CM4655 Polymer Rheology Lab

Shear Viscosity Measurement in a Capillary Rheometer

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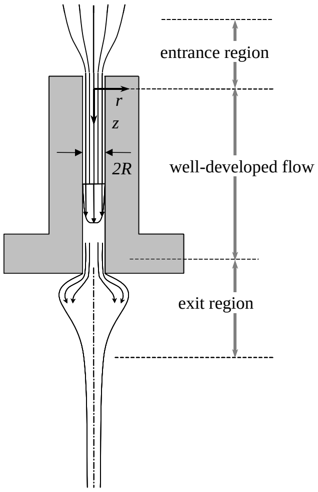


Shear Rheometry

Goal:

Measure the viscosity of a very viscous, non-newtonian fluid.

Difficulty:

Because we do not know the rheological behavior of our sample (whether it is newtonian, power-law, etc.) we do not know how it will behave. If we do not know how it will behave, it is difficult to design an experiment to measure its behavior (Catch 22).

Strategy:

Design experiments making the fewest assumptions possible so that the design is applicable to all (most) fluids.

Begin with: What is the definition of viscosity?

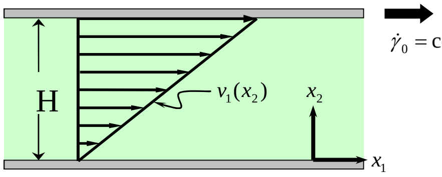
By definition, viscosity is measured in pure, homogeneous shear flow

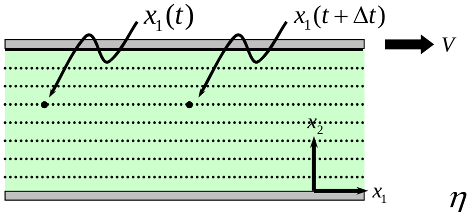
velocity field

force=F

$v_1(H) = V = \dot{\gamma}_0 H$

$\dot{\gamma}_0 = \text{constant}$





path lines

shear rate:

$$\dot{\gamma}_0 = \left| \frac{\partial v_1}{\partial x_2} \right|$$

$$\underline{v} \equiv \begin{pmatrix} \dot{\gamma}_0 x_2 \\ 0 \\ 0 \end{pmatrix}_{123}$$

$$\eta = \frac{\tau_{21}}{\dot{\gamma}_0} = \frac{F/A}{V/H}$$

Drawbacks

- Parallel plate flow is difficult to produce (maintaining constant gap for example)
- Stress measurements are affected by edge effects and spacers used to maintain gap
- Signal can be low
- Sample loading is inconvenient and complex

It has been done: J. M. Dealy and S. S. Soong J. Rheol. 28, 355 (1984); doi:10.1122/1.549756

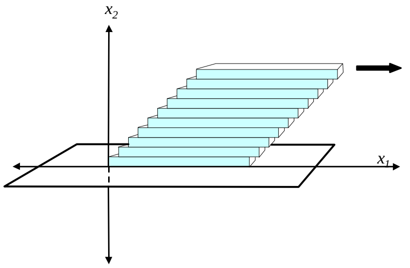
A Parallel Plate Melt Rheometer Incorporating a Shear Stress Transducer

(Sliding Plate Rheometer)

Can we use an alternate, easier geometry?

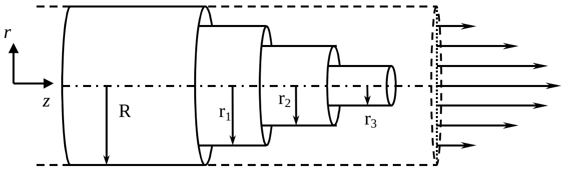
Shear is fundamentally a sliding flow

We can infer a viscosity from any sliding flow if we can relate the data back to homogeneous shear flow



Cartesian geometry

(parallel plates)



Cylindrical geometry

Telescoping sliding flow

(capillary flow)

Strategy:

Use capillary flow experiments (pressure drop versus flow rate) to infer viscosity

Implications:

- The flow is not the simple shear flow assumed when viscosity was defined;
- We need to analyze the flow with as few assumptions as possible;
- We need to design the apparatus to conform to the assumptions we make;
- When our assumptions are only approximately satisfied, we must correct the data where possible.

Capillary Rheometer

- **Shear viscosity** is the most widely measured rheological property
- For high shear rates, the **capillary rheometer** is the most effective instrument for measuring shear viscosity.
- The capillary rheometer does not produce pure, homogeneous shear flow, however.
- Various corrections are necessary in order to turn pressure-drop/flow-rate data from a capillary rheometer into viscosity.

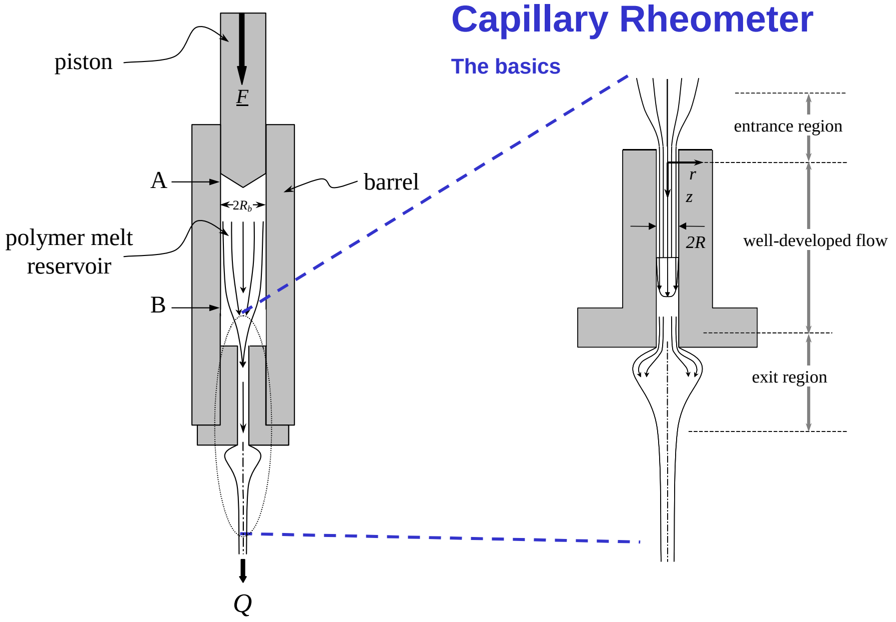
$$\eta = \eta(\dot{\gamma}) = \frac{\tau_{21}}{\dot{\gamma}}$$

Three corrections:

1. Entrance/exit pressure loss
2. Slip at the wall
3. Non-parabolic velocity profile

Capillary Rheometer

The basics

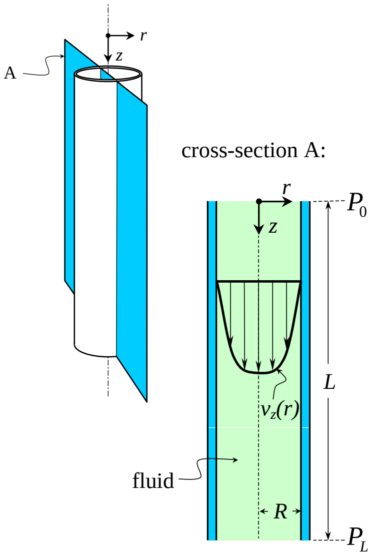


Goettfert Rheo-Tester 1000

Classic Newtonian flow problem:

Pressure-driven flow of a fluid in a tube

- •steady state
- •well developed
- •long tube (no z-dependence)
- •no-slip at the wall



For all fluids:

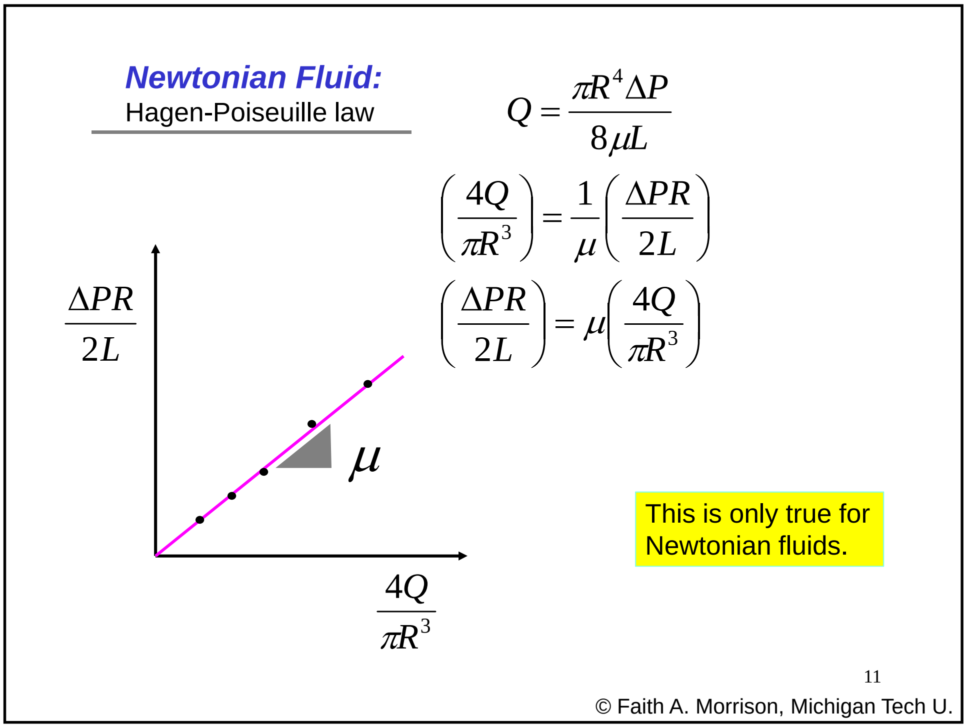
Stress at the wall: $\tau_R = \frac{\Delta PR}{2L}$

For Newtonian fluids only:

Shear rate at the wall: $\dot{\gamma} = \frac{4Q}{\pi R^3}$

Definition of viscosity:

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



Newtonian Fluid:

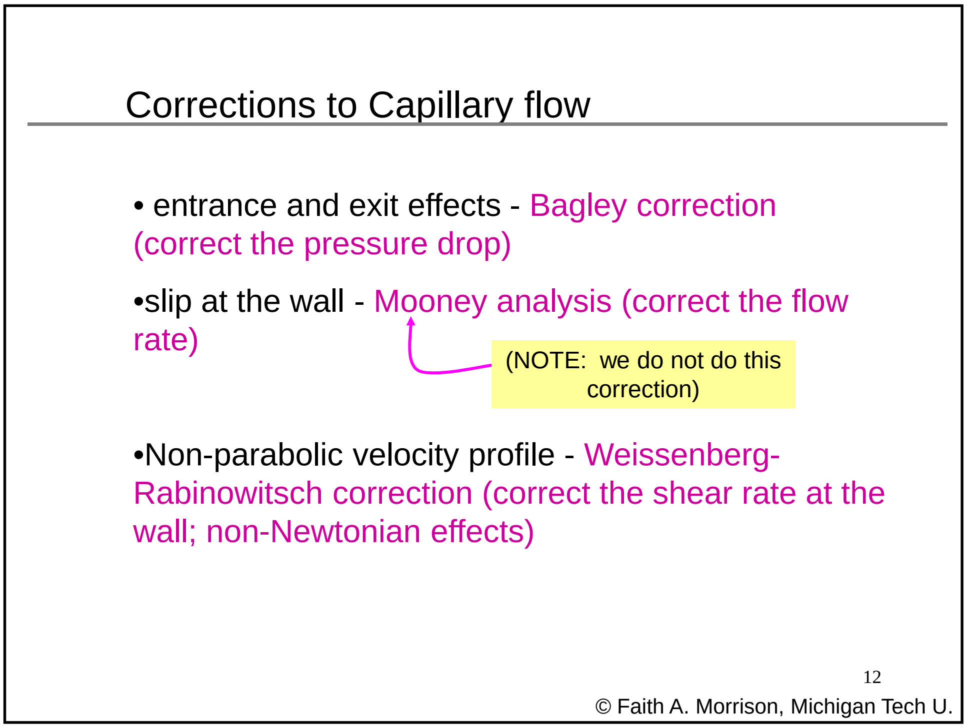
Hagen-Poiseuille law

$$Q = \frac{\pi R^4 \Delta P}{8\mu L}$$

$$\left(\frac{4Q}{\pi R^3}\right) = \frac{1}{\mu}\left(\frac{\Delta PR}{2L}\right)$$

$$\left(\frac{\Delta PR}{2L}\right) = \mu\left(\frac{4Q}{\pi R^3}\right)$$

This is only true for Newtonian fluids.



Corrections to Capillary flow

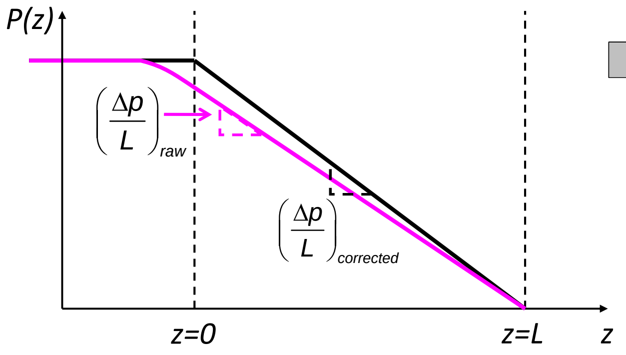
- entrance and exit effects - Bagley correction (correct the pressure drop)
- slip at the wall - Mooney analysis (correct the flow rate)

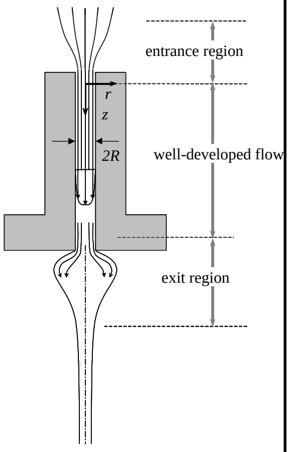
(NOTE: we do not do this correction)

- Non-parabolic velocity profile - Weissenberg-Rabinowitsch correction (correct the shear rate at the wall; non-Newtonian effects)

Entrance and exit effects - Bagley correction

The pressure gradient is not accurately represented by the raw Δp/L





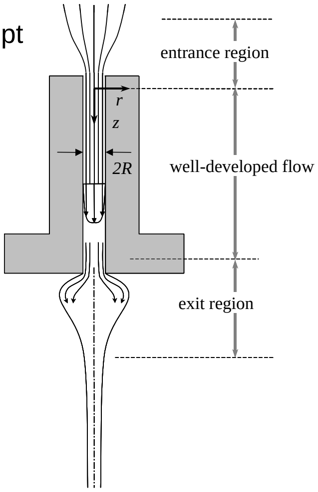
Entrance and exit effects - Bagley correction

$$\tau_R = \frac{\Delta PR}{2L} \Rightarrow \quad \Delta P = \left(2\tau_R\right)\frac{L}{R} \boxed{+0}$$

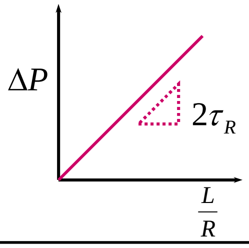
no intercept

Constant at fixed $\dot{\gamma}_a$

Run for different length capillaries



Straight line through origin with slope of $2\tau_R$:



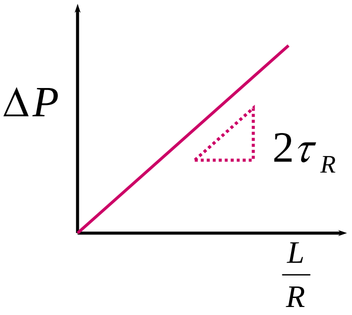
This is the result when the end effects are negligible.

Entrance and exit effects - Bagley correction

Procedure:

- For a standard set of apparent shear rates $4Q/\pi R^3$, measure ΔP in capillaries of different L/R (usually different lengths)
- Plot results and infer corrected shear stress from slope

$$\Delta P = (2\tau_R)\frac{L}{R}$$



Bagley Plot

$$\Delta P_{\substack{end\\effects}} = f(Q) = f(\dot{\gamma}_a)$$

$$\dot{\gamma}_a \equiv \frac{4Q}{\pi R^3} = \text{apparent shear rate}$$

$$\Delta P_{\substack{end\\effects}}\Big|_{\dot{\gamma}_a = 250 s^{-1}}$$

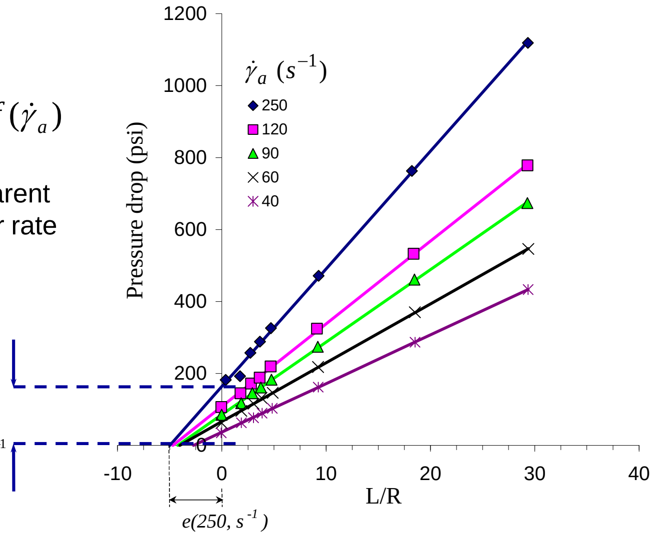
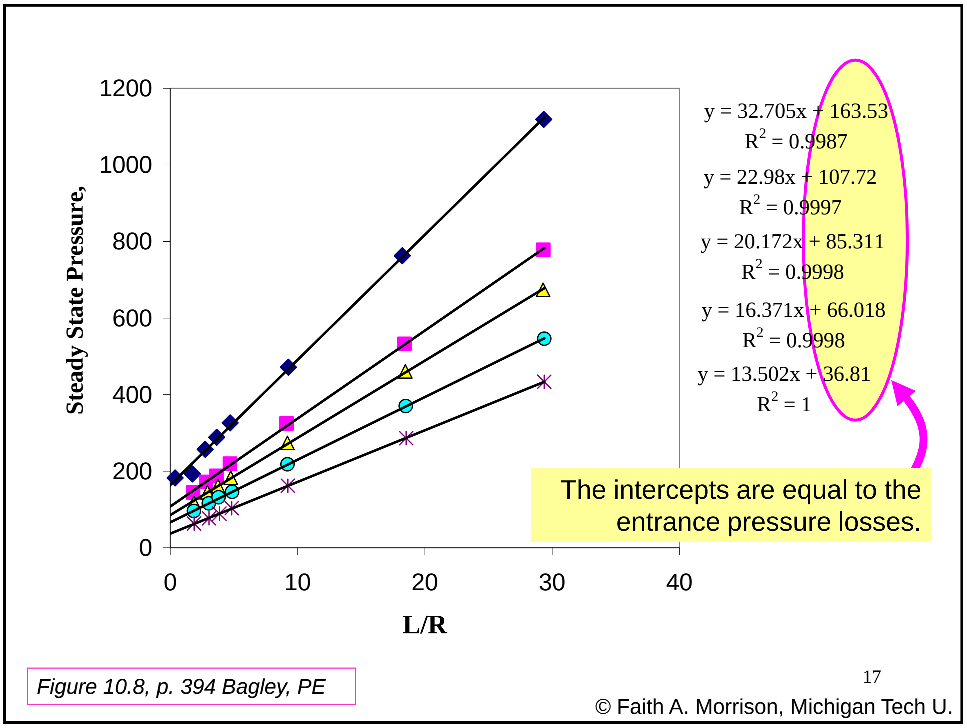


Figure 10.8, p. 394 Bagley, PE



y = 32.705x + 163.53
R^2 = 0.9987

y = 22.98x + 107.72
R^2 = 0.9997

y = 20.172x + 85.311
R^2 = 0.9998

y = 16.371x + 66.018
R^2 = 0.9998

y = 13.502x + 36.81
R^2 = 1

The intercepts are equal to the entrance pressure losses.

Figure 10.8, p. 394 Bagley, PE

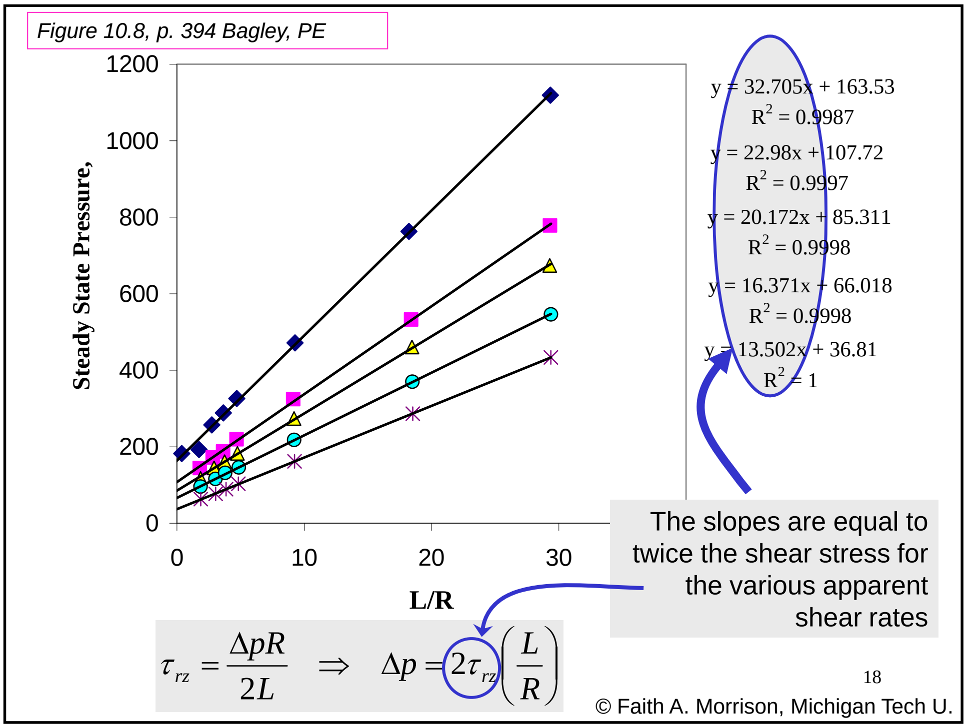


Figure 10.8, p. 394 Bagley, PE

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y = 13.502x + 36.81
R^2 = 1

The slopes are equal to twice the shear stress for the various apparent shear rates

$$\tau_{rz} = \frac{\Delta p R}{2L} \quad \Rightarrow \quad \Delta p = \boxed{2\tau_{rz}}\left(\frac{L}{R}\right)$$

The data so far:

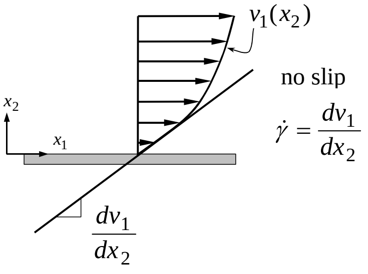
gammdotA (1/s)	deltPent psi	slope psi	sh stress psi	sh stress Pa
250	163.53	32.705	16.3525	1.1275E+05
120	107.72	22.98	11.49	7.9220E+04
90	85.311	20.172	10.086	6.9540E+04
60	66.018	16.371	8.1855	5.6437E+04
40	36.81	13.502	6.751	4.6546E+04

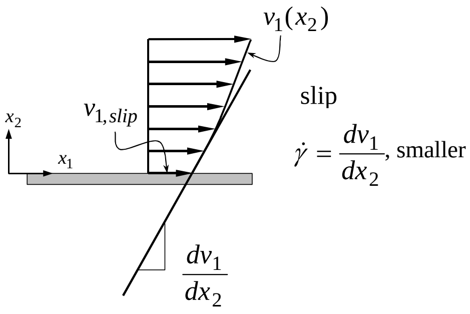
Now, correct shear rate
for slip at the wall

Figure 10.8, p. 394 Bagley, PE

Slip at the wall - Mooney analysis

Slip at the wall reduces the shear rate near the wall.





Slip at the wall - **Mooney analysis**

Slip at the wall reduces the shear rate near the wall.

$$\boxed{\dot{\gamma}_a = \frac{4Q}{\pi R^3} = \frac{4v_{z,av}}{R}}$$

without slip

$$\dot{\gamma}_{a,slip-corrected} \equiv \frac{4}{R}\left(v_{z,av} - v_{z,slip}\right)$$

with slip

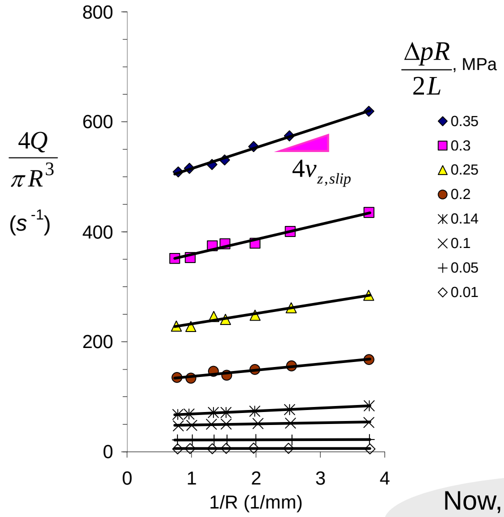
$$\equiv \frac{4v_{z,av}}{R} - \frac{4v_{z,slip}}{R}$$

$$\boxed{\frac{4v_{z,av}}{R} = \underbrace{4v_{z,slip}}_{\text{slope}}\left(\frac{1}{R}\right) + \underbrace{\dot{\gamma}_{a,slip-corrected}}_{\text{intercept}}}$$

Need capillaries of various radii

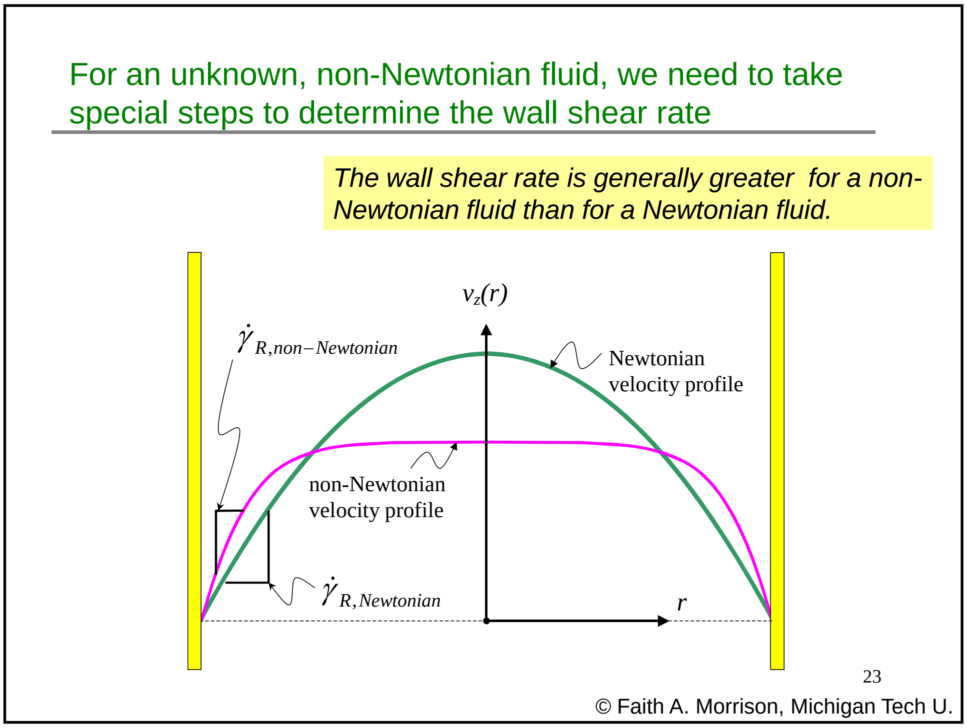
$$\frac{4v_{z,av}}{R} = \frac{4Q_{measured}}{\pi R^3}$$

Slip at the wall - **Mooney analysis**



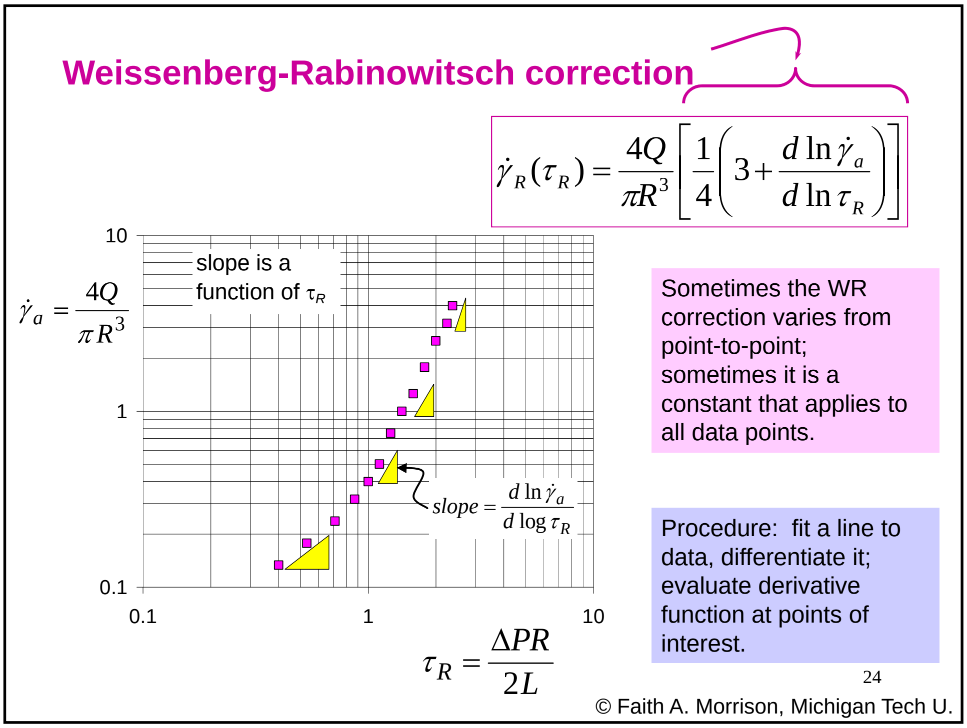
Now, correct shear rate for non-parabolic velocity profile.

Figure 10.10, p. 396
Ramamurthy, LLDPE



For an unknown, non-Newtonian fluid, we need to take special steps to determine the wall shear rate

The wall shear rate is generally greater for a non-Newtonian fluid than for a Newtonian fluid.



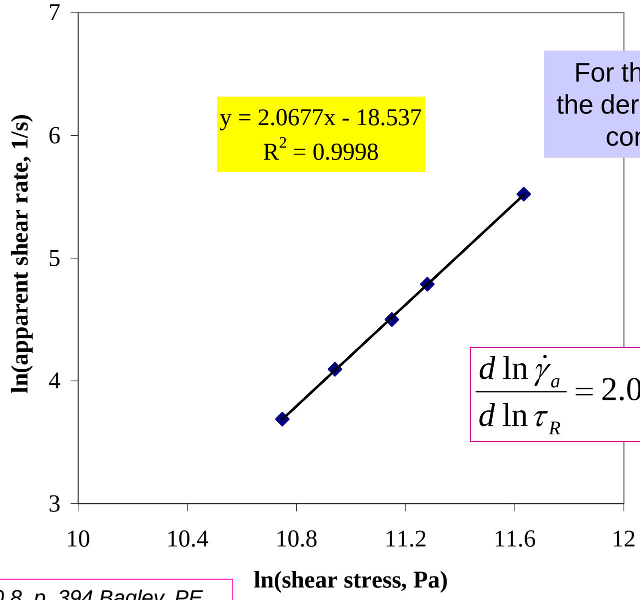
Weissenberg-Rabinowitsch correction

$$\dot{\gamma}_R(\tau_R) = \frac{4Q}{\pi R^3}\overbrace{\left[\frac{1}{4}\left(3+\frac{d\ln\dot{\gamma}_a}{d\ln\tau_R}\right)\right]}$$

Sometimes the WR correction varies from point-to-point; sometimes it is a constant that applies to all data points.

Procedure: fit a line to data, differentiate it; evaluate derivative function at points of interest.

Weissenberg-Rabinowitsch correction



For these data the derivative is a constant.

This slope is usually >1

$$\frac{d \ln \dot{\gamma}_a}{d \ln \tau_R} = 2.0677$$

Figure 10.8, p. 394 Bagley, PE

The data corrected for entrance/exit and non-parabolic velocity profile:

$$\eta = \frac{\tau_R}{\dot{\gamma}_R}$$

gammdotA (1/s)	deltPent psi	deltPent Pa	sh stress Pa	ln(sh st)	ln(gda)	WR correction	gam-dotR 1/s	viscosity Pa s
250	163.53	1.1275E+06	1.1275E+05	11.63289389	5.521460918	2.0677	316.73125	3.5597E+02
120	107.72	7.4270E+05	7.9220E+04	11.2799902	4.787491743	2.0677	152.031	5.2108E+02
90	85.311	5.8820E+05	6.9540E+04	11.14966143	4.49980967	2.0677	114.02325	6.0988E+02
60	66.018	4.5518E+05	5.6437E+04	10.9408774	4.094344562	2.0677	76.0155	7.4244E+02
40	36.81	2.5380E+05	4.6546E+04	10.74820375	3.688879454	2.0677	50.677	9.1849E+02

Now, plot viscosity versus wall-shear-rate

Figure 10.8, p. 394 Bagley, PE

Viscosity of polyethylene from Bagley's data

$$\eta = \frac{\tau_R}{\dot{\gamma}_R}$$

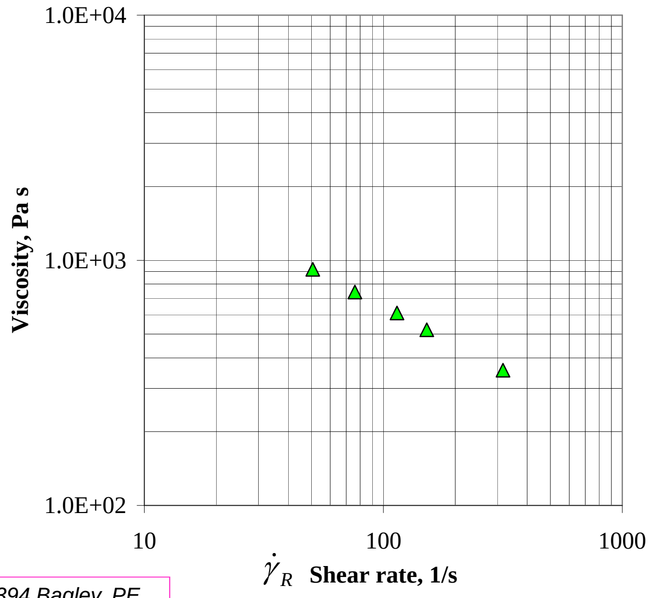


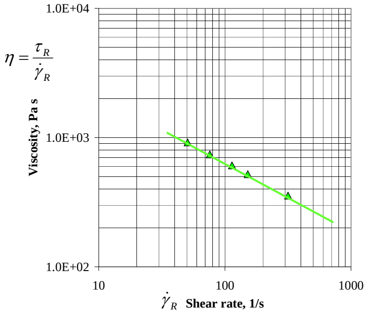
Figure 10.8, p. 394 Bagley, PE

Viscosity from Capillary Experiments, Summary:

1. Take data of pressure-drop versus flow rate for capillaries of various lengths; perform Bagley correction (entrance pressure)
2. If slip is an issue, take data for capillaries of different radii; perform Mooney correction (slip)
3. Perform the Weissenberg-Rabinowitsch correction (wall shear rate)
4. Plot true viscosity versus true wall shear rate
5. Fit to a power-law to obtain model parameters if desired.

raw data: $\Delta P(Q)$

final data: $\eta = \tau_R / \dot{\gamma}_R$



Capillary Rheometer Measurement Procedure

- Choose range of apparent shear rates and capillary sizes to allow for desired corrections.
- Conduct experiments: load reservoir; push polymer through capillary with piston
- **Measure pressure** at top of capillary at the base of the barrel (correct for entrance and exit losses); obtain wall shear stress.
- **Measure piston speed** (convert to flow rate, correct for slip); obtain apparent shear rate.
- Plot apparent shear rate versus wall shear stress; obtain WR correction
- Correct apparent shear rate for non-parabolic velocity profile (non-Newtonian)
- Calculate true non-Newtonian viscosity from stress/shear rate.