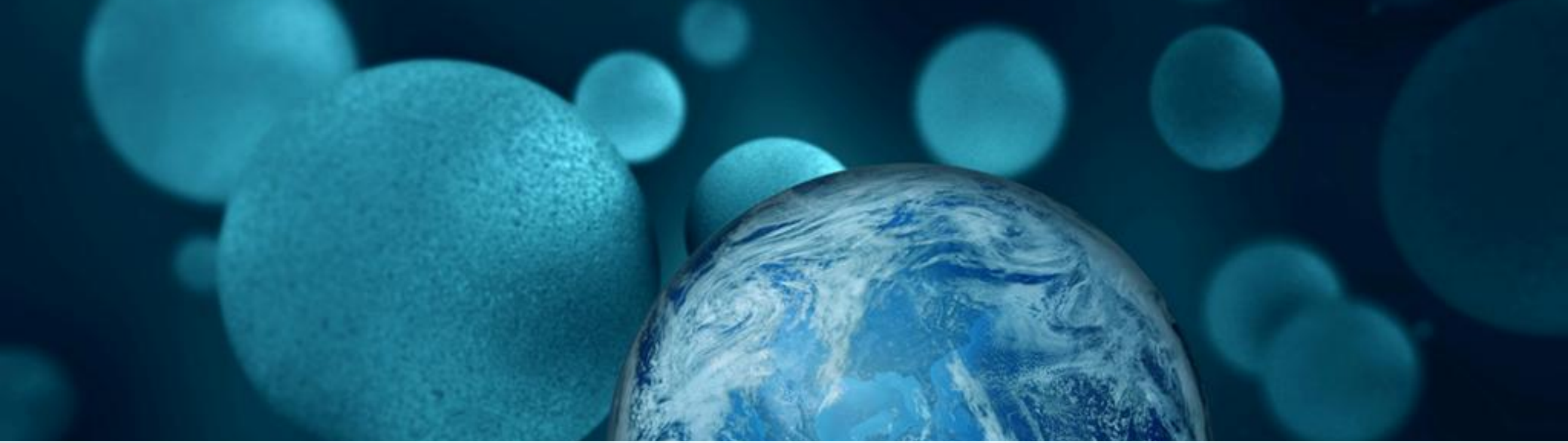

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Handle Food Samples with Care for Reliable Rheological Results

Dr. Klaus Oldörp

Overview

- Food and rheology
- Sample handling before the measurement
- The right measuring geometry
- The suitable measuring method

FOOD AND RHEOLOGY

How a Rheologist sees Food

- Natural Food
  - Has been grown
  - Can be tested for texture
  - Natural variations are commonly accepted
- Processed Food
  - Has undergone a production process
  - Can be tested for
    - Properties during production
    - Properties of the final product
  - Constant properties are linked to the brand
    - In spite of variations of raw materials
    - In spite of different production sites

Rheology and Food – Where is the Connection?

- During Production
  - Pumping
  - Mixing
  - Cooking
  - Filling
  - Storage Stability
- During Consumption?
  - Look
  - Mouthfeeling
  - Creaminess
  - Spreadability
  - …

- Viscosity
- Viscoelasticity
- Yield Stress
- Moduli
- Temperature Dependence
- Time Dependence
- Elongational Viscosity

Texture – One of Food‘s Key Properties

- For the Consumer
  - Part of the pleasure when consuming food
  - Important criterion to accept or reject food
- For the Rheologist
  - Sth. to describe with rheological parameters
  - Sth. to bear in mind when
    - Handling the sample
    - Selecting the measuring geometry
    - Designing the test method
  - A lot of trouble if ignored!

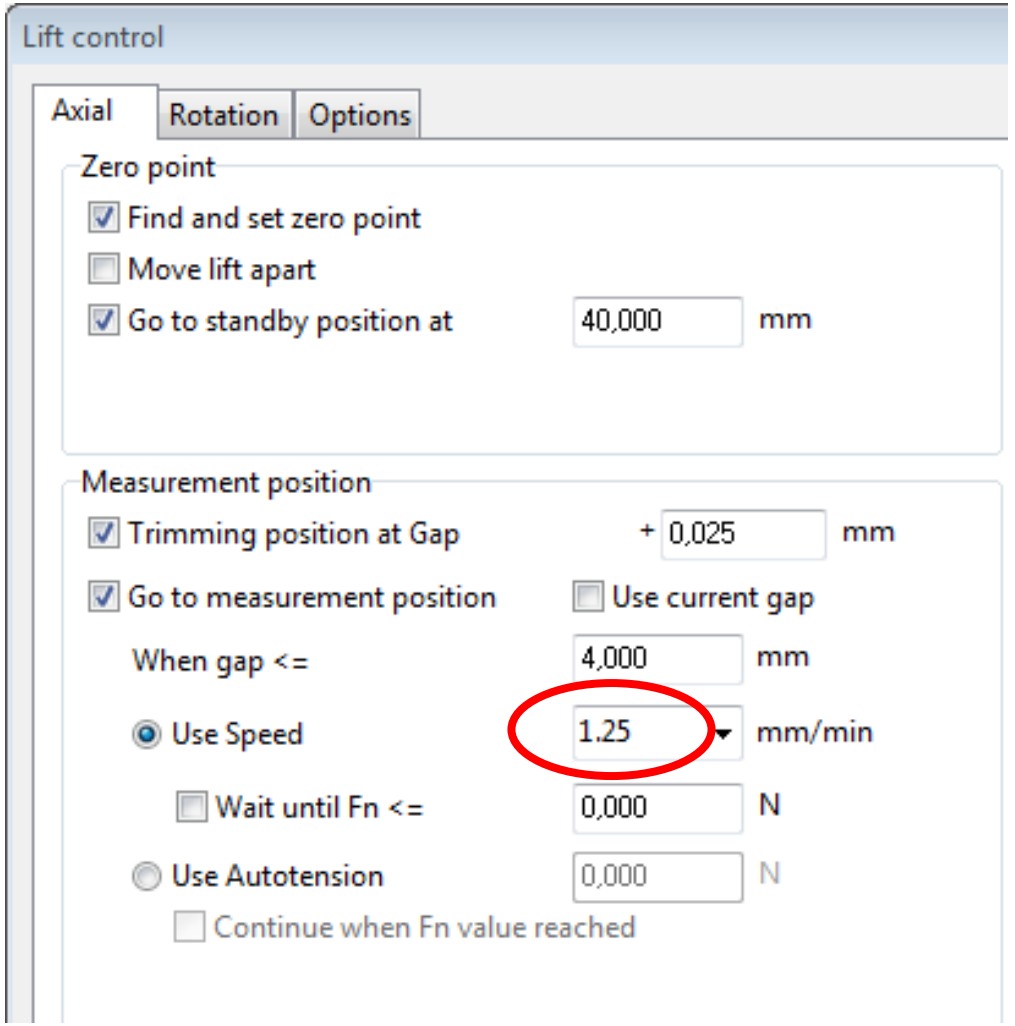
SAMPLE HANDLING

Handling of Texturized Samples

- Visual assessment
  - Does it flow?
  - Does it appear elastic?
- Handle with care
  - No shaking
  - No stirring
- Use suitable tools
  - Spoon, spatula
  - No pipettes etc.
- Possible risks
  - Damage of texture
  - Inclusion of air bubbles

Preparing the Sample into the Rheometer

- Take sample from container carefully
  - Not squeezing it through a nozzle etc.
  - Best using spatula or spoon
- Carefully placing it onto the lower plate
  - Centred
  - Not squeezing or distributing it
- Closing the gap with reduced speed
- Allow suitable recovery time
  - Thermal equilibration
  - Remove mechanical stress



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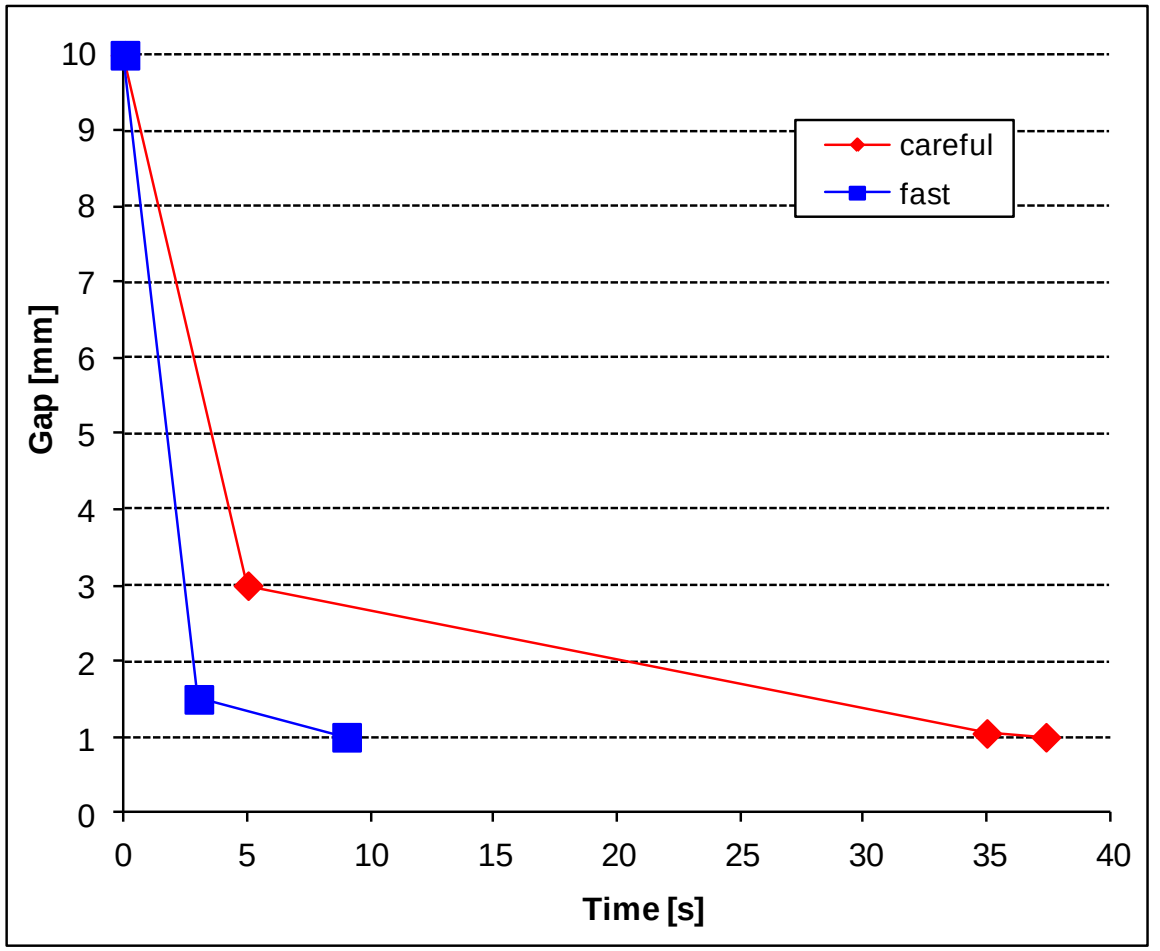
Closing the Gap with Reduced Speed

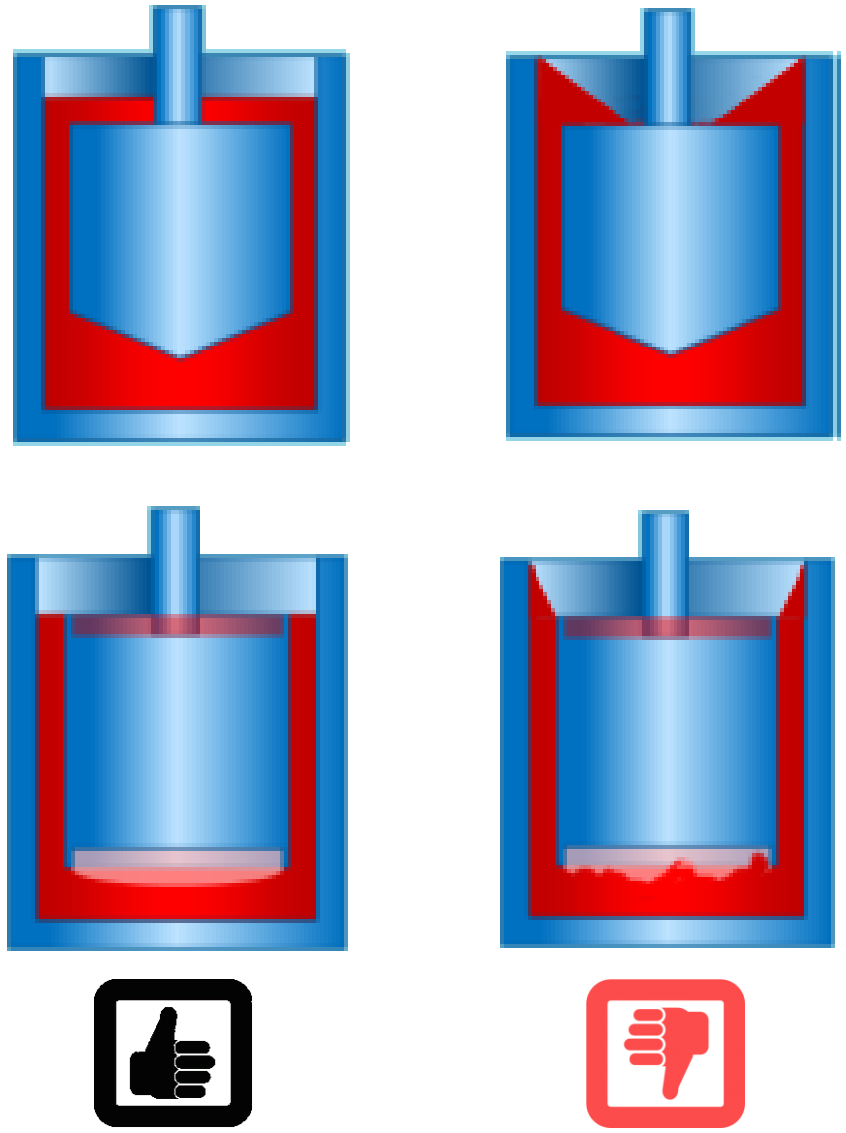
- Takes more time
- Causes less damage of sample structure
- Saves recovery time afterwards
- Reduces risk of including air bubbles
- Results are less affected by method



THE MEASURING GEOMETRY

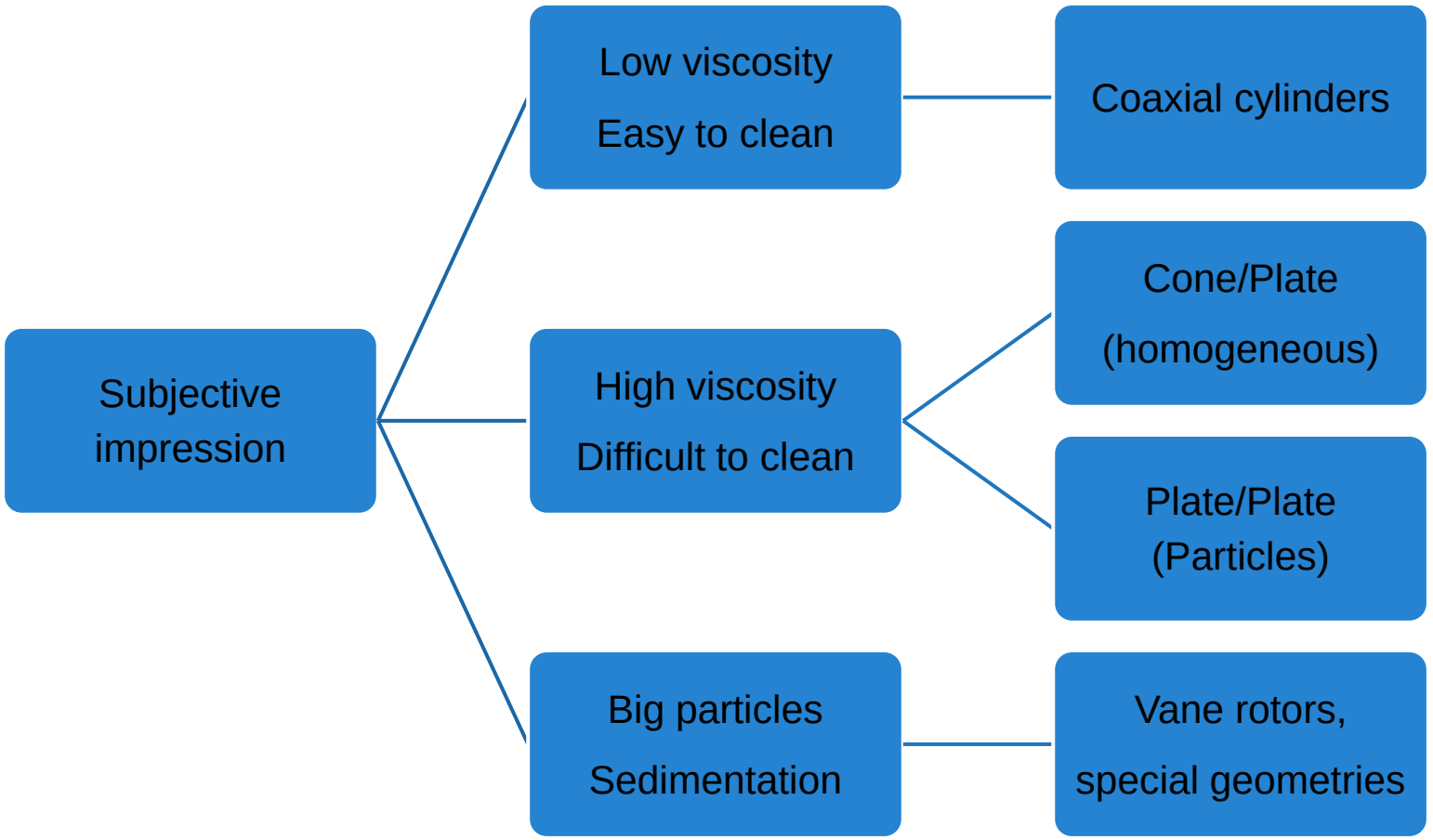
Is There a Universal Measuring Geometry?

- Concentric cylinder geometry
  - Due to historical reasons
  - Large geometry surface increases sensitivity
  - Relatively simple instruments can be used
  - Used in several standards (e.g. ICA 76)
- Disadvantages
  - Difficult for higher viscosities or samples with higher yield stress
  - Significant preshearing when inserting bob
  - Fixed gap size
  - Difficult cleaning
  - Long thermal equilibration time (15 – 30 min)
  - Heating/cooling ramps very slow



Choose the Right Geometry

- There is no such thing as the universal measuring geometry!
- Have a look at the sample
- Consider the test planned
- Select the size
  - Low viscosities → big geometries
  - High viscosities → small geometries
- When using special geometries
  - It‘s a relative test
  - Standardize the method

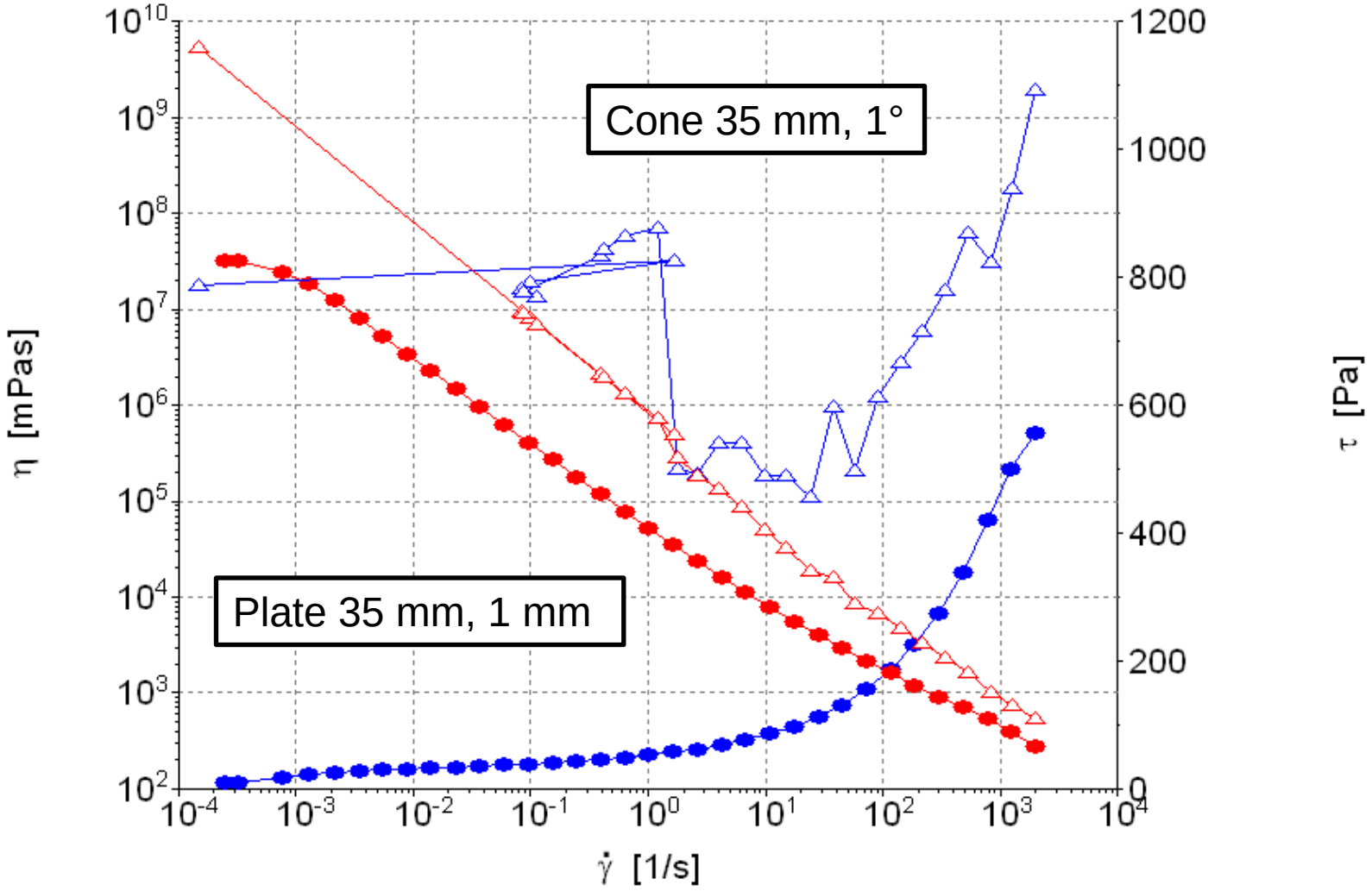


Cone/Plate and Plate/Plate Geometries

- Advantages
  - Smaller sample volume
  - Faster temperature equilibration
  - Much easier to clean
  - Using plate/plate, gap can be adapted
- Disadvantages
  - Drying effects can be more disturbing
  - Sample can leave the gap
    - At higher speeds
    - Due to elastic effects

Samples Containing Particles

- Cone/plate geometry
  - 35 mm / 1°
  - 52 µm gap (fixed)
  - Particles interlock under shear
  - Grinding and blocking
- Plate/plate geometry
  - 35 mm
  - 1 mm gap (flexible)
  - Gap can be adapted to avoid grinding and blocking
- Example
  - Smoothies with seeds



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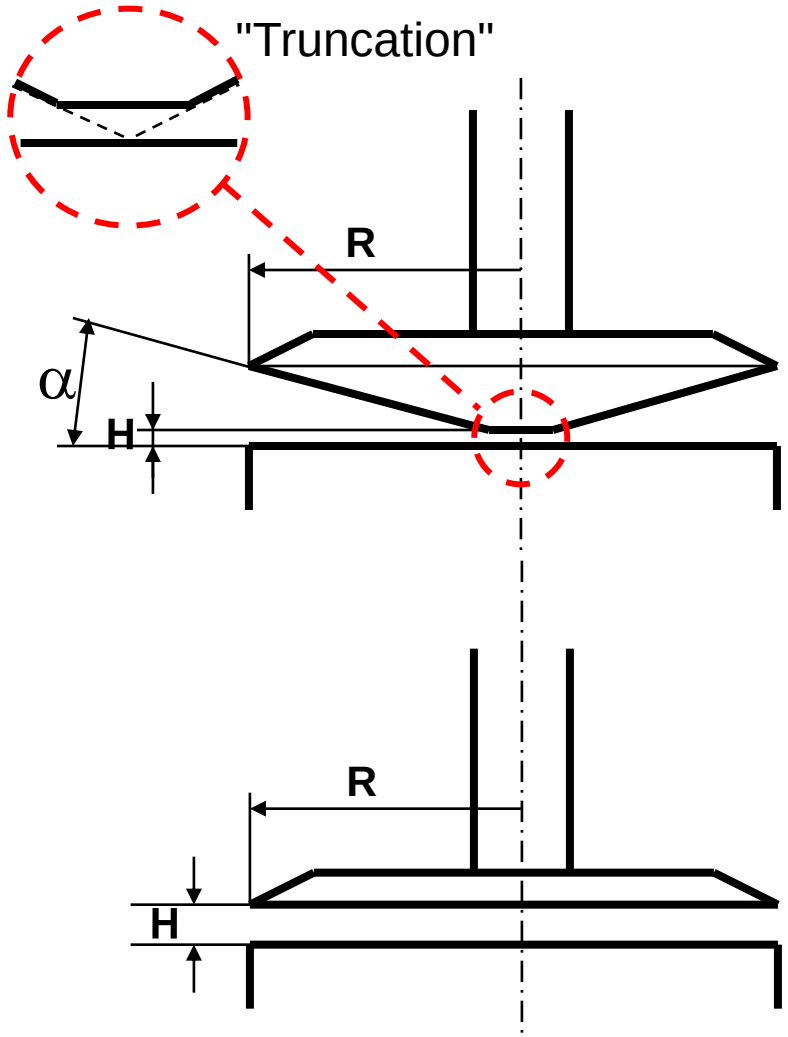
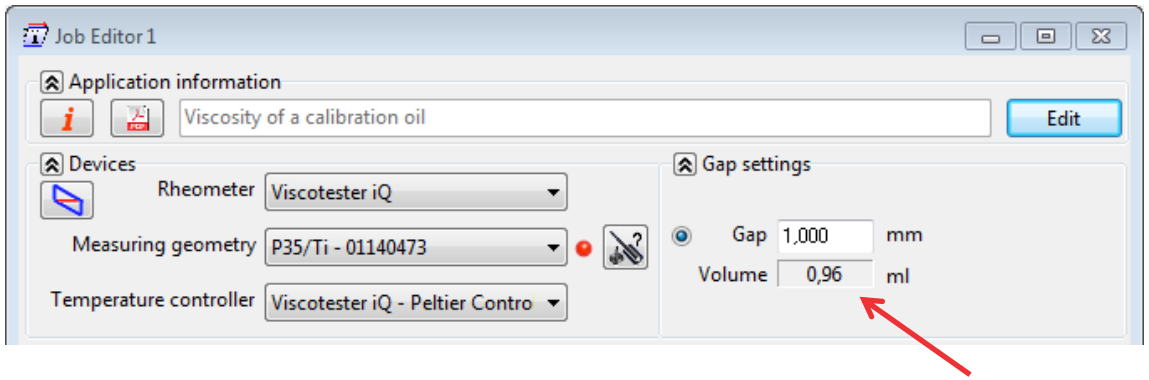
Particles in the Measuring Gap

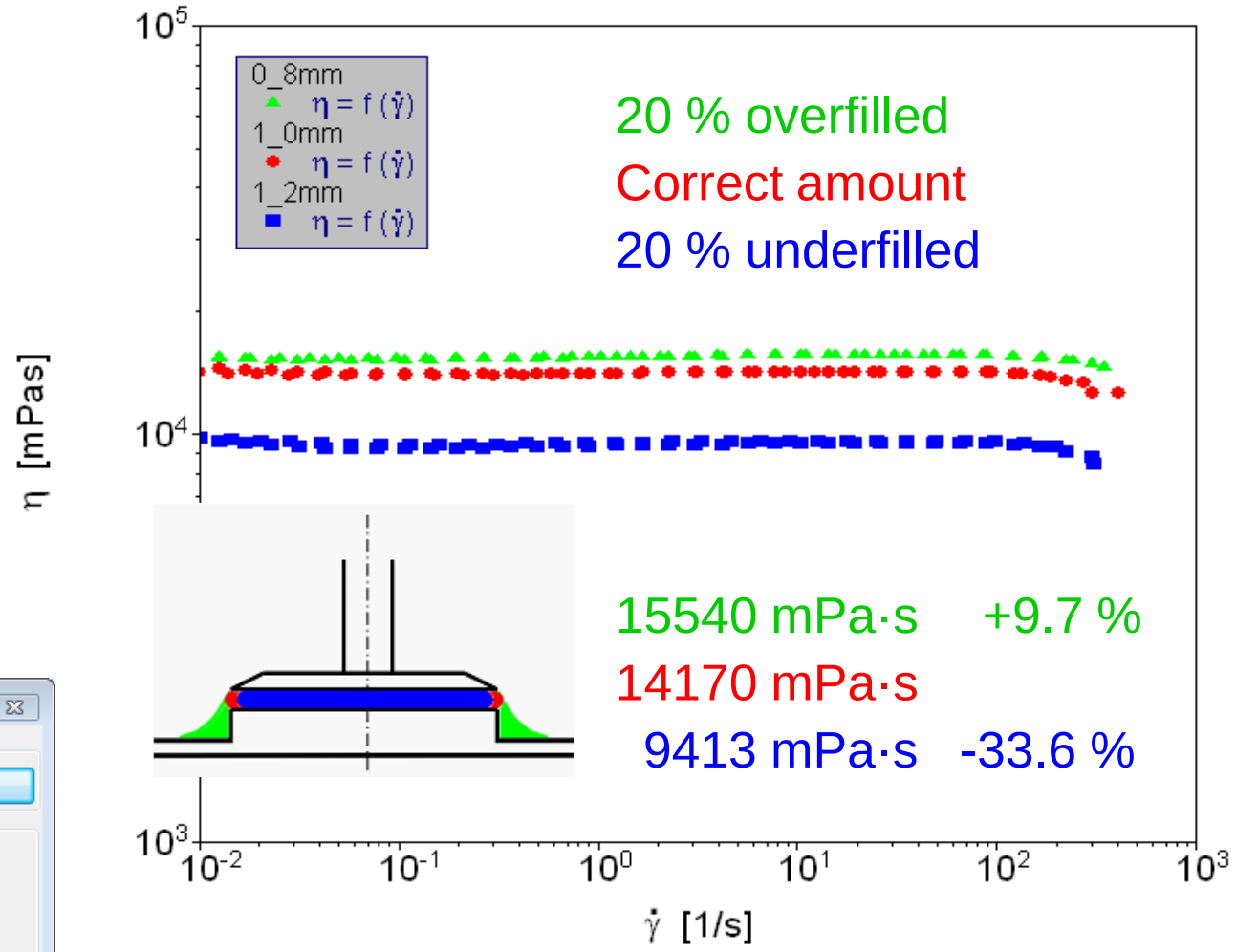
- Keep in mind for
  - Cone/plate geometries
  - Plate/plate geometries
  - Concentric cylinder geometries
- Rule of thumb
  - Gap > 5 × max. particle diameter
  - Gap > 10 × average particle diameter
- Solution
  - Use bigger gap or cone angle
  - Use vane rotors for relative tests



Use Right Amount of Sample

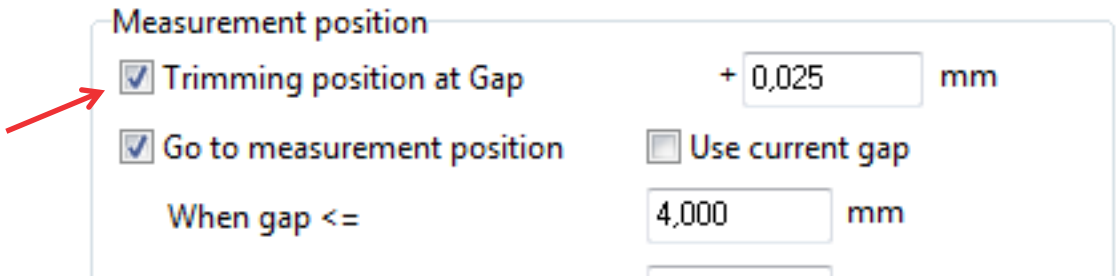
- PP- or CP-geometry: overfill and trim
- CC geometry: use pipette or balance
- Correct amount is given in RheoWin
- If correct amount not possible
  - Overfill with a constant amount
  - Standardize filling procedure for comparable results

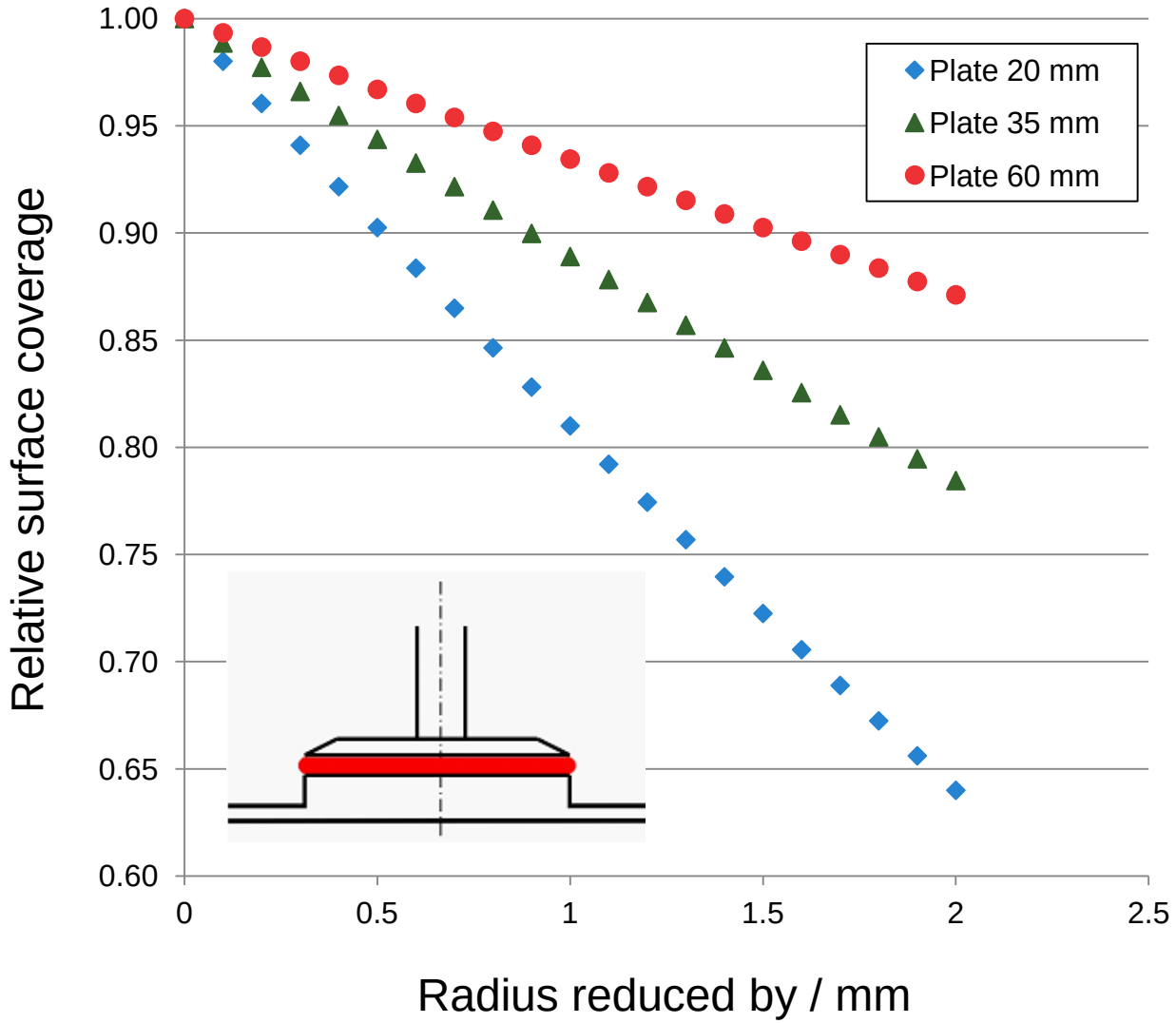




Effect of Underfilling Quantified

- Underfilling reduces contact surface
- Smaller surface results in smaller response
- Rheometer detects e.g. lower viscosity
- Small geometries are affected the most
- Carefully remove excess sample
- Use trimming position





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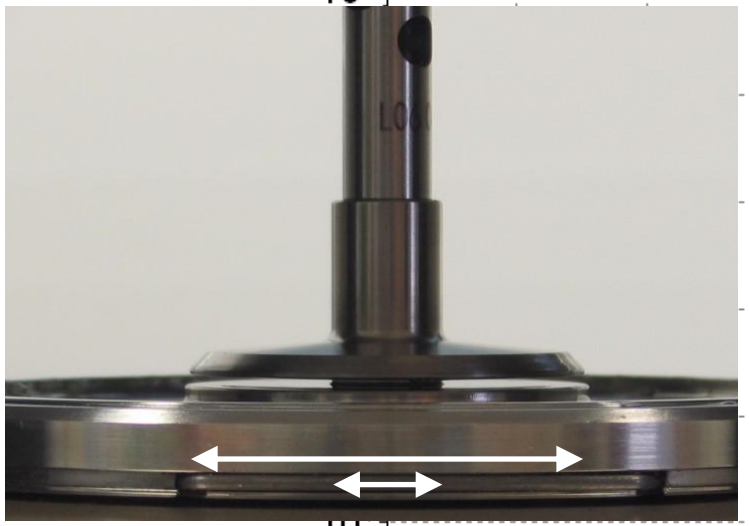
Gap Depletion During Rotational Measurements

- PP- or CP-geometry
- Happens earlier with increasing
  - Radius
  - Gap (or cone angle)
  - Elasticity of sample
- To reduce this effect use
  - Smaller radii
  - Smaller gaps or cone angles
  - CC-geometries
  - Oscillatory measurement



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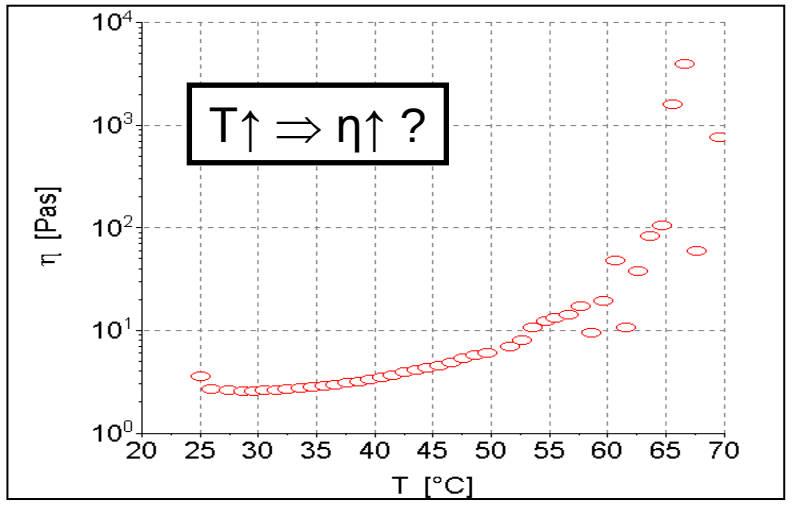
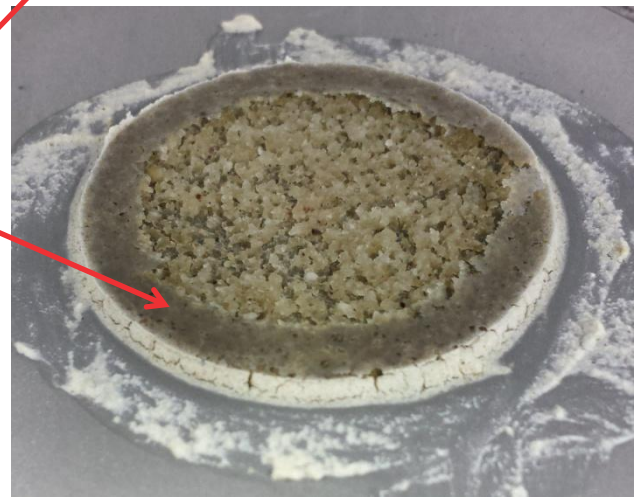
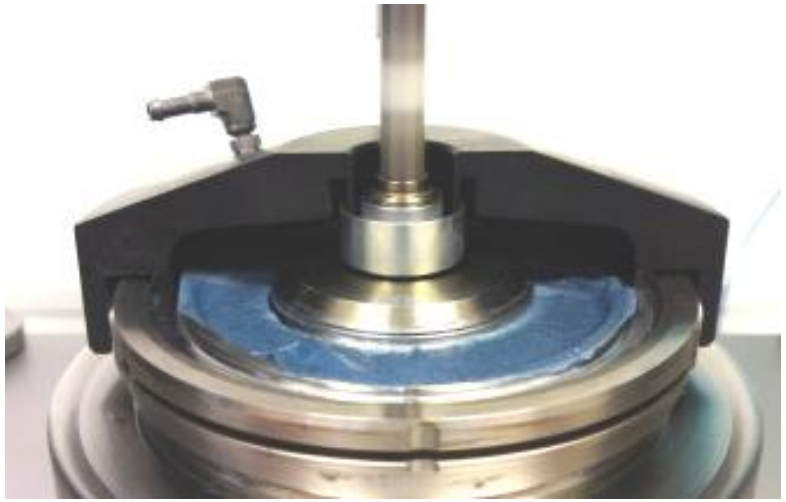
Drying Effects

- The majority of foods contains water
- Evaporation can occur during:
  - Long time measurements
  - Tests at higher temperatures
- Evaporation causes
  - Underfilled geometry
  - Film formation
  - Lost contact to part of the geometry
- Countermeasures:
  - Use solvent trap
  - Cover sample with low viscosity oil
  - Use CC-geometry (plus cover with oil)



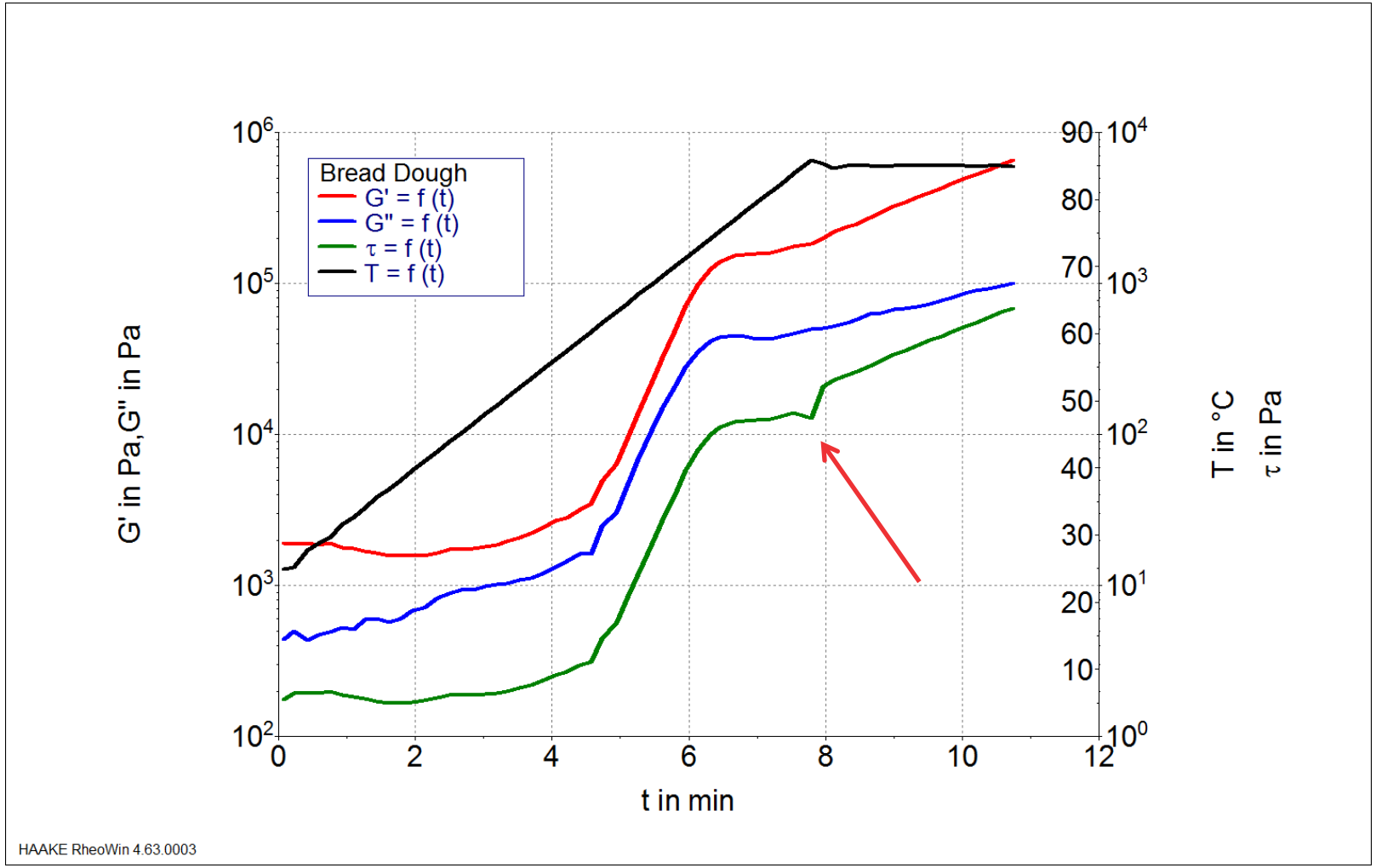




Bread dough with dried rim

Indication of Drying Sample

- Rheological data looks unsuspicious
- Stress curve reveals reduced contact with upper geometry
- Sometimes F_N can also indicate drying/shrinking



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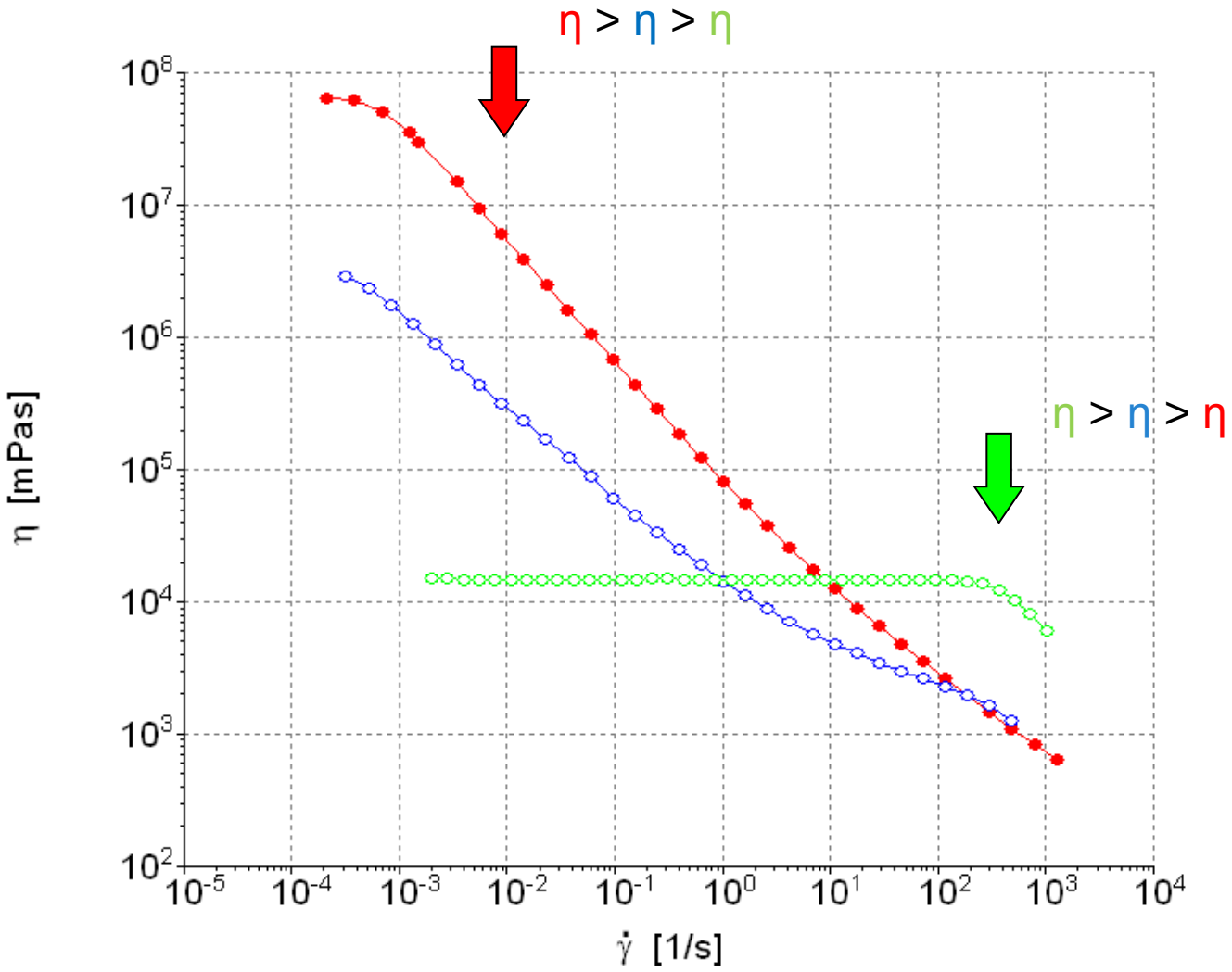
THE MEASURING METHOD

Viscosity

- Most frequently measured rheological parameter
- Can be measured with a variety of methods
  - Capillaries
  - Cups
  - Falling ball viscometer
  - Rotational viscometer
- Many historical methods are used
  - For simplicity
  - To be able to compare results
  - Due to loss of knowledge

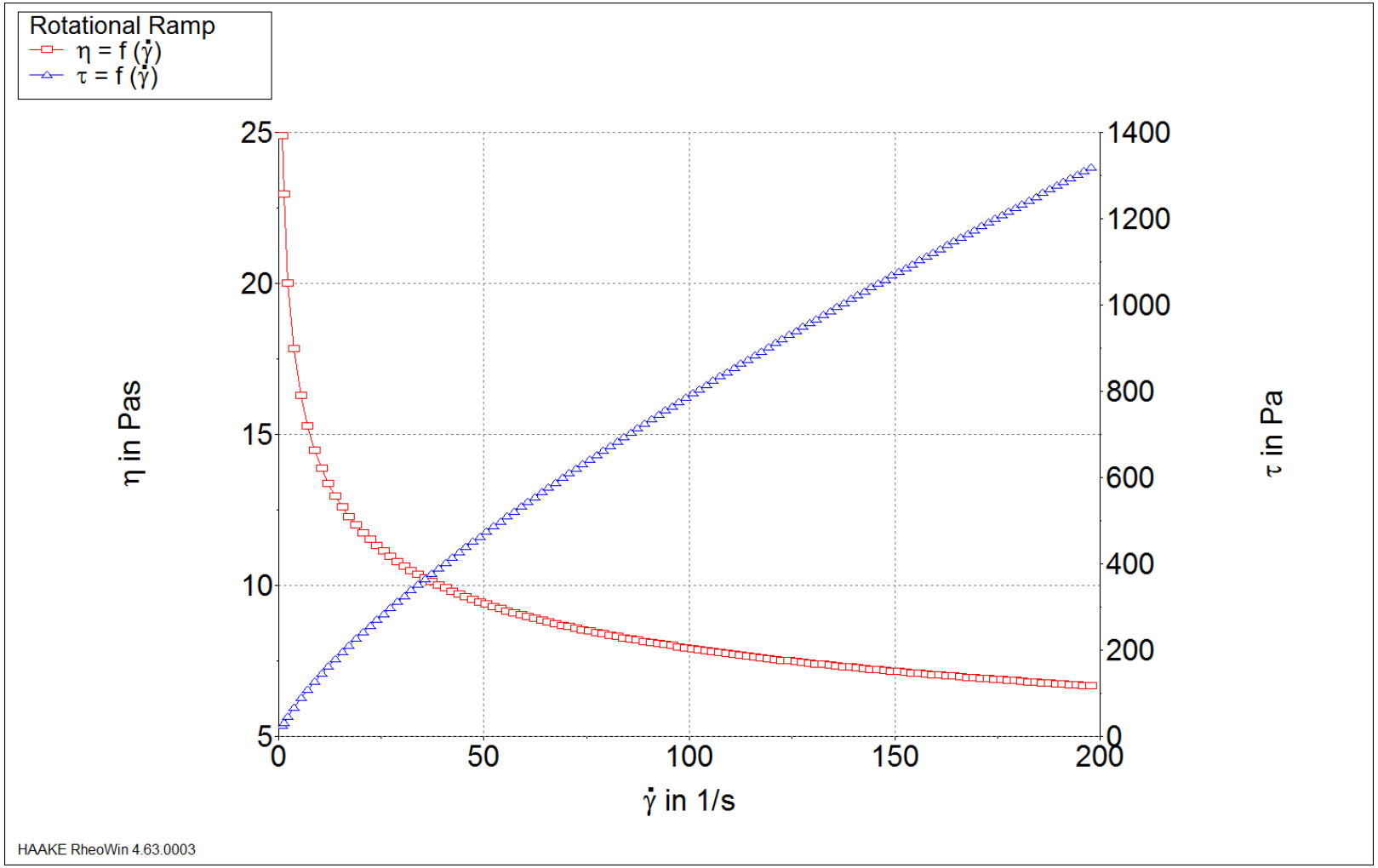
Viscosity: The Single Point Measurement

- Popular test because it is fast → QC
  - Often done with constant shear rate
  - Shear rate should relate to application
  - Wait until values stays constant over time
  - Values are taken from plateau
- It‘s an absolute viscosity (if done like described above)
  - Thermal equilibrium
  - Constant laminar flow
- But it does not show the shear rate dependence
- Always measure viscosity over a suitable shear rate range



The Rotational Ramp

- Commonly used
- Fast, usually 1 – 2 min
- Reproducible
- QC method „fingerprint“
- Transient measurement
- Gives no absolute viscosity
- Relative test
- Only comparable using the same ramp



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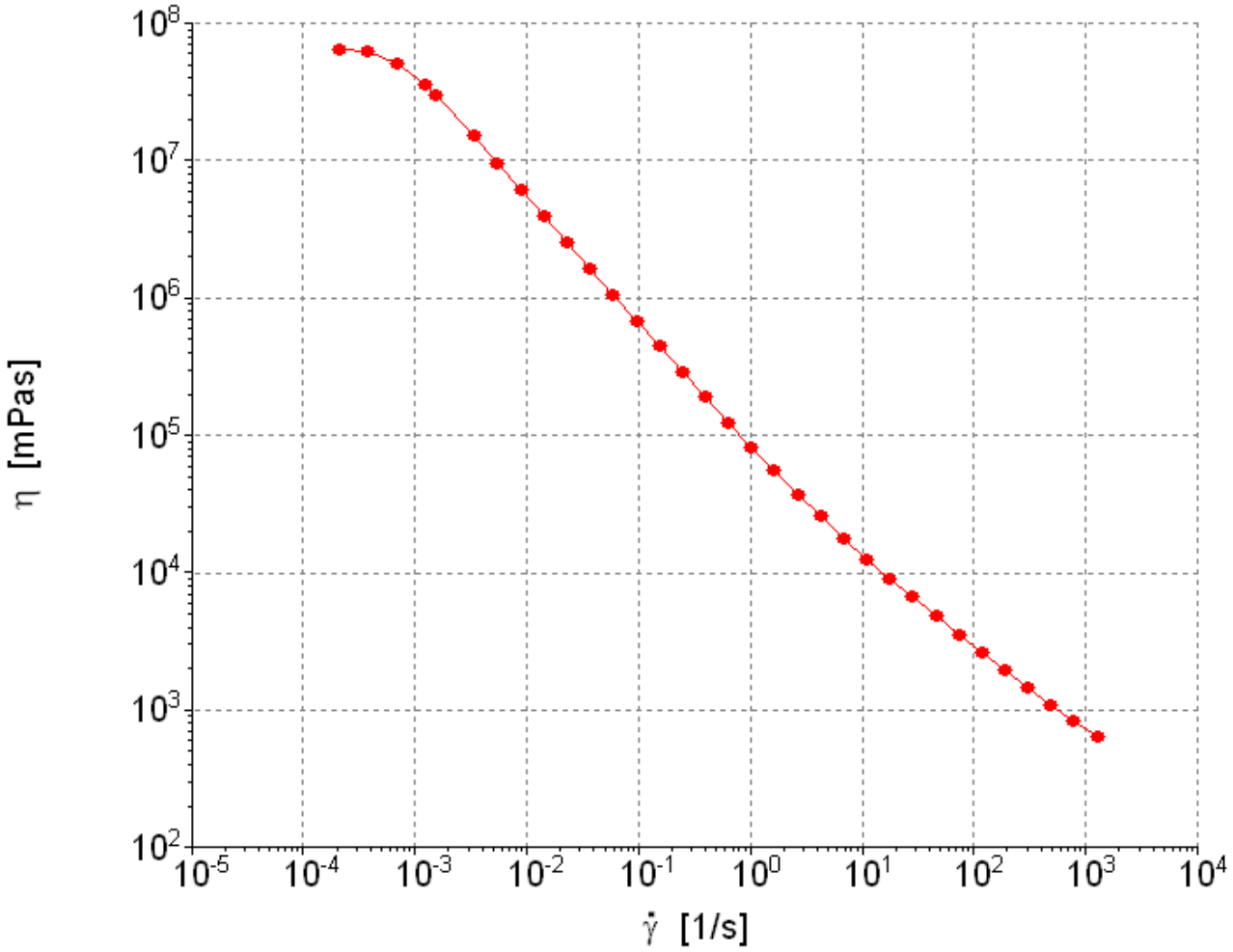
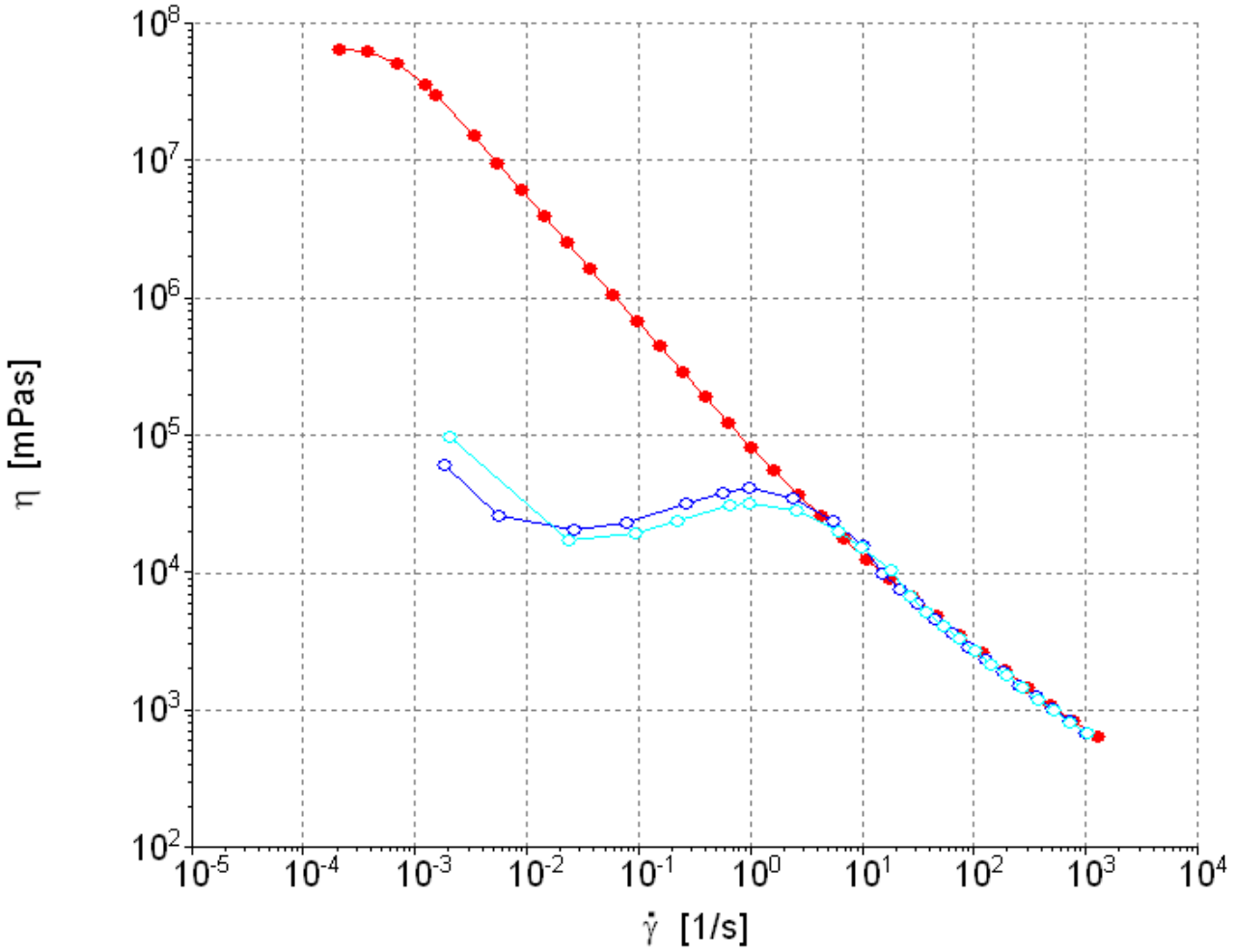
The Real Viscosity Curve

- This viscosity curve looks good



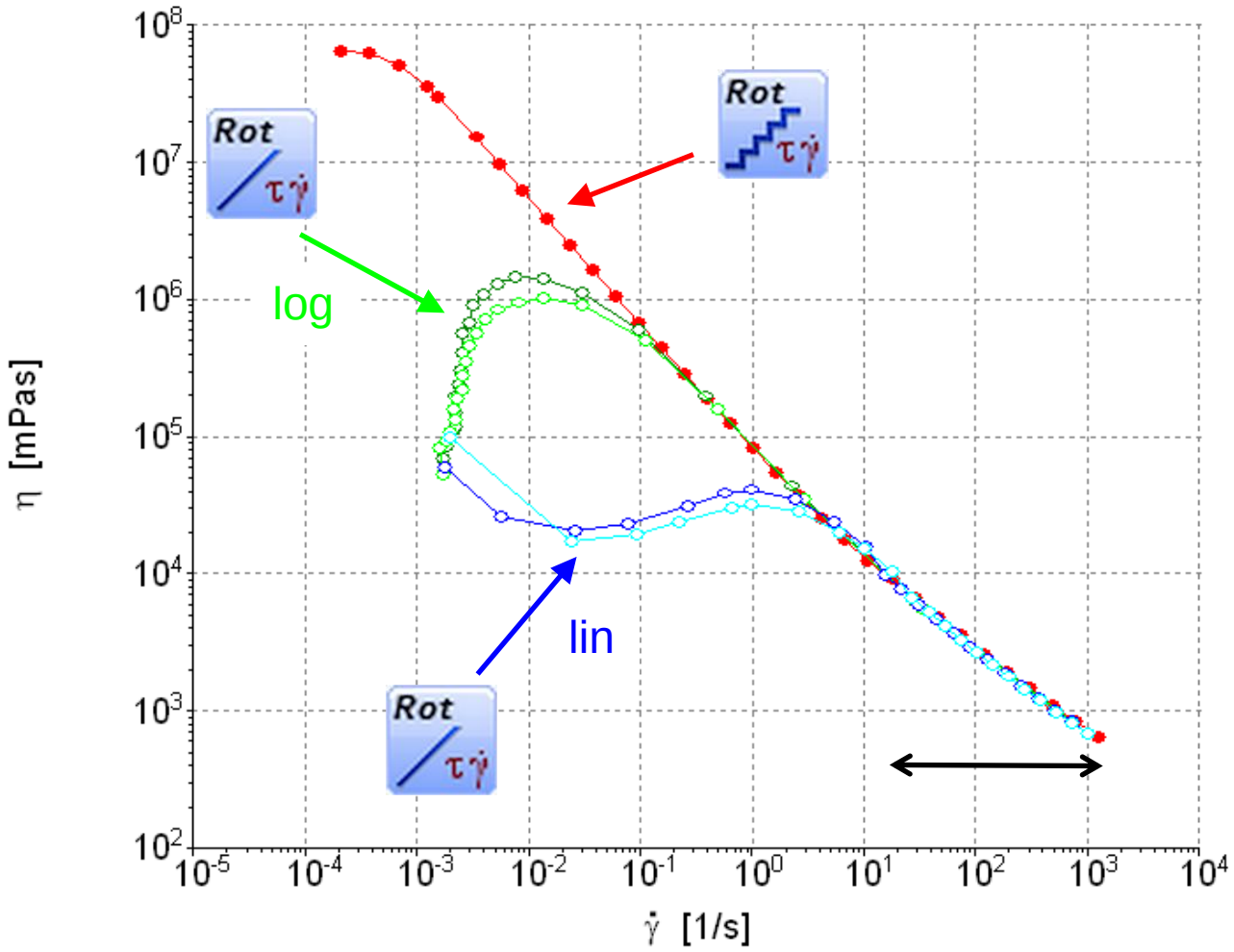
The Real Viscosity Curve

- This viscosity curve looks good
- But this one comes from the same product



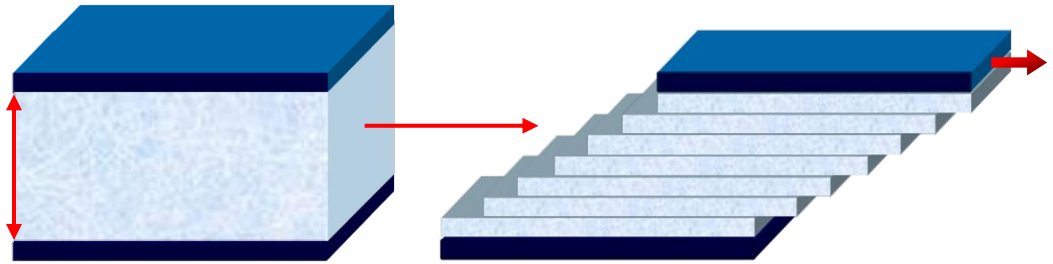
The Real Viscosity Curve

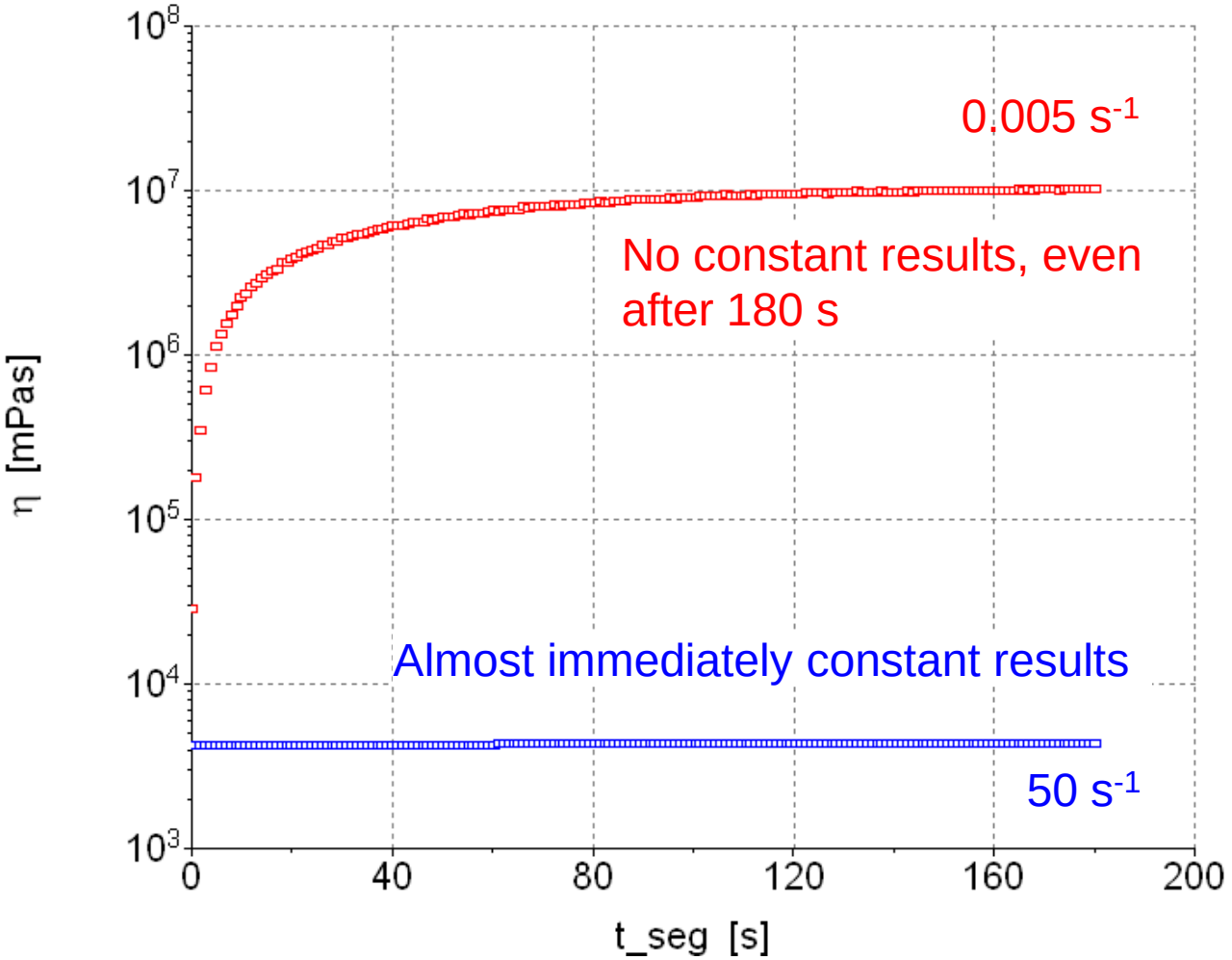
- This viscosity curve looks good
- But this one comes from the same product
- And this one as well
- The difference lies in the method used
  - Step wise increase of shear rate
  - Linear shear rate ramp
  - Logarithmic shear rate ramp
- Big differences at low shear rates
- Identical results at high shear rates



Shear Rate Dependent Equilibrium

- Instrument instantaneously at right speed
- Time to establish flow profile depends on
  - Sample viscosity
  - Sample elasticity
  - Shear rate





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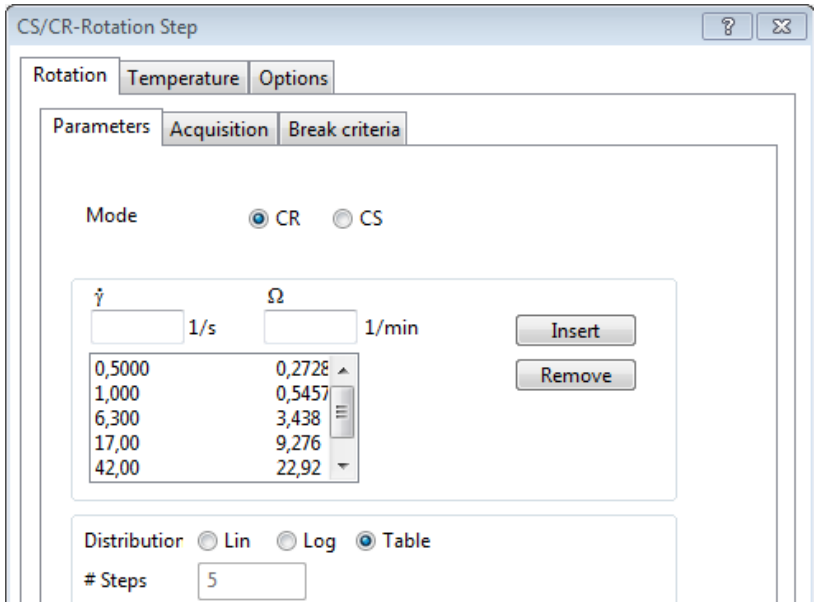
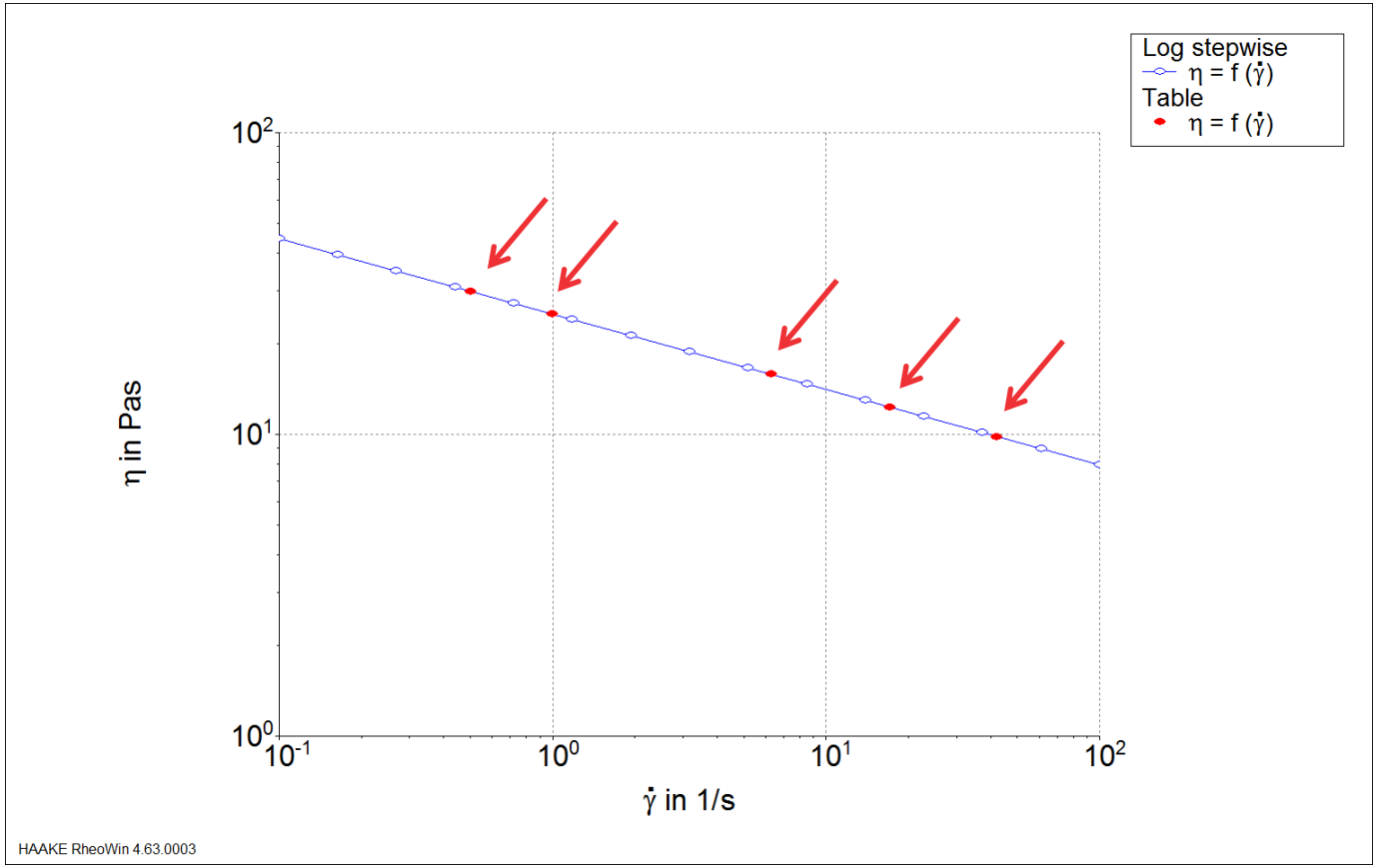
The Time Efficient Viscosity Curve

- Mayor criticism against shear rate step: it takes more time than a shear rate ramp
- This can be true, depending on
  - Shear rate range
  - Number of data points (→ interpolation)
  - Sample‘s properties





Using a shear rate table saves time but still gives more info!

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Rheology Beyond Viscosity

- Viscosity is needed to quantify
  - Pumping (production)
  - Dosing (e.g. syrup from a dispenser)
  - Visual impression (sauces, syrups, molten cheese…)
- It is not sufficient to quantify
  - Long time stability (salad dressing)
  - Gel stability (pudding)
  - Mouth feeling (sausage-meat)
  - …

Oscillatory Measurements – A possible Alternative

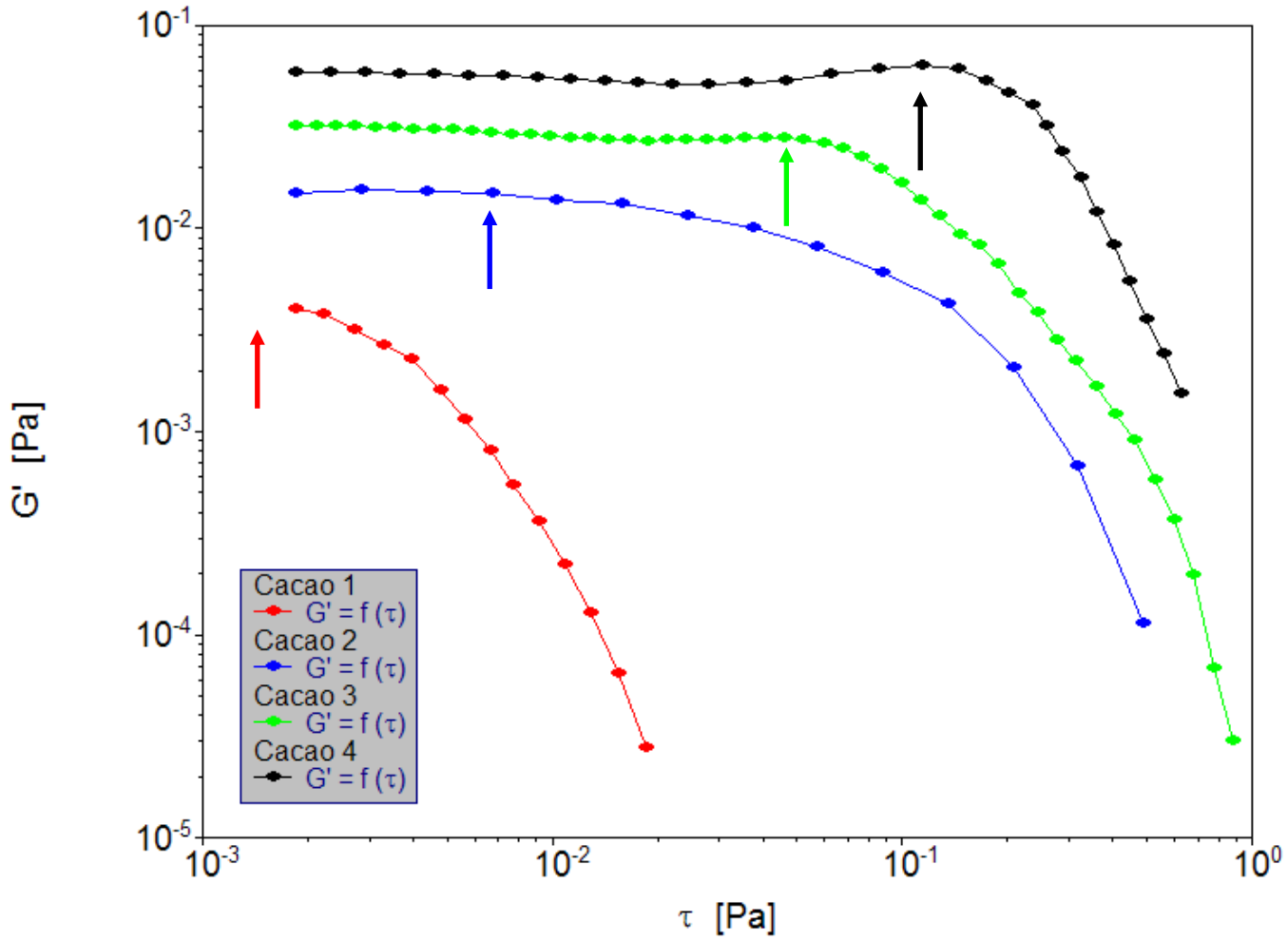
- When sample comes out of the gap due to elastic effects
- When geometry loses contact with sample
  - CC geometry in peanut butter
  - CC geometry in mayonnaise
- When you are interested in the sample‘s structure
  - Emulsion
  - Gel

This is what rotation sees

Oscillation sees the undamaged structure

Oscillatory Measurements – Stability of a Structure

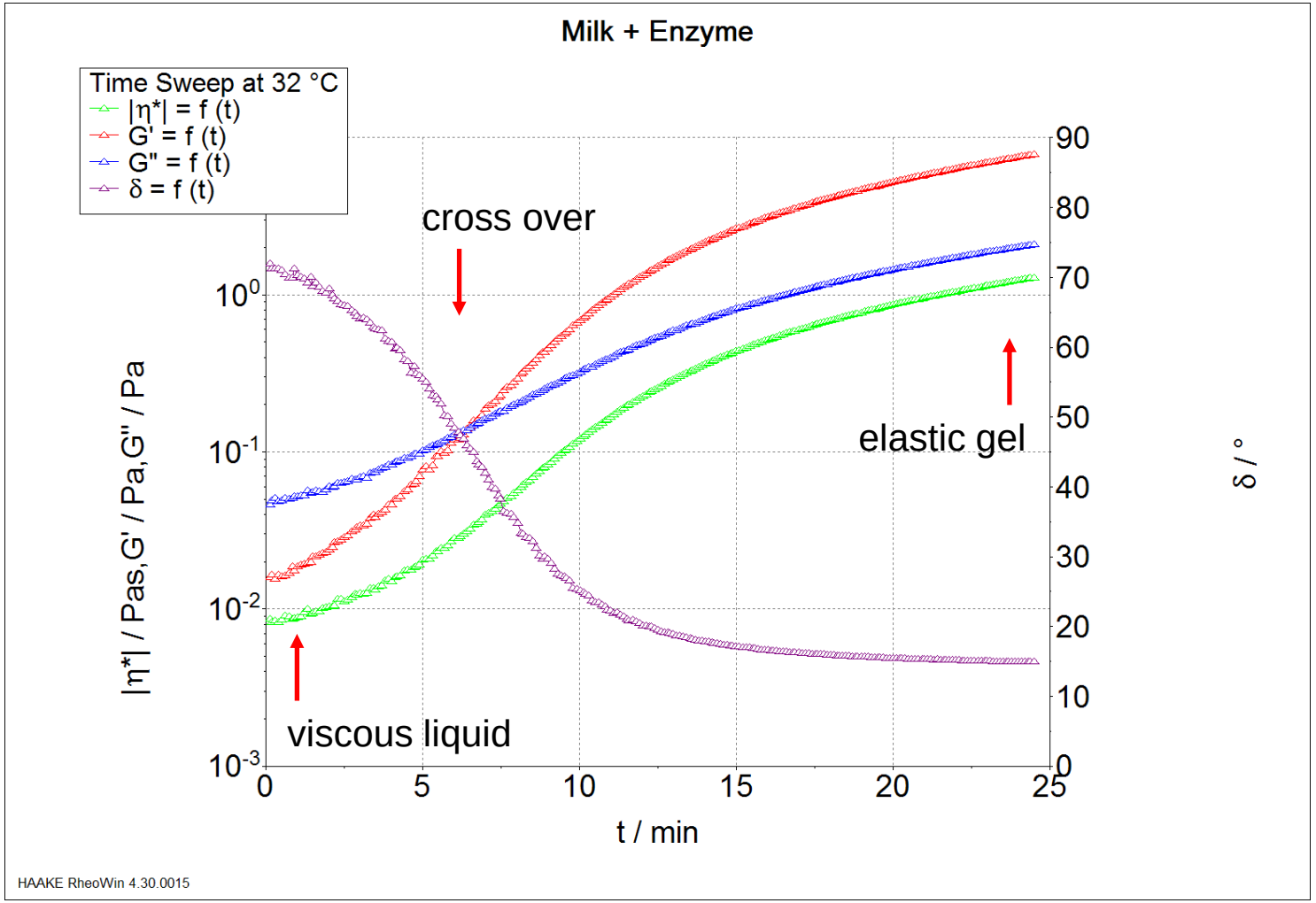
- Stability against sedimentation
- Stability of a foam
- Stability of an emulsion
- Related to yield stress



Different chocolate milks with different stabilizers

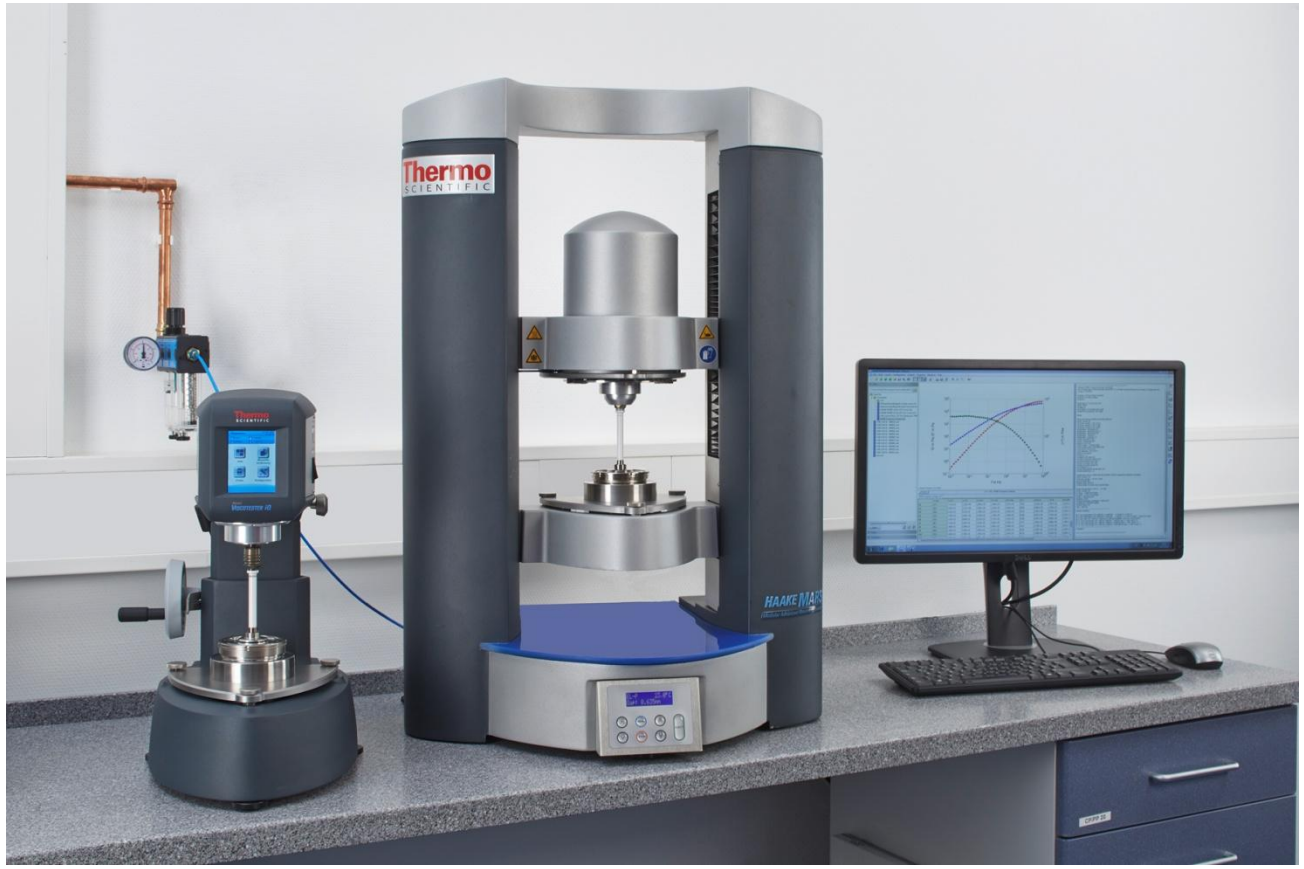
Oscillatory Measurements – Development of a Structure

- Gelling of protein
- Baking of dough
- Crystallization of fat
- Oscillation
  - does not affect the development of the structure
  - Yields valuable information about the structure



Summary

- Food samples can be tricky to handle
- But reliable results are easily achived when
  - The sample is handled carefully
  - The right geometry is used
  - The measuring methods fits to the sample
- There is no universal test method!
- The measuring method should be
  - More than one dot
  - But still time efficient
  - Sometimes an oscillatory measurement



Thank you for your attention!
Please let me have your questions now!