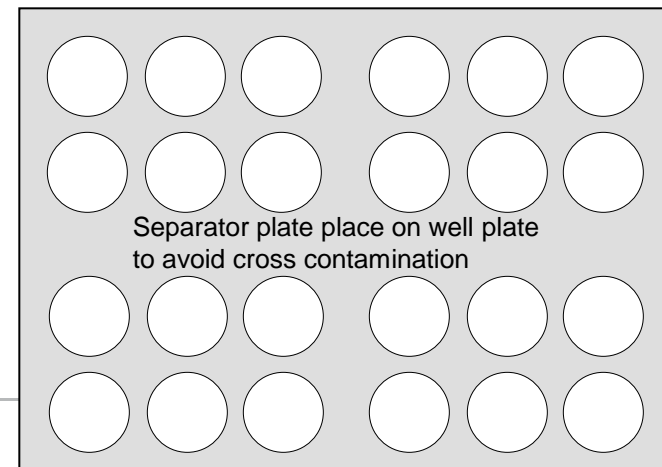
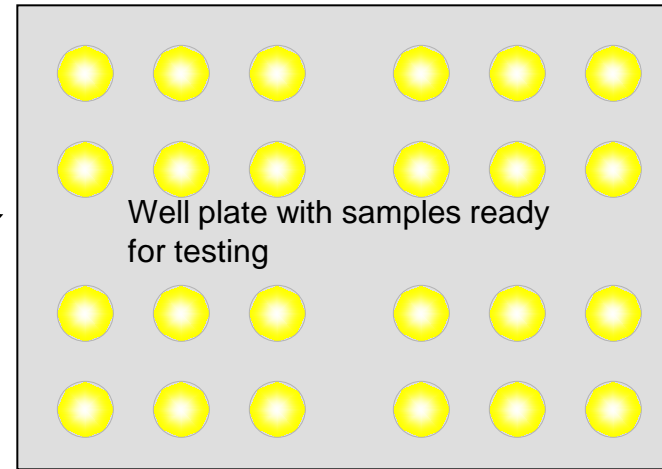


# Sealant Elasticity Formulation

## Testing procedure with lubricant - top side

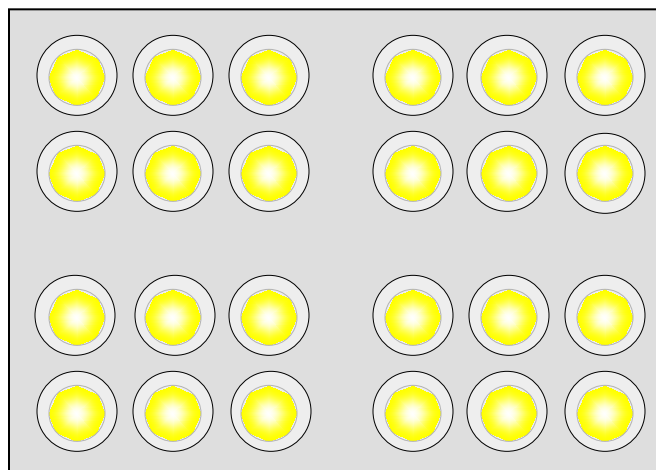


Sealant cut by wire and pushed out fully

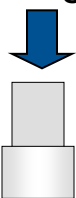


# Sealant Elasticity Formulation

Testing procedure with lubricant - top side



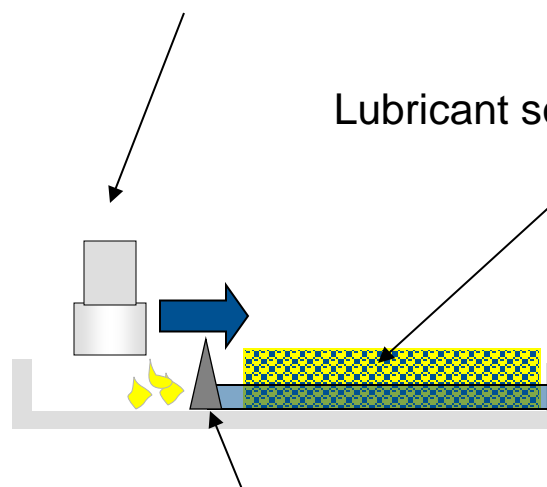
Well plate assembly ready for testing



## Cleaning and lubricating stage

Compression platen (attached to load cell)

Lubricant soaked sponge

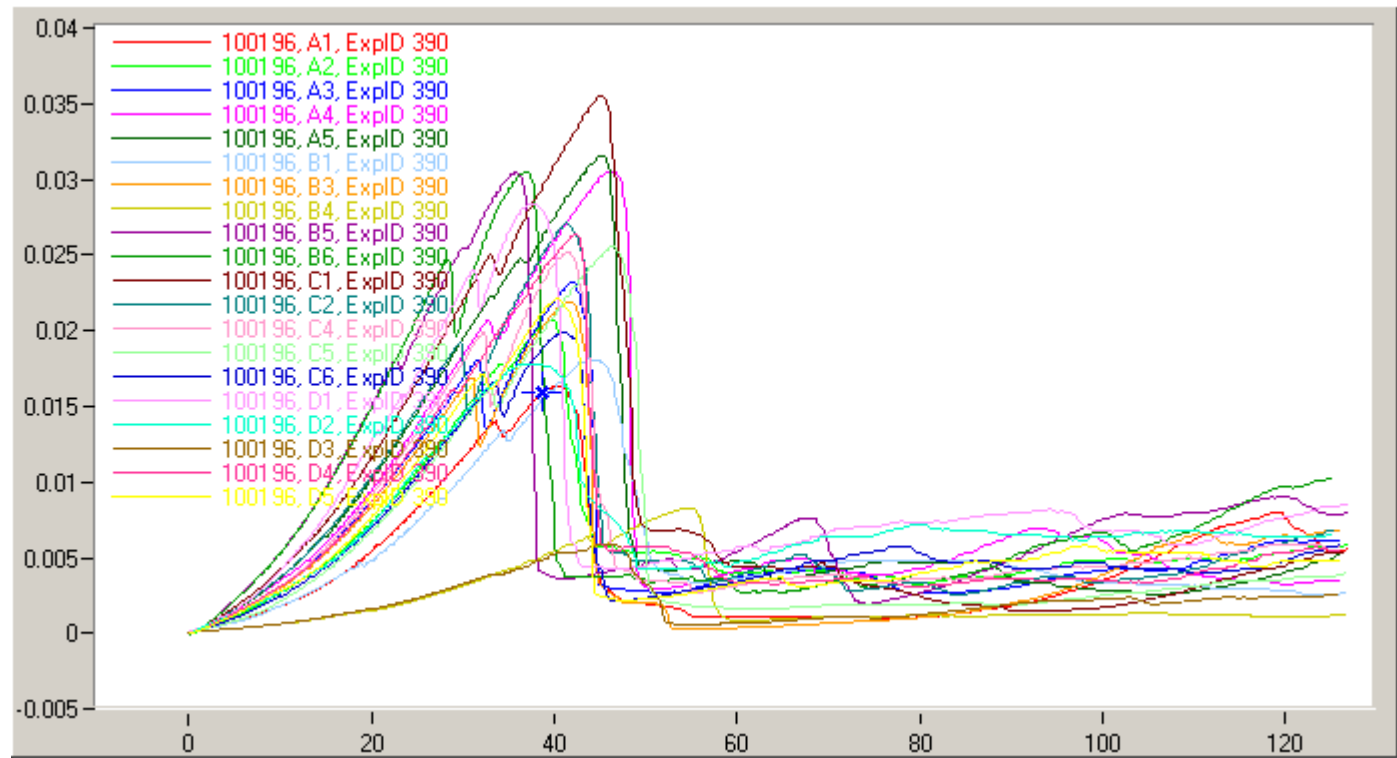


Rubber wiper blade to remove test fragments



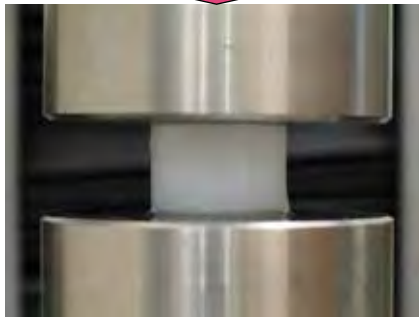
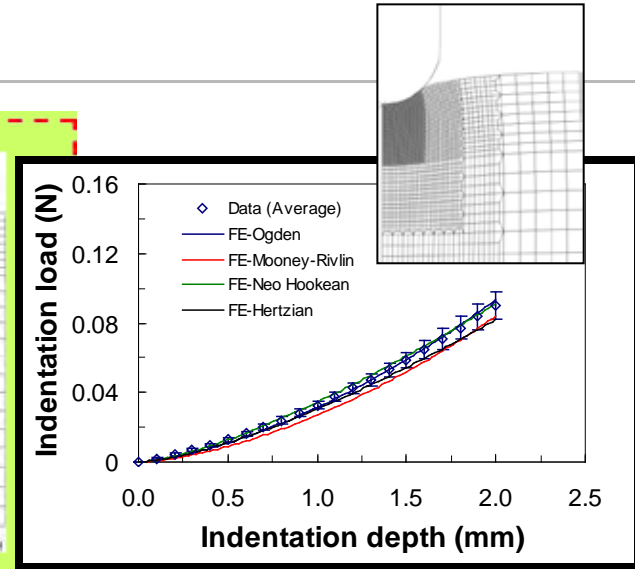
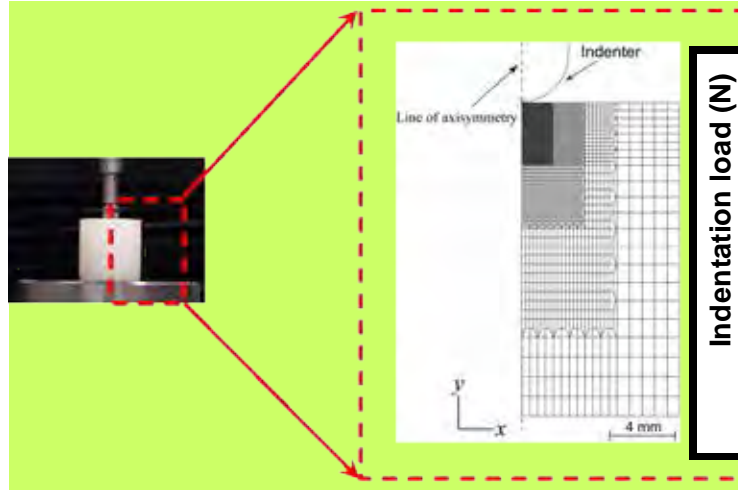
# Sealant Elasticity Formulation

## Force vs. Deformation

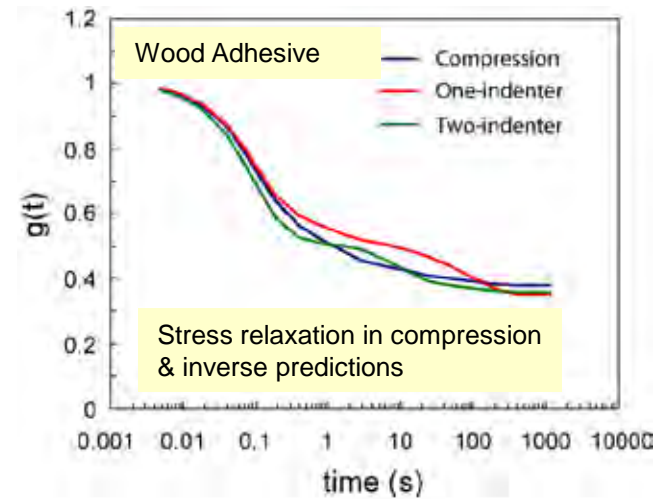
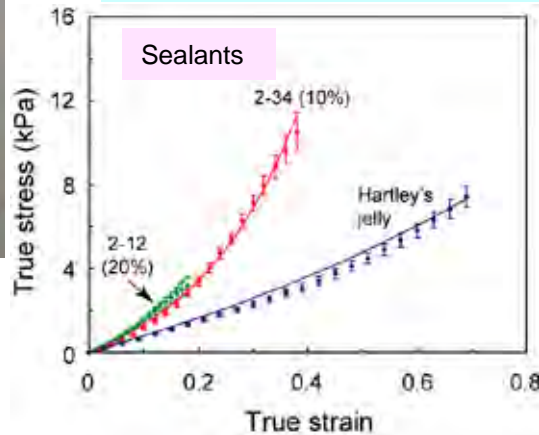


# Sealant Elasticity Formulation

## Inverse Analysis



Inverse predictions and data



## Characterisation and HTE

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**HTE approach is best suited to:**

- **Products where large samples sets need to be tested**
- **Products where the same test will need to be applied for foreseeable future**

**Development of clever screens / measurements requires good understanding of physical control of application performance:**

- **Clever characterisation of controlled sample set**
- **Supported by modelling based on appropriate science**

**Clever screening can make products HTE suitable:**

- **Screens directly related to application**
- **Screens giving insight into physical properties**
- **Reduce sample set that needs full measurement**

**Never forget what the formulation tools you already know / use**

## Acknowledgements

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