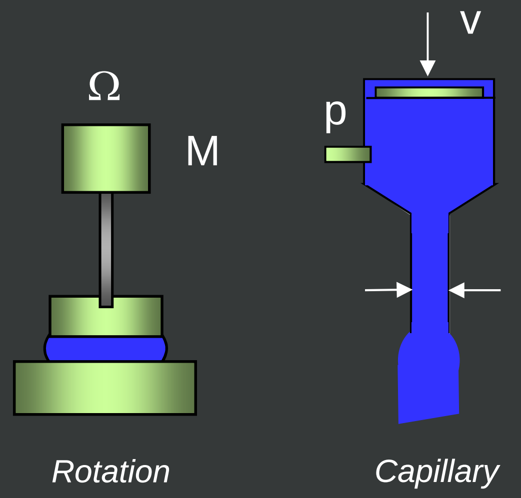
How to measure the shear viscosity properly?

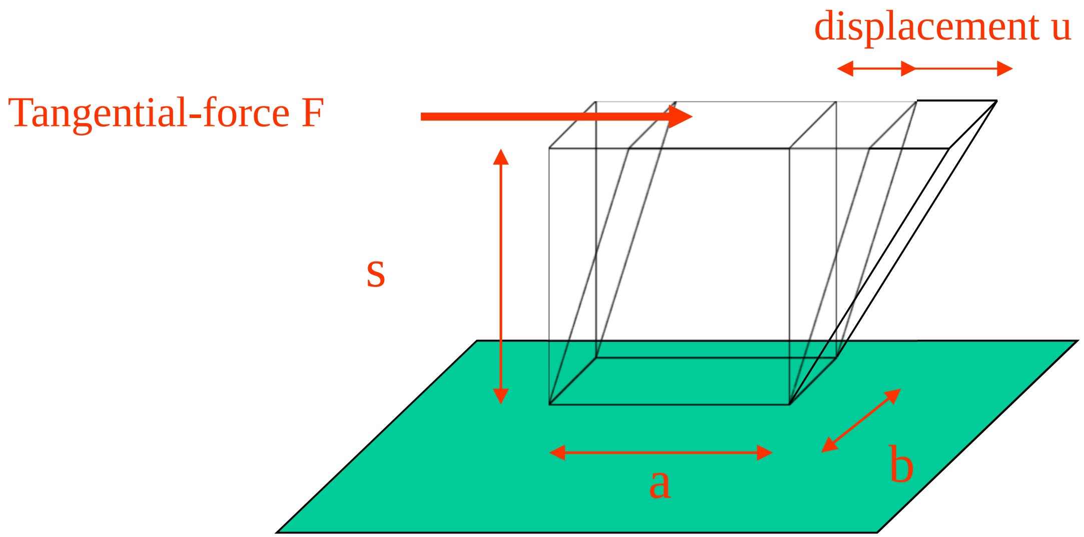


Torsten Remmler, Malvern Instruments

Outline

- How is the Shear Viscosity defined?
- Principle of Operation: Rotational and Capillary Rheometer
- Choice of the Correct Geometry
- Steady State Condition
- Example for Steady State Shear Viscosity Curve

Basic Terms in Shear Rheometry



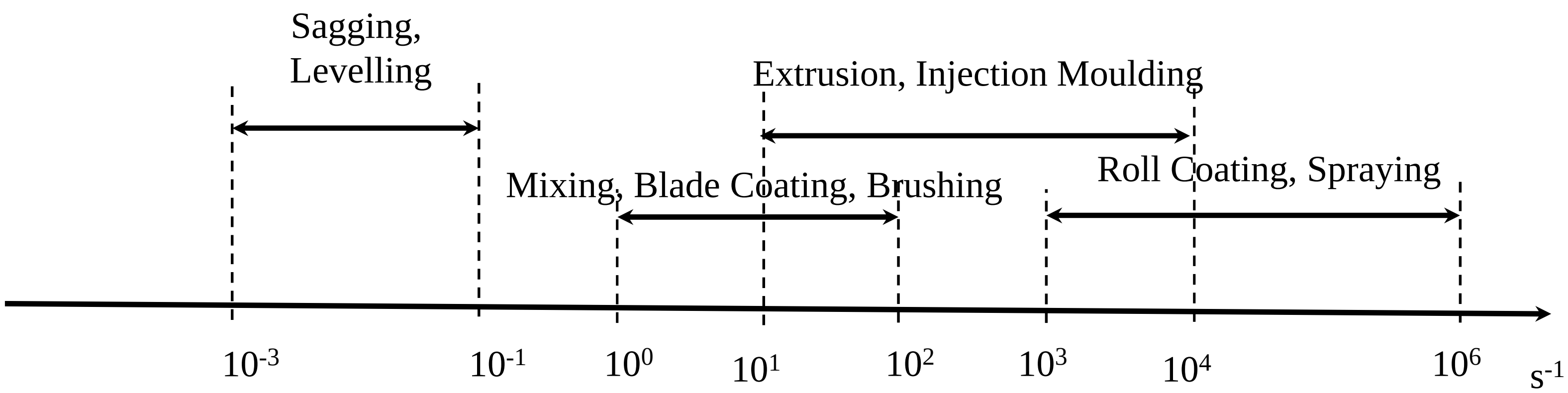
area = a · b
Gap = s

$$\gamma = \frac{u}{s}$$ strain []

$$\dot{\gamma} = \frac{d\gamma}{dt}$$ Shear rate [1/s]

$$\sigma = \frac{F_{\tan}}{A}$$ Shear stress [Pa=N/m²]

Typical Shear Rate Ranges



Rotational-Rheometer

Sample: Water up to solids
Results: Shear-Viscosity, Yield Stesses, Visco-Elasticity, Relaxation...

High Pressure Capillary-Rheometer

Sample: Water up to high viscous
Results: Shear-Viscosity, Elongational-Viscosity, Wall Slip...

Shear Viscosity

Resistance of a sample against the flow

$$\eta = \frac{\sigma}{\dot{\gamma}}$$

η – Shear Viscosity

σ – Shear Stress

γ – Shear Rate

Typical Shear Viscosities

Material	Shear-Viscosity (Pas)
Air	10^{-6}
Aceton	10^{-4}
Water	10^{-3}
Olive Oil	10^{-1}
Glycerol	10^{0}
Molten Polymers	10^{3}
Bitumen	10^{8}
Glass at 500°C	10^{12}
Glass at ambient	10^{40}

Units:		Remember
Pascal second	Pas (SI)	1 Pas = 10 P
Poise	P (CGS)	1 mPas = 1 cP

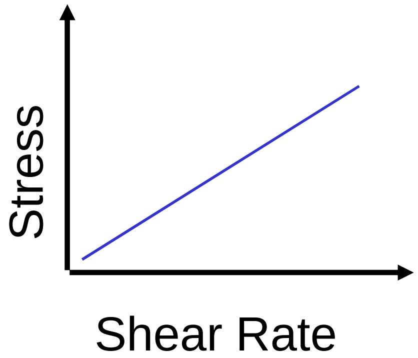
Shear-Viscosity depends on…

$$\eta\,(T, p, t, \dot{\gamma}) = \frac{\sigma}{\dot{\gamma}}$$

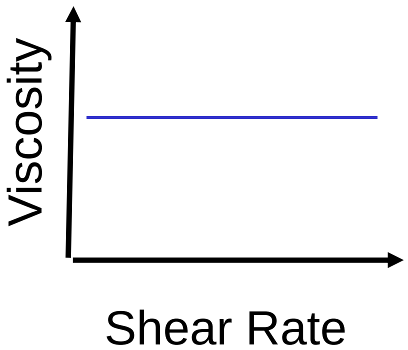
- Physical-chemical structure of the sample
- Temperature (up to 20% / K)
- Pressure
- Time
- Shear Rate

Steady-State Flow Behaviour

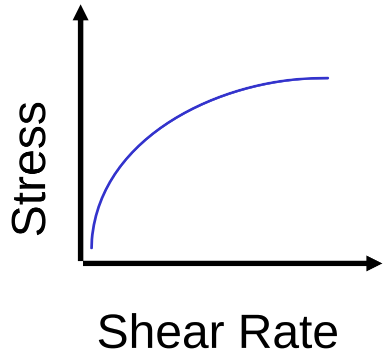
Newtonian



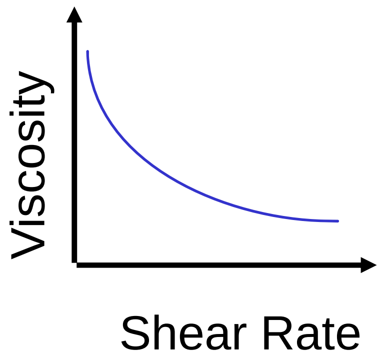
Silicon Oil, Suspension



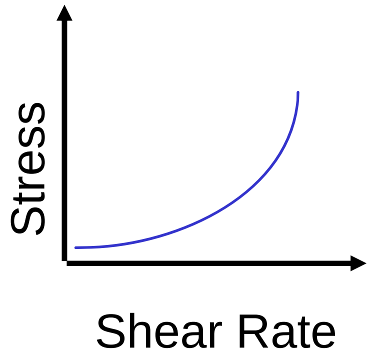
Shear Thinning



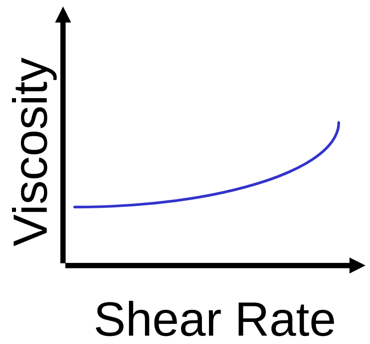
Inks, Paints



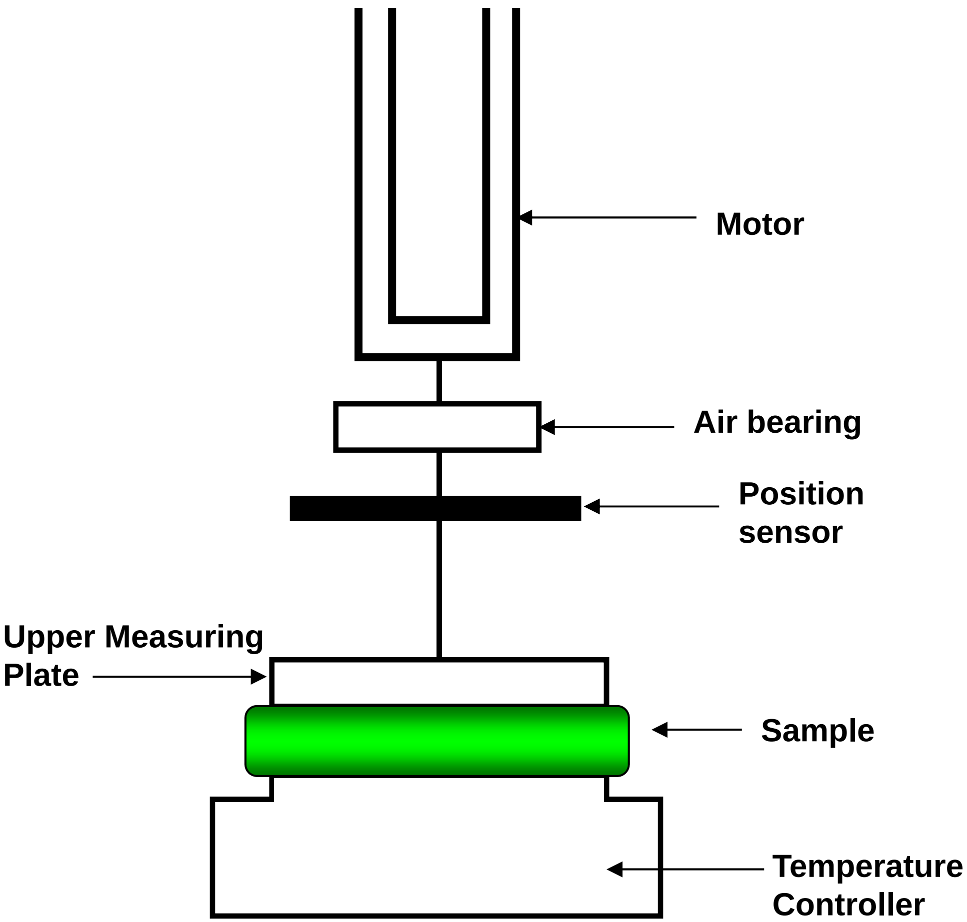
Shear Thickening



Cornflower



Principle of Operation: Rotational Rheometer



Stress- and Strain Control possible.

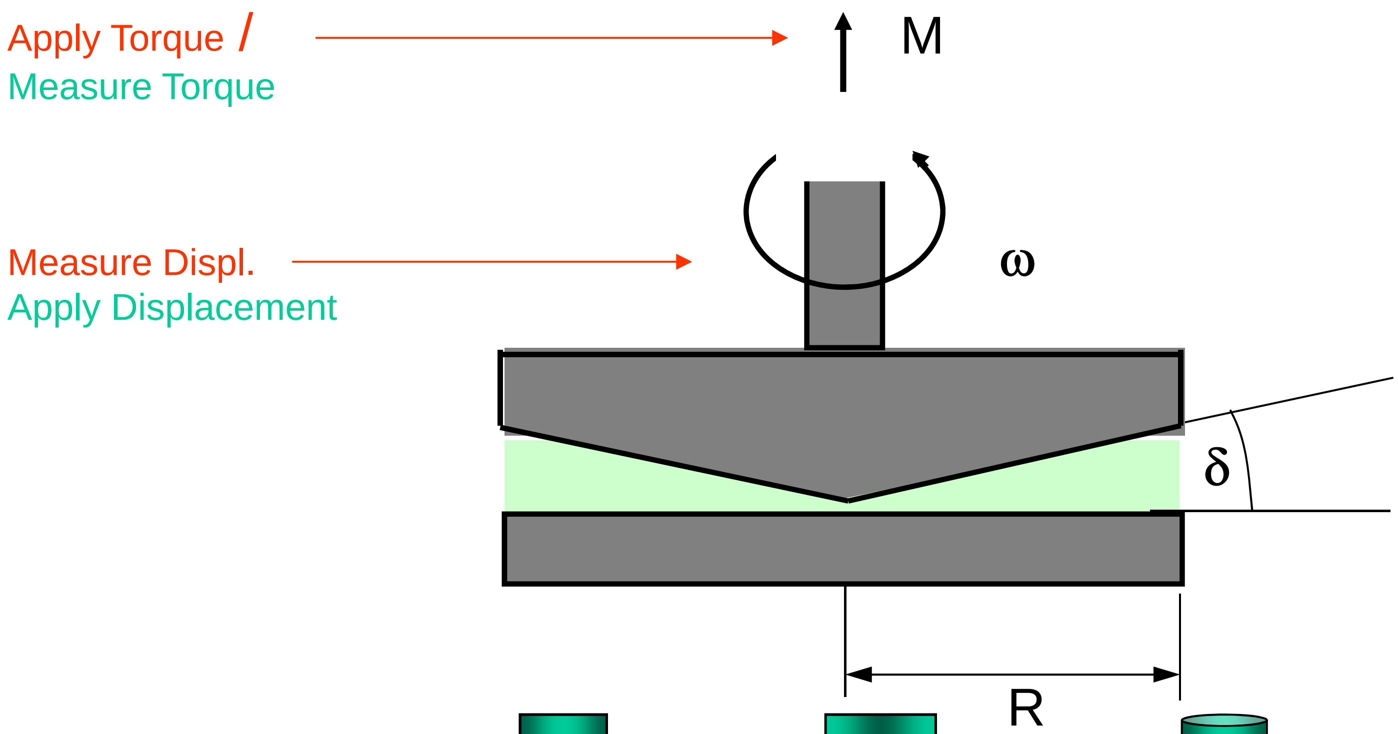
kinexus

- The drive is situated above the sample, not below.
- The driven spindle is air bearing supported so torque can be measured.
- The separate torque transducer is eliminated!

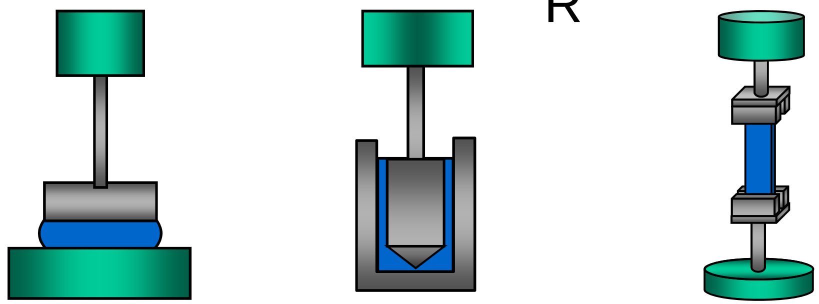
Advantages:

- Wide Torque Range 10e-9 to 10e-1 Nm
- Short Response times
- Small inertia design
- Direct Stress and Direct Strain

Choice of Geometry: Rotational Rheometry



Rule of Thumb
for dispersions:
*Gap Size > 10 * D90*

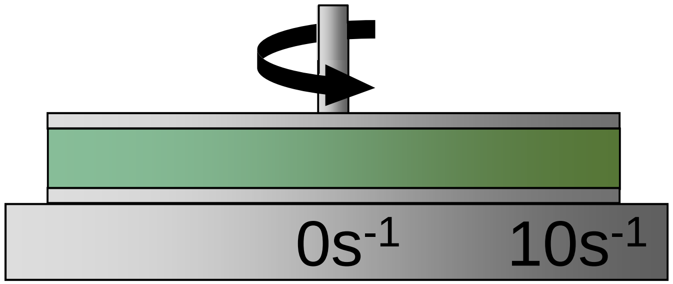


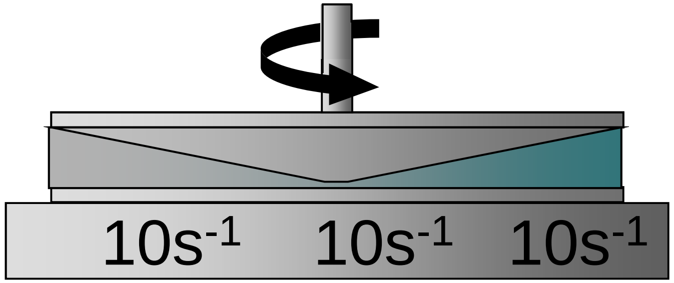
Parallel Plates **Cup&Bob** **Solids Fixture**

- the higher the viscosity, the smaller the geometry
- the higher the shear rate, the smaller the gap.

Cone-Plate / Plate-Plate

- ***Cone Adv: Const Shear Rate along the complete gap, easy cleaning, low sample volume, wide viscosity range***
- ***Cone DisAdv: only for homogeneous samples, for disperse samples D90 < 10 x gap, solvent evaporation***
- ***Plate Adv: flexible gap, auto-tension possible, low sample volume, often used for temperature dependent tests, good for disperse systems***
- ***Plate DisAdv: shear rate dependency, solvent evaporation***





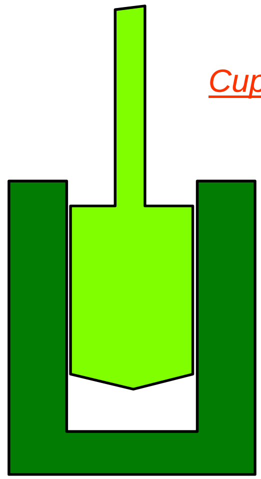
Cup & Bob / Double Gap

- ***Cup&Bob Adv: large gap, works well for disperse systems, also for samples showing sedimentation, large surface area, nearly no evaporation effects, good for low viscous samples, less impact of loading errors***

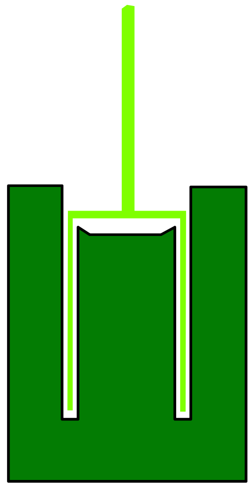
- ***Cup&Bob DisAdv: high moment of inertia limits oscillation and transient steps, high cleaning effort, large sample volumes (ca 2ml – 15ml)***

- ***Double Gap Adv: highest sensitivity for low viscous samples, lower inertia compared to cup&bob, nearly no impact on loading errors***

- ***Double Gap DisAdv: large sample volume (ca. 15ml – 30ml), difficult cleaning***



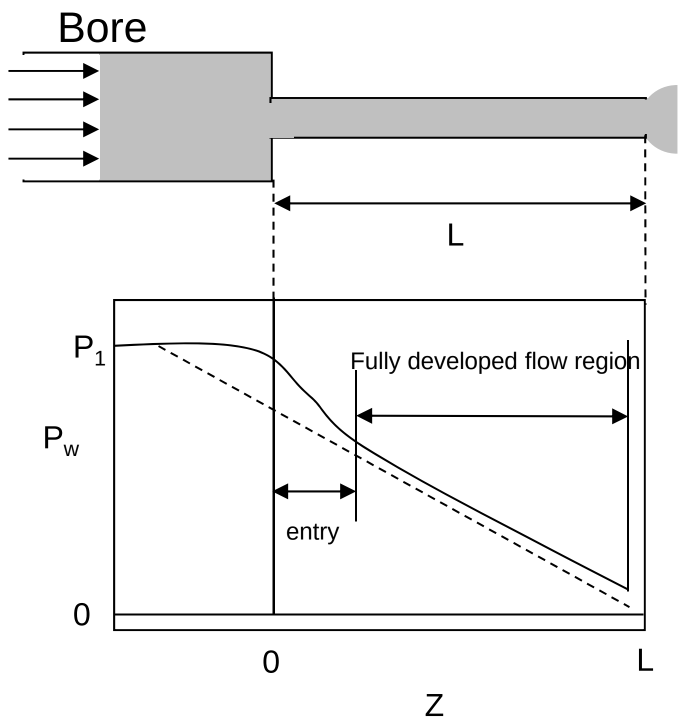
Cup&Bob acc DIN53019

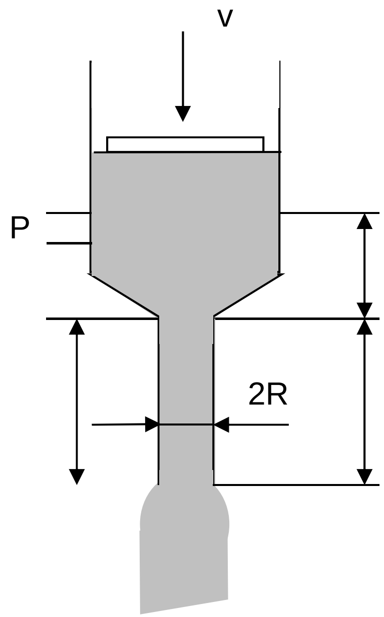


Double Gap

Principle of Operation: Capillary Rheometer

Given quantity:	piston speed	$\Rightarrow$	wall shear rate
Measured quantity:	pressure drop	$\Rightarrow$	wall shear stress





Full pressure drop

=

Entrance pressure drop

+

Shear pressure drop

RH2000

Laminar Pipe Flow

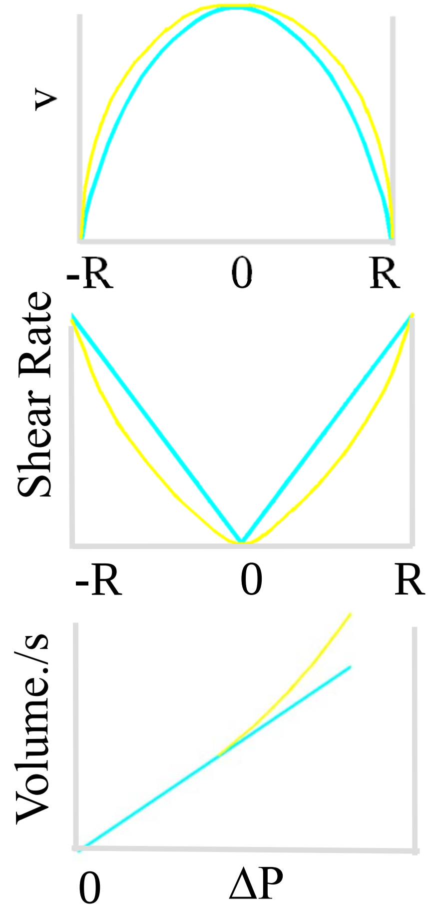
Isothermal, stationary Flow of an incompressible fluid

Newtonian

$$\dot{\gamma}_{app} = \frac{4 \cdot Q}{\pi R^3}$$

$$\sigma_{app} = \frac{R \cdot \Delta P}{2 \cdot L}$$

Q = Volume Flux, R= Die Radius,
L= Die Length, ΔP=Pressure Drop



Newtonian

Non-Newtonian

Choice of Geometry: Capillary Rheometer

$$\dot{\gamma}_{app} = \frac{4 \times Q}{\pi R^3}$$

$$\sigma_{app} = \frac{R \times \Delta P}{2 \times L}$$

Shear Rate Ranges:

2.0mm = ca. 0.1 bis 100 /s
1.5mm = ca. 1 bis 1000 /s
1.0mm = ca. 10 bis 10000 /s
0.5mm = ca. 100 bis 100.000 /s
0.3mm = ca. 1000 bis 1.000.000 /s

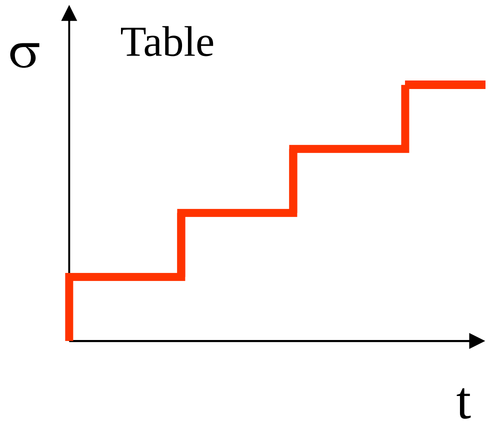
$\Rightarrow$ 2-3 decades of shear rate can be achieved with a capillary die

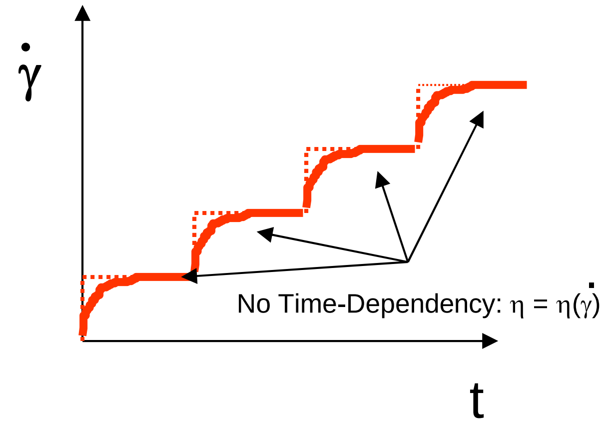
Q = Volume Flux, R= Die Radius, L= Die Length, ΔP=Pressure Drop

Basic Viscometry: How to run a flow curve

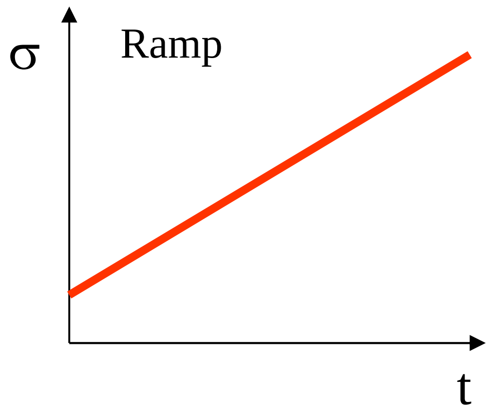
CS-Mode: Steady state and non-steady state measurements

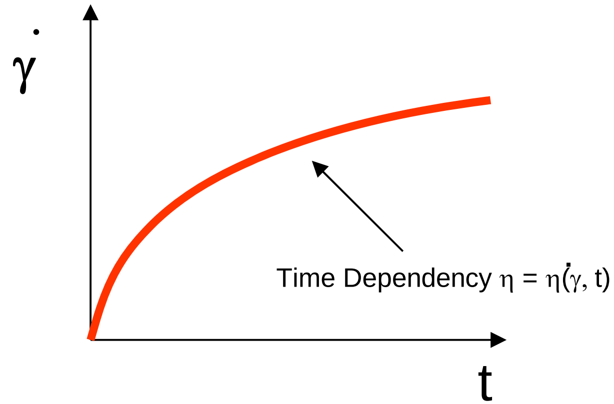
<u>Steady state:</u>





<u>non-steady state:</u>





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Steady State Flow Properties

Newton: $$\eta = \frac{\sigma}{\dot{\gamma}}$$

Flow Curve: $\sigma = \sigma(\dot{\gamma})$ ⟵ equivalent ⟶ $\dot{\gamma} = \dot{\gamma}(\sigma)$

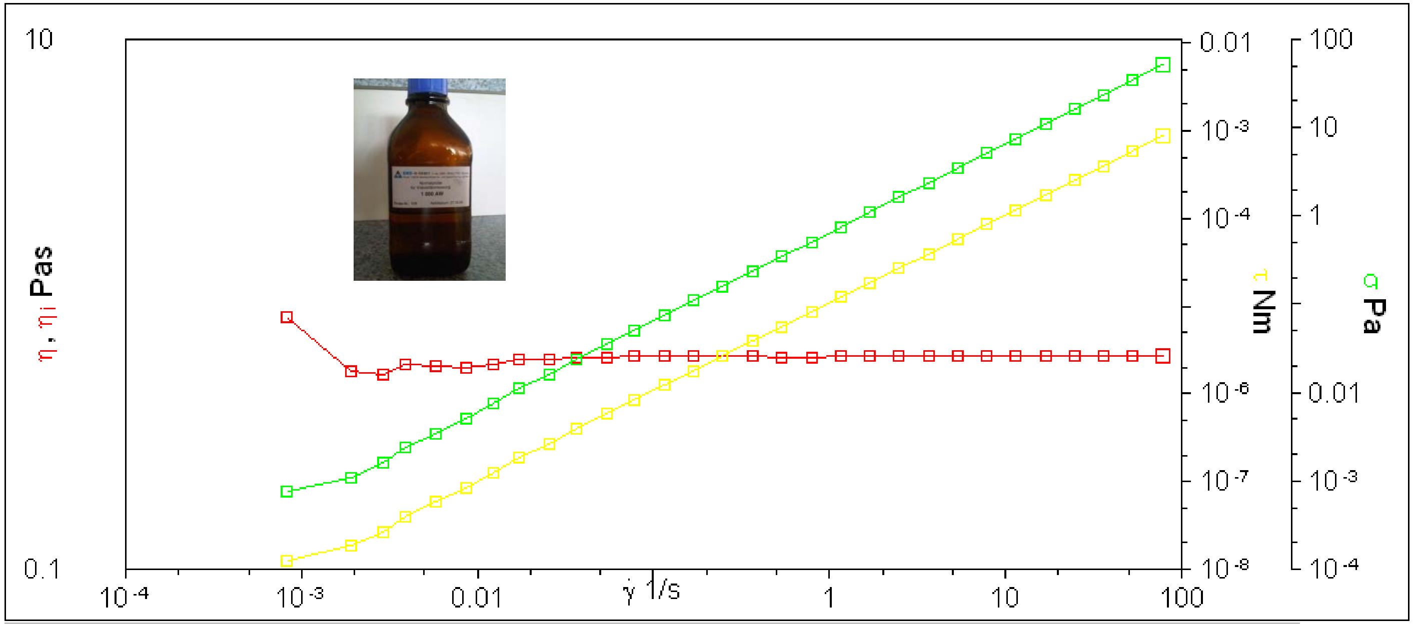
CR-Mode | CS-Mode

Shear Viscosity Curve: $\eta = \eta(\dot{\gamma})$ ⟵ equivalent ⟶ $\eta = \eta(\sigma)$

Steady State Condition: Rotational Rheometry

Kinexus Rheometer

$$J = \frac{\gamma}{\sigma}$$

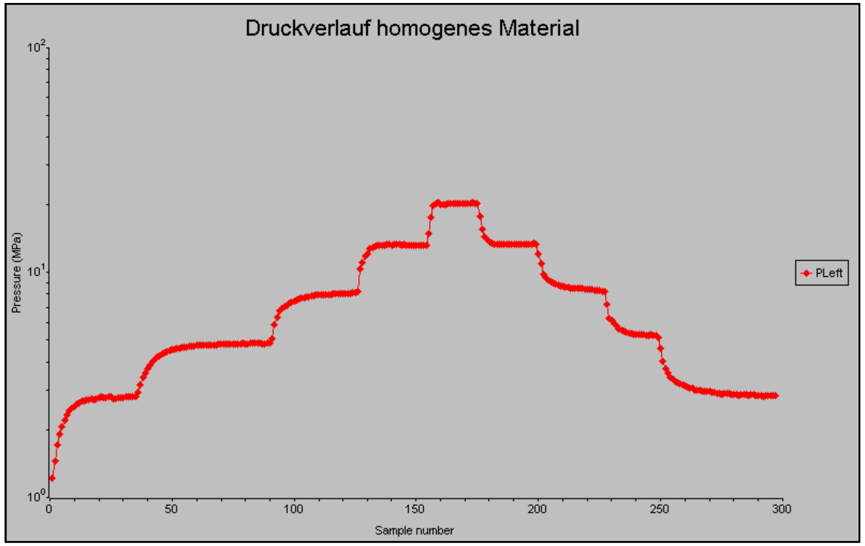


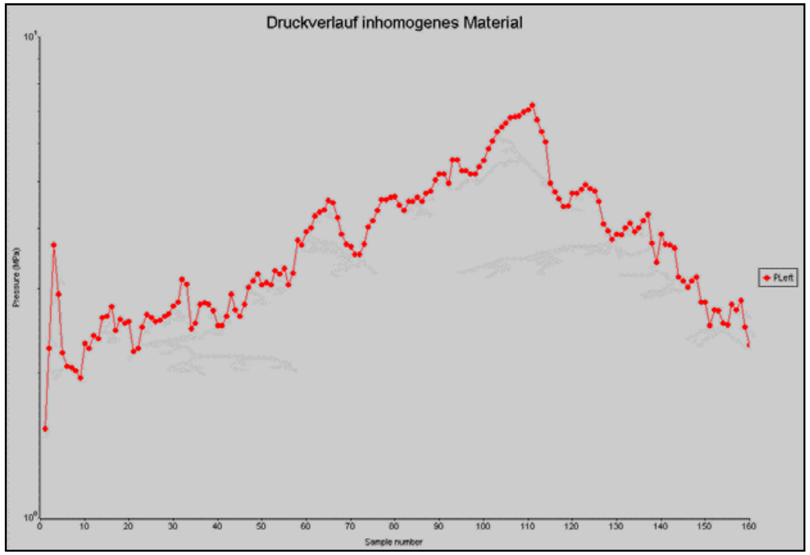
	t s	T °C	σ Pa	$\dot{\gamma}$ 1/s	η Pas	dLn J/dLn t	Hinweis
1, 27	406.366	25.0	2.321E-3	3.927E-3	0.59101	1.005	
1, 28	421.430	25.0	1.581E-3	2.937E-3	0.53832	0.988	8
1, 29	436.494	25.0	1.077E-3	1.936E-3	0.5563	0.960	8
1, 30	451.572	25.0	7.339E-4	8.261E-4	0.88841	0.899	8

⇒ dLnJ/dLnt = 1 for pure viscous flow!

⇒ Deviations show measurement errors!

Steady State Condition: Capillary Rheometry



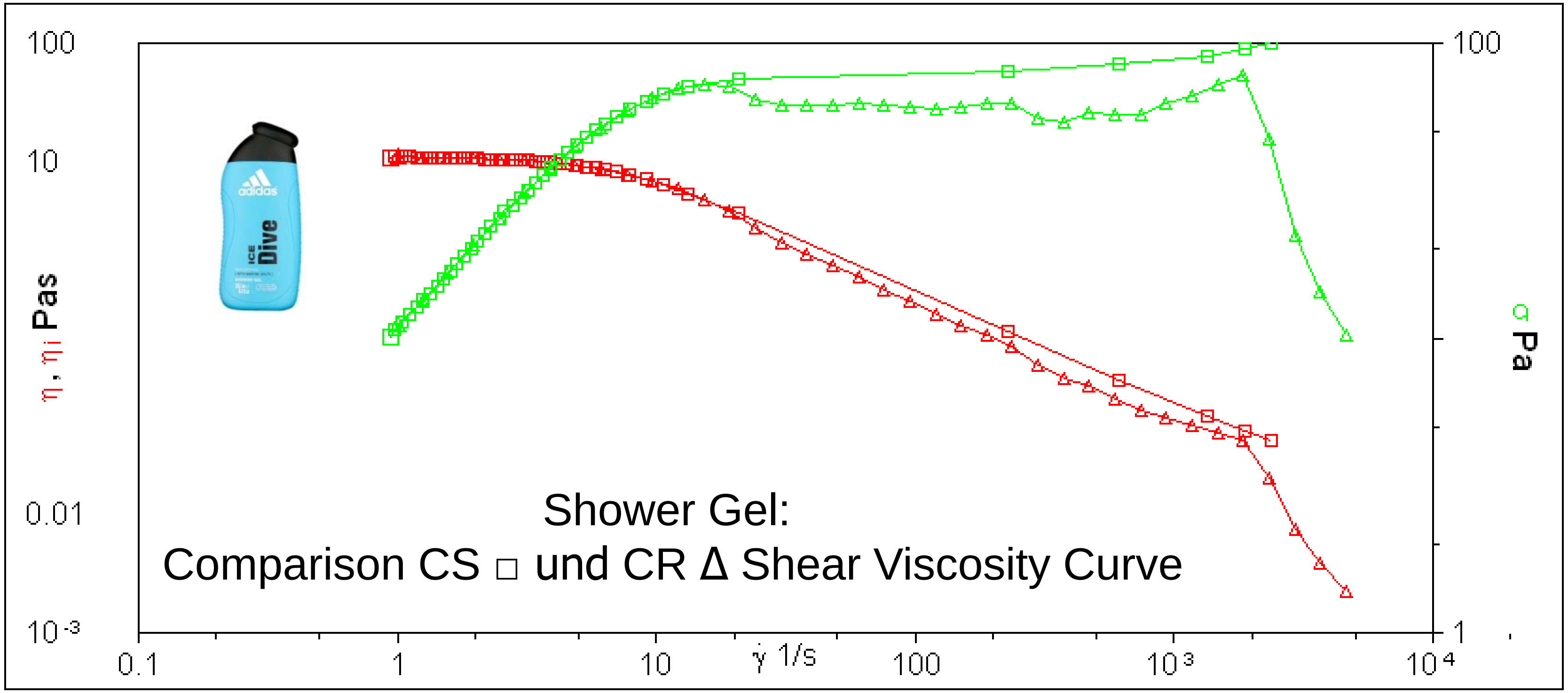


homogeneous

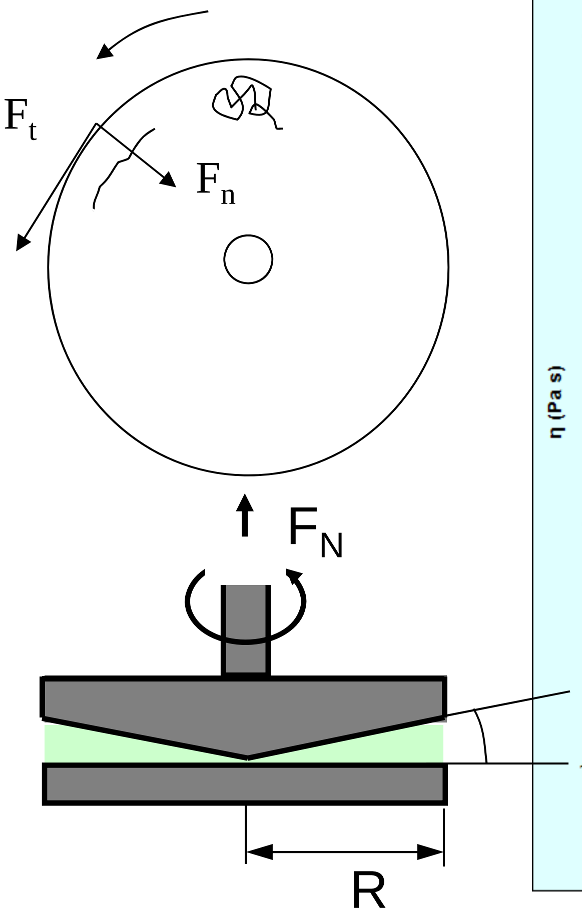
inhomogeneous

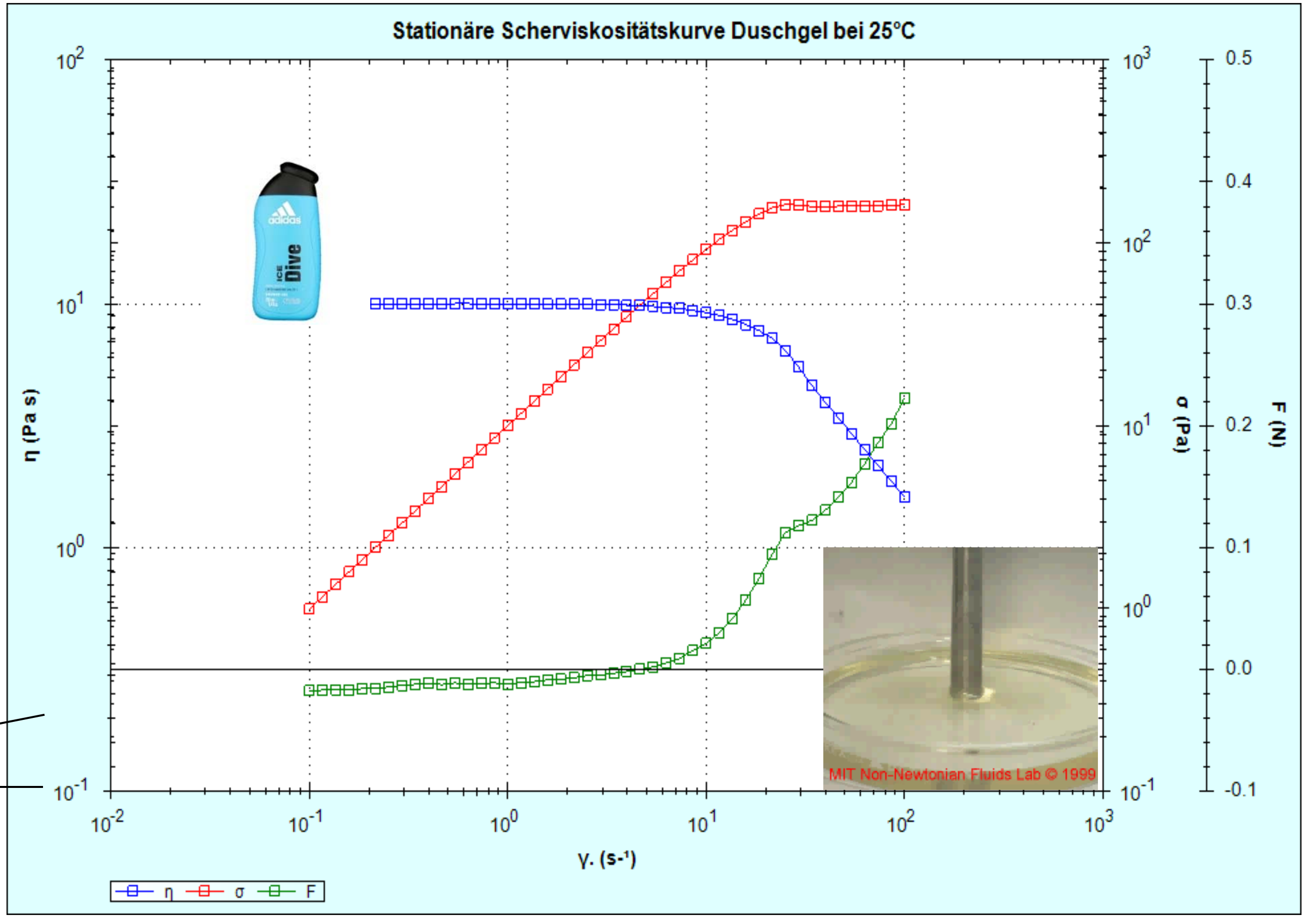
⇒ Equilibrium Pressure Drop is needed for Steady State Viscosity.

Comparison Stress- and Rate Controlled Test



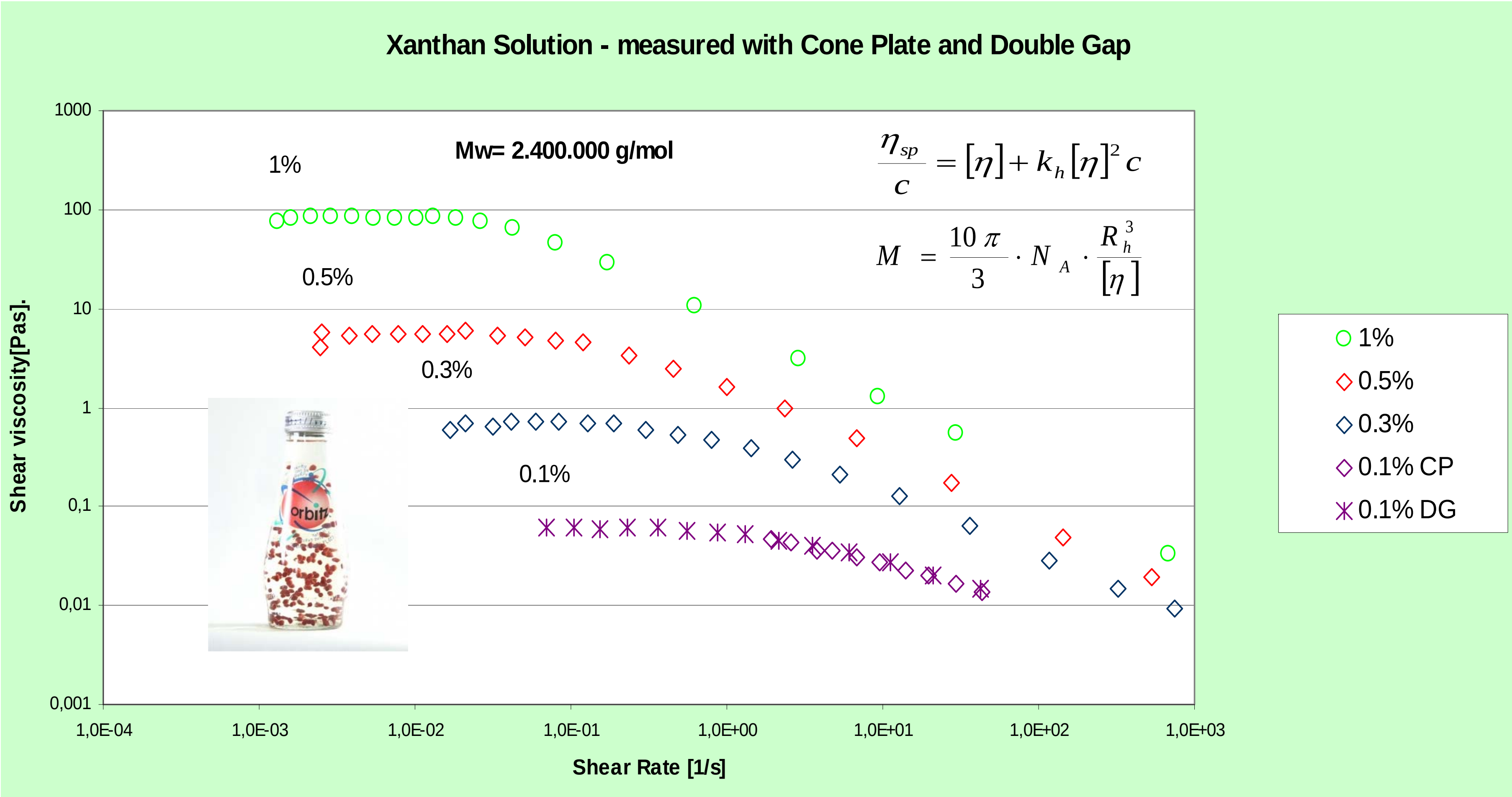
Normal Stress Difference N1





⇒ Always watch the Normal Stress during a Shear Viscosity Measurement!

Example: Steady State Viscosity Curve



Conclusion

- Correct Geometry Choice is key for Viscosity Measurement
- Steady State Condition in both Rotational and Capillary Rheometry
- Monitoring N1
- Interpretation: Correct Stress / Shear Rate Range for Shear Viscosity Curve

Thank you for your attention.

Email: torsten.remmler@malvern.com