Practical Rheology
LCR 7001 Capillary Rheometer

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1. Introduction
2. Shear Sweep Test (Polymer Flow Behavior)
3. Rabinowicz and Bagley Corrections
4. Extensional Viscosity Measurements
5. Wall Slip Velocity Calculation
6. Time Sweep Test (Thermal Stability of Polymers)
7. Die Swell Measurements
8. MFR Correlation
9. Intrinsic Viscosity of PET
10. LCR Dies Information
Introduction
Why Capillary Rheometer?

- The most common melt rheometer to analyze flow behavior of polymers under processing condition (shear rate, time, temperature).

- Duplication of processing parameters for design, simulation, and trouble shooting purposes in a faster way.

- Predict optimal operating condition based on correlation of rheological data from capillary rheometer to processing parameters.

- Analyze different materials for various applications and design.

APPLICATIONS

- Polymer viscosity at wide deformation rate range
- Polymer melt flow behavior
- Shear thinning behavior
- Polymer stability over time/temperature
- Elastic properties (die swell, wall slip)
Dynisco® LCR 7001

Force measurements from **Load Cell**

Pressure measurement from **Pressure Transducer**

**RESULTS:**
Polymer flow curve (LabKars Software)

**MEASURES:**
- Force
- Pressure
- Ram rate
- Time
- Temperature
- Expansion of extrudate

**CALCULATES:**
- Shear stress
- Shear rate
- Shear/extensional Viscosity
- Die swell ratio
General Specification

LCR7001 SPECIFICATION

- Temp range: 25-500 °C
- Shear rate range: 1-100000 1/s
- Barrel diameter: 9.55 mm
- Available length: 227 mm
- Working length: 125 mm
- Min piston speed: 0.03 mm/min
- Max piston speed: 650 mm/min
- Max force measurement from load cell: 10 KN
- Max pressure measurements: 1400 Bar
- Accuracy of test-to-test ~1.5-2%
- Accuracy of rheometer-to-rheometer ~8%

Extrusion & Capillary Rheometer

Motor

Hopper

Screw

Barrel

Thermocouple

Pressure Transducer

Die

http://www.polymerprocessing.com
LCR Preparation Before Running the Test

1. Swing out barrel.
2. Insert the die in the die holder and die fastener.
3. Fasten die to bottom of barrel. Rotate un-clockwise to close.
4. Pour sample into funnel – about 10 grams.
5. Compress pellets with tamp.
6. Place the piston inside barrel and lock it.
7. Lower safety shield.
LCR Cleaning After the Test

1. Clean plunger with a brush while hot.
2. Ensure grooves are clean.
3. Remove and clean die. (rotate clockwise to open)
4. Put cotton swabs into position to clean barrel.
5. Push cotton swabs through barrel.
Shear Sweep Test

(Polymer Flow Behavior)
What is Shear?

Shear Stress

\[ \tau = \frac{F}{A} \] (Pa or N/m²)

Shear Rate

\[ \gamma = \frac{\Delta x}{\Delta t} \] (1/S)

Where:
- F = Force
- A = Area of layer
- H = Gap between layers
- x = Displacement of layer
- T = Time
Calculation of Rheological Data in LCR

### Apparent shear rate (based on piston speed)

\[ \dot{\gamma}_a = \frac{4Q}{\pi R_c^3} \]

- \( \dot{\gamma}_a (1/s) \): apparent shear rate
- \( Q (mm^3/sec) \): volumetric flow rate
- \( S (mm/min) \): piston speed
- \( D_b (mm) \): barrel diameter
- \( R_c (mm) \): die radios

### Wall shear stress (based on force or driving pressure)

\[ \tau_w = \frac{F/\pi D_b^2}{4(L/D)_{\text{die}}} \]

- \( \tau_w (Pa) \): wall shear stress
- \( F (N) \): force from “load cell” on piston
- \( D_b (mm) \): barrel diameter
- \( L/D \): length to diameter ratio of the die
- \( P_d (Pa) \): driving pressure at the die entrance from “pressure transducer”

\[ \tau_w = \frac{P}{4(L/D)_{\text{die}}} \]

### Apparent shear viscosity

\[ \eta_a = \frac{\tau_w}{\dot{\gamma}_a} \]

- \( \eta_a (Pa-s) \): apparent shear viscosity
- \( \tau_w (Pa) \): wall shear stress
- \( \dot{\gamma}_a (1/s) \): apparent shear rate

---

**Assumptions:**

1. Fully developed, isothermal, steady state, and Laminar Flow
2. No radial or circumferential velocity components
3. Incompressible fluid
4. No slip at the wall of the die
Random coil
Highly entangled

Stretching
Partially aligned

Oriented
Highly aligned

Shear Flow in Viscoelastic Polymer Melts

Log Viscosity (Resistance to Flow)

Log Shear Rate (Rate of Flow)
Effect of Various Factors on Polymer Flow Curve

How an **Increase** in Various Factors Affects the Polymer Melt Viscosity Curve

![Graph showing the effect of various factors on polymer melt viscosity curve](image-url)
Flow Curve of Polymer Melts

- **Newtonian plateau**
- **Shear thinning (Power-law) Region**
- **Log viscosity** vs. **log shear rate**

- **$\eta_0$** → **$\dot{\gamma}_0$**
  - Zero-shear viscosity
  - Characteristic shear rate

- **Mw** → **MWD**
  - Higher Mw
  - Lower Mw
  - Narrow MWD
  - Broad MWD

- **Log viscosity** vs. **log shear rate**

**Dynisco**
Molecular Weight Distribution (MWD)

Polymers with Broader MWD:
- Better processability (fluidity)
- Less viscous dissipation during their process
- Less energy consumption during their process
- Lower mechanical properties

Narrow MWD

Broad MWD
Analyzing Polymer Degradation from Flow Curve

Flow curve of plastic samples before process and after process at various screw speeds in an extrusion.

Extrusion Condition
- Heat
- Barrel temp
- Mechanical shear
- Screw speed
- Residence time
- Flow rate

Higher $\eta_0$ and $M_w$

Lower $\eta_0$ and $M_w$

Degradation

**Mw Dependence of Zero-Shear Viscosity**

Critical Molecular wt. \((M_C)\) molecular entanglements occur

Polymers with higher Mw:
- Higher intermolecular entanglement
- Higher strength
- Higher chemical resistance

**Fox-Flory Equation**

\[
\eta_0 = K_2 (M_W)^{3.4}
\]

Where:
- \(\eta_0\) = “Zero shear rate” melt viscosity.
- \(K_2\) = Constant
- \(M_W\) = Wt. avg. molecular wt.
- \(M_C\) = “Critical” molecular wt.

Quantitative Relationships for the Dependence of Viscosity upon Shear Rate

**Power-law Model**

\[ \eta(\dot{\gamma}) = k\dot{\gamma}^{n-1} \]

- \( k \) (Pa-s): Consistency
- \( n \): Power-law index

- “Only” fits the shear-thinning (power-law) portion of the curve.
- Shear-thinning exponents dependent on intermolecular forces.
- \( n \) ranges from 0.2-0.9 depending upon the type of polymer.
- \( n \) equals to 1 for Newtonian materials.
- \( n \) represents the processability (shear-thinning intensity).
- Polymers with lower \( n \) are more sensitive to the shear rate.
- \( n \) decrease with broader MWD.
- \( k \) has temperature dependency and controlled by Mw.

Quantitative Relationships for the Dependence of Viscosity upon Shear Rate

Modified Cross Model

\[ \eta(\dot{\gamma}) = \frac{\eta_0}{1 + \left(\frac{\eta_0 \dot{\gamma}}{\tau^*}\right)^{1-n}} \]

- \( \eta_0 \) (Pa-s): Zero-shear viscosity
- \( \tau^* \) (Pa): Critical shear stress
- \( n \): Power-law index

- Combines the power-law and Newtonian regions.
- Also fits the low shear Newtonian plateau.
- \( \eta_0 \) is controlled by molecular weight.
- \( \tau^* \) is stress at the beak in curve and controlled by MWD.
- Broader MWD (by blending or branching) cause earlier \( \tau^* \).

Viscosity-Temperature Dependence

**Window into the process**

- **Williams-Landel-Ferry (WLF) model:** \( T_g < T < T_g + 100 \)

\[
\log(a_T) = \frac{\eta_0(T)}{\eta_0(T_{ref})} = \left[ \frac{-C_1(T - T_g)}{C_2 + T - T_g} \right]
\]

\( C_1 = 17.44 \)
\( C_2 = 51.6 K \)

- **Arrhenius model:** \( T > T_g + 100 \)

\[
a_T = \frac{\eta_0(T)}{\eta_0(T_{ref})} = \exp\left[ \frac{E_a}{R} \left( \frac{1}{T} - \frac{1}{T_{ref}} \right) \right]
\]

\( E_a: \text{Activation Energy} \)
\( R: \text{Universal gas constant} = 8.314 \times 10^{-3} \text{ kJ/mol.K} \)
A Window into Your Process!

- At shear rate 100-1000 1/s (common at normal RPM in extrusion), the processing of which polymer cause higher torque and head pressure, and viscous heating in the extrusion?

- Which polymer has higher shear-thinning behavior (easier process-ability)?

- With increasing screw speed which polymer will have the highest flow rate?

- Which polymer has the highest molecular weight?

- Which polymer had the Broader MWD?

- Which polymer might have linear might have long chain-branching and which one might have linear structure?

[Image: Resin Viscosity Comparison at 225 C]

https://www.ptonline.com/columns/how-to-spec-a-flat-die
Which polymer might face higher thermal degradation with increasing temperature?

Which polymer has wider processing temperature?
Shear-Sweep Test Setup in LabKars

1. Add/delete Data Point Setup
2. Program Name
3. Start Position (mm): 89-100 mm
4. Temperature (°C): barrel temperature set point
5. Melt time (sec): 180-360 sec
6. Sensor1 ID: LC-103N
7. Die: choose entrance angle, D and L/D from list
8. Minimum/Maximum Speed (mm/min): Min at 0.03 and Max at 650 mm/min
10. Speed Control range (mm/min)
11. Shear Rate range (1/s)
12. Send: sending test setup to rheometer
Real-time data graph during the test

1. RCAL: balance transducers!
2. Run: run the test!
3. Acquire: collect data!
4. Stop: stop the test!
5. Purge: purge all the material in barrel!
6. Up: move plunger upward!
7. Down: move plunger downward!
8. Park: go to park position (25mm)!
9. Sensor #1 (N): force on load cell
10. Sensor #2 (N): force on pressure transducer
11. Active Ram Rate (mm/min): piston speed
12. Temperature (°C): actual barrel temperature
13. Laser (mm): die swell detection
14. Plunger position (mm)
15. Run time (sec)
16. Collected point: at each shear rate
Data Analysis

Plot of log apparent viscosity versus apparent shear rate

Click to make logarithmic scale

Right Click to change the axis
Data Analysis

Power-law model analysis

Choose “Power Law”

Power law fitting data
Data Analysis

Modified cross model analysis

Choose “Modified Cross”

Modified cross fitting data
Select “Single Run/Modify” to see the raw data.

- Max, min, average, mean, and standard deviation information of all parameters.

Select the type of sensor:
- Sensor 1: Load cell
- Sensor 2: Pressure transducer

Raw data: [table of data]
Export Raw Data in Excel

Select "Export" to begin the process.

Choose your data set from the available options.

Select "Single File" to export the data in Excel format.

Raw data in Excel sheet is displayed, ready for further analysis or use.
Rabinowicz and Bagley Corrections
All calculations we discussed are “Apparent” values since they assume Newtonian behavior and that the entire pressure drop occurs inside through die. (Assuming no die entrance/exit effect)

Rabinowicz correction needs to be applied in order to rectify the data for non-Newtonian character of polymer melt. (Calculation of corrected shear rate)

Bagley correction needs to be applied to consider the extra pressure drop that may happen at the entrance/exit of the die. (Calculation of corrected shear stress)
Why Rabinowicz Corrections?

Flow through a die

Flow Velocity

Newtonian fluid (Parabolic Flow)

Polymer melt (Plug Flow)

Shear rate profile

\( \dot{\gamma} \) Newtonian

\( \dot{\gamma} \) Non-Newtonian

http://www.eng.uc.edu/~beaucag/RyanBreese/Rheolog/rheology.htm
Rabinowicz Corrections

\[
\dot{\gamma}_{\text{corrected}} = \left(\frac{3n + 1}{4n}\right) \dot{\gamma}_{\text{apparent}}
\]

Correction Factor

Pseudoplastic polymer melts [n<1]

Newtonian [Slope n=1]
How to Apply Rabinowicz Corrections in LabKars?

Click to plot viscosity versus “corrected” shear rate.
Why Bagley Corrections?

- **A = Δp_{Barrel}**: Very minor pressure drop in barrel
- **B = Δp_{Entrance}**: Excess pressure drop in die entrance
- **C = Δp_{Capillary}**: Fully developed flow region in capillary die

- Large pressure drop associated with the flow entrance region due to viscoelasticity of polymers.
- After entrance region, the pressure gradient approaches a constant value (fully developed flow region).
- In reality:
  \[ \Delta P_{total} = \Delta P_{Entrance} + \Delta P_{capillary} \]
- Bagley correction needs to be applied to calculate the entrance pressure drop.
How to Calculate the Entrance Pressure?

Three dies, same diameter but different lengths (e.g. L/D: 10, 20, and 30)

How do you calculate the entrance pressure when dealing with three dies of the same diameter but different lengths (e.g., L/D: 10, 20, and 30)?

The entrance pressure, $P_{Entrance}$, is calculated as follows:

$$P_{Entrance} = P \left( \text{at } \frac{L}{D} = 0 \right)$$

Where $P$ is the pressure at the entrance, $L$ is the length, and $D$ is the diameter.

The corrected stress, $\tau_{Corrected}$, is calculated using:

$$\tau_{Corrected} = \frac{P_{Reading} - P_{Entrance}}{4\left(\frac{L}{D}\right)}$$

**OR**

$$\tau_{Corrected} = \frac{P_{Reading}}{4\left(\frac{L}{D} + e\right)}$$

Where $e$ is the end correction defined as:

$$e = \frac{-L_{Entrance}}{D}$$

---

How to Apply Bagley Corrections in LabKars?

Select “Global Preferences” under “File” menu

Select “Attempt Bagley Correction”

Insert number “2”

Select “Copy Table to Clipboard” under “Edit” menu to get the raw data

Short die (L ≤ 10 mm)

Long die (L ≥ 15 mm)
How to Calculate True Viscosity?

\[ \eta_{\text{True}} = \frac{\tau_C}{\dot{\gamma}_C} \]

where

- \( \eta_{\text{True}} \) (Pa-s): Viscosity with Bagley and Rabinowicz Corrections
- \( \tau_C \) (Pa): Bagley corrected shear stress
- \( \dot{\gamma}_C \) (1/s): Rabinowicz corrected shear rate
Viscoelasticity
Mechanical Models of Viscoelastic Behavior

**Purely Elastic Response**

\[ \sigma = E \varepsilon \]

**Purely Viscous Response**

\[ \sigma = \eta \frac{d\varepsilon}{dt} \]

Hooke's law:

\[ \sigma = E \varepsilon \]

Newtonian fluid:

\[ \tau_{xy} = \eta \dot{\gamma} \]
Mechanical Models of Viscoelastic Behavior

(c) VISCOELASTIC RESPONSE

Strain

Creep

Recovery

Stress applied

Stress removed

Time

Permanent deformation

VISCOELASTIC

Ideally elastic behaviour

Ideally viscous behaviour
Extensional Viscosity Measurements
When Extensional Viscosity is important?

Any process that has stretch!
What is Extensional (Elongational) Viscosity?

- Since the diameter of the barrel versus die is very large, there is a high degree of stretching along streamlines at the entrance to the die.
- Cogswell assumes that the pressure drop in entrance has two components, one due to shear and one due to extension.
### Cogswell’s Equations

<table>
<thead>
<tr>
<th>Extensional Stress</th>
<th>[ \sigma_{Ext} = \frac{3}{8}(n + 1)P_{Ent} ]</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \sigma_{Ext} ) (Pa): Extensional stress</td>
<td></td>
</tr>
<tr>
<td>( n ): Power law index</td>
<td></td>
</tr>
<tr>
<td>( P_{Ent} ) (Pa): Entrance pressure</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Extensional Rate</th>
<th>[ \dot{\varepsilon} = \frac{4\dot{\gamma}<em>a^2\eta_a}{3(n+1)P</em>{Ent}} ]</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \dot{\varepsilon} ) (1/s): Extensional rate</td>
<td></td>
</tr>
<tr>
<td>( \dot{\gamma}_a ) (1/s): Apparent shear rate</td>
<td></td>
</tr>
<tr>
<td>( P_{Ent} ) (Pa): Entrance pressure</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Extensional Viscosity</th>
<th>[ \eta_{Ext} = \frac{9(n+1)^2P_{Ent}^2}{32\eta_a\dot{\gamma}_a^2} ]</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \eta_{Ext} ) (Pa-s): Extensional viscosity</td>
<td></td>
</tr>
</tbody>
</table>

---

How to Perform Extensional Viscosity Measurements in LabKars?

Select “Cogswell Extensional Viscosity Calculation”

Insert number “2”

Select “Global Preferences” under “File” menu

Select “Copy Table to Clipboard” under “Edit” menu to get the raw data

Right click on each axis and select the extensional type of axis

Short die ($L \leq 10$ mm)

Long die ($L \geq 15$ mm)
LLDPE (Linear structure) is stiffer than LDPE (branched structure) in shear but softer in extension.

Example!

http://www.eng.uc.edu/~beaucag/RyanBreese/Rheolog/rheology.htm
Wall Slip Velocity & Melt Farcture
Wall slip velocity increases dramatically above critical shear stress (~0.1 MPa).

- Slippage reduces apparent viscosity. Also, the surface of the extrudate begins to be rough (melt fracture).
- Wall slip happens due to elastic properties of polymer materials.
- Critical shear stress is lower for polymers with higher molecular weight.
- Trouble shooting by using larger die diameter, longer die, tapering the die, higher temperature, or lower shear rate.
Effect of Wall Slip on Capillary Rheometer Results

Wall slip causes formation of plug flow and a discontinuity in flow curve.


How to Calculate Wall Slip Velocity?

\[ \dot{\gamma}_a = 4V_w \left( \frac{1}{R_C} \right) + X \]

where

- \( V_w \text{ (mm/sec)} \): Wall slip velocity
- \( \dot{\gamma}_a \text{ (1/s)} \): apparent shear rate at a given value of shear stress
- \( R_C \text{ (mm)} \): Capillary die radios
- \( X \): Dimensionless parameter (a function of the shear stress)

Reference:

**Steps:**
1. Produce a series of flow curves using a set of dies of varying radius \((R_C)\).
2. At a given value of shear stress, make a plot of apparent shear rate \((\dot{\gamma}_a)\) versus inverse radius \((\frac{1}{R_C})\).
3. Slip velocity at a given shear stress will be one quarter of the slope of \(\dot{\gamma}_a\)-vs-\(\frac{1}{R_C}\) plot.
4. Repeat the test at other shear stresses and calculate the slip velocity at each specific shear stress.
5. Make the plot of slip velocity versus shear stress.
How to Notice Wall Slippage from Flow Curve in LCR?

Perform shear rate sweep on two dies with same L/D but different diameters.

NOT overlapping of results from two dies means slippage happened at the smaller die.

Viscosity is lower when slippage happens in smaller die diameter.

Slippage affects Power-Law model parameters.
This phenomena happens due to elasticity of polymer melts. **Any procedure that reduces the melt elasticity will help to troubleshoot.** e.g.:

- Increasing temperature
- Reducing screw speed (shear rate)
- Increasing the die length or die diameter
- Tapering the die
Time Sweep Test

(Thermal Stability of Polymers)
 Thermal Stability of Polymer Melt

The stability test can determine the resistance of a material to a change in viscosity at the test temperature.

The stability of polymer melts varies depending on temperature, time at temperature, formulation, and contaminants.

This test can be used to show the presence of moisture or reactive chemicals in a polymer.

This test can be used to measure the degradation rate or reactivity of a sample.

- Very hot for a short time or
- Hot for a long time

Amorphous

Polymerization
Cross-linking
Degradation

Time at Temperature

Viscosity (Pa-s)

(Coast shear rate or shear stress)

Polymerization

Cross-linking

Stable

Degradation

Time at Temperature (Minutes)
How to Set up Thermal Stability Test (Time Sweep) in LabKars?

Select "Position" Test Type

\[ \Delta L = S \times \Delta t \]

- \( \Delta L \): Piston travel distance between points
- \( S \): Piston speed
- \( \Delta t \): Piston time travel between points

\[
\begin{align*}
S &= \frac{\text{Max plunger travel}}{\text{Test time}} \quad (130 \text{ mm}) \\
\Delta t &= \frac{\text{Test time}}{\# \text{ of points}} \quad (\text{e.g.} 20 \text{ min})
\end{align*}
\]

Applying the same speed and shear rate multiple times

\[
\begin{align*}
\Delta L &= S \times \Delta t \\
13 &= 6.5 \times 2 \\
6.5 &= 130 \text{ mm/sec sec}
\end{align*}
\]
How to Set up Thermal Stability Test (Time Sweep) in LabKars?

Add delay time to increase the stability test time while keeping shear rate constant.
Thermal Stability Test Results (Time Sweep) in LabKars

Plot of apparent viscosity versus residence time
Die Swell
Expansion (re-coiling) of extrudate after exiting die.
Qualitative measure of melt elasticity.
Relaxed die swell is used to predict part dimension
Running die swell is used to predict productivity of extrusion process.
Analysis of extrudate smoothness.
Trouble shooting by using larger die diameter, longer die, tapering the die, lower shear rate, or higher temperature.

**Percent die swell**

\[
\text{Percent die swell} = \frac{D_{\text{Extrudate}} - D_{\text{die}}}{D_{\text{die}}} \times 100
\]
Die Swell Measurement in LCR

- Detection: CCD element
- Light source: 800 nm laser
- Resolution: 2.75 µm
- Measuring range: 0.13-23 mm
- Response time: 1.4 ms
- Accuracy: ±0.003 mm
Melt fracture produce a noisy die swell measurements.

This phenomena happens due to elasticity of polymer melts. **Any procedure that reduces the melt elasticity will help to troubleshoot.** e.g.:

- Increasing temperature
- Reducing screw speed (shear rate)
- Increasing the die length or die diameter
- Tapering the die

http://www.rheoheat.se/vr_rheo.html
MFR Correlation
How to Calculate MFR from Capillary Rheometer?

1. Calculate the shear stress in the melt flow rate tester (using a standard die)

<table>
<thead>
<tr>
<th>Weight in MFR test (kg)</th>
<th>Shear stress (Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.16</td>
<td>19350</td>
</tr>
<tr>
<td>5.00</td>
<td>44792</td>
</tr>
<tr>
<td>10.00</td>
<td>89584</td>
</tr>
<tr>
<td>21.60</td>
<td>193502</td>
</tr>
</tbody>
</table>

2. Determine the shear rate achieved at this shear stress from material flow curve
3. Calculation of melt volume rate (MVR) as follow:

\[ \text{MVR} = 600\dot{\gamma}\frac{\pi R^3}{4} \]

where
- \( \text{MVR} (cm^3/10\text{min}) \): Melt volume rate
- \( \dot{\gamma} (\frac{1}{s}) \): Shear rate determined at “step 2”
- \( R (cm) \): Standard melt flow rate tester die radius

4. MFR calculation knowing polymer melt density as follow:

\[ \text{MFR} = \text{MVR} \times \rho_m \]

where
- \( \rho_m (\frac{g}{cm^3}) \): Polymer melt density
How to Measure MFR in LabKars?

Select "Interpolate"

Insert "Load" in g and polymer "Melt Density" in $g/cm^3$ in "Melt Flow" section

Set "Coefficient in Polynomial" as 3
How to Measure Melt Density in LCR?

Select “Position” Test Type

Manage the positions to have 14 mm of piston travel distance between points

Five measurements of melt density

Whenever the plunger is in its delay time, cut the extrudate and weigh it
Intrinsic Viscosity of PET
Creating a dilute solution of the polymer and comparing the flow rate of the solution to the flow rate of the pure solvent

**Advantages**
- Performing at room temperature
- No need to melt the polymer
- No need to dry hygroscopic polymers (e.g. PET, PA)
- Common flowability specification (rather than MFR) among the manufactures of hygroscopic and filled polymers

**Disadvantages**
- Delicate apparatus
- Using of noxious chemicals as solvent
- Not environmental friendly

What is Intrinsic/Solution Viscosity (IV)?

http://alokrij.weebly.com/dpt-3rd-sem.html
Relationship between Melt Viscosity and Intrinsic Viscosity

Melt viscosity: $\eta_{\text{melt}} \propto M_w$

Fox-Flory Equation:

$\eta_0 = K_1(M_w)^{3.4}$

$\ln[\eta_0] = \ln K_1 + 3.4 \ln[M_w]$

Intrinsic viscosity: $\eta_{\text{Intrinsic}} \propto M_w$

Mark-Houwink Equation:

$\eta_{\text{Intrinsic}} = K_2(M_w)^a$

$\ln[\eta_{\text{Intrinsic}}] = \ln K_2 + a \ln[M_w]$

PET Melt Viscosity Versus Intrinsic Viscosity

\[ \text{Intrinsic Viscosity (dl/g)} \]

\[ \text{Zero Shear Rate Melt Viscosity (Pa-s)} \]

Temperature = 285°C

\[ \ln[\eta_{\text{Intrinsic}}] = \frac{a}{3.4} \ln[\eta_0] + K'' \]

Slope: \( \frac{a}{3.4} \)

For PET: a=0.75

Intercept: \( K'' \)
How to Determine IV of PET in LCR Capillary Rheometer?

Use Modified Cross to Get IV Button for PET at 285 C
LCR Dies Information
LCR Die Part Number Code

Formula for die part number:

**ADDD-LL**

where

- **A**: entry angle  
  \((W=60^\circ, Y=90^\circ, X=120^\circ, Z=180^\circ)\)
- **DDD**: diameter in inches \(\times 10000\)
- **LL**: length to diameter ratio
**Die Diameter**

- Smaller diameter produces higher shear rates.
- Larger diameter causes less elastic deformation applied at the entrance of the die.
- Larger diameter causes less entrance pressure drop, less die swell, less extensional deformation, and less slippage.
- For calculation of wall slip velocity at least 2 dies with different diameters (same L/D) are required.

<table>
<thead>
<tr>
<th>Die Diameter (mm)</th>
<th>Max shear rate (1/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.38</td>
<td>142000</td>
</tr>
<tr>
<td>0.75</td>
<td>18700</td>
</tr>
<tr>
<td>1.00</td>
<td>7900</td>
</tr>
<tr>
<td>1.50</td>
<td>2300</td>
</tr>
<tr>
<td>2.00</td>
<td>980</td>
</tr>
</tbody>
</table>
Die Length

- The portion of fully developed flow in compare with entrance region increases with increasing the die length.
- The percent error produced by entrance region is less with longer die.
- Short die for measuring elastic deformation (e.g. die swell, slippage) and long die for measuring shear viscosity.
- Noisy reading from short die at very low shear rates.
- For calculation of entrance pressure (Bagley correction) and extensional viscosity, at least 2 dies with different L/D ratio (same diameter) are required.
Die Entrance Angle

- Entrance angle has effect on flow patterns at the entrance to the capillary die.
- Lower angle causes smoother flow, less vortex flow, and less energy consumption at the die entrance.
- Lower angle reduces the elastic deformations (e.g. less entrance pressure drop, extensional deformation, die swell, melt fracture and wall slippage).
- Lower angle favors shear flow.

Stationary vortex flows in the corner region

Flow pattern at the entrance to a die with a flat entrance (180°)
“Everything Flows” - Heraclitus

QUESTIONS?