



From lab to production,  
providing a window into the process



# LMI Melt Flow Rate Test Applications & Calculation

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# Outline

- ❖ Melt Flow Rate Test Applications
- ❖ LMI Melt Flow Rate Tester
- ❖ Method A
- ❖ Method B
- ❖ Method A to B Conversion
- ❖ Operation Points
- ❖ Other Applications (Shear thinning behavior, intrinsic viscosity, and thermal stability)

# Melt Flow Rate Test Applications & Benefits

Standardized measure of flow characteristic of polymer melts at a single point specific condition



ASTM D1238  
ISO 1133

| HDPE 9018   |  | Density: 0.958-0.96  | MFI: 14-20  |
|---|--|--|-------------|
| Characteristic Properties   | Main Applications  | Additives  |             |
| <ul style="list-style-type: none"> <li>Excellent stability with high stress</li> </ul>                    | <ul style="list-style-type: none"> <li>Injection molding grade</li> <li>Nonwoven and tape application</li> <li>Fast cycle</li> </ul> | <ul style="list-style-type: none"> <li>Thermal Stabilizer Phases</li> <li>Stabilizer</li> <li>Compatibilizer and compatibilizer</li> </ul> |             |
| Material properties (This data are typical values and are not to be construed as product specifications.) |  |  |             |
| Basic Properties  | Unit   | Typical Value  | ASTM Method |
| Melt Index (300°C/ 2.16kg)  | g/10 min   | 18   | D1238       |
| Density   | g/cm <sup>3</sup>  | 0.960  | D1505       |
| Physical Properties @   |  |  |             |
| Flexural modulus  | (MPa)  | 1430   | D790        |
| Residual bend impact at 23°C  | (J/m)  | 28   | D256A/B     |
| Tensile strength at yield   | (MPa)  | 31.5   | D638        |
| Tensile strength at break   | (MPa)  | 29   | D638        |
| Ultimate elongation   | (%)  | 15   | D638        |
| Heat softening point  | (°C)   | 125  | D3121       |
| W.D.T   | (°C)   | 102  | D646        |
| Shore hardness D  | (Rc)   | 65   | D2240       |
| @ an compression moulded specimen obtained according to ASTM D 1828°C                                     |  |  |             |
| Fabrication conditions for injection moulding   |  |  |             |
| Recommended barrel temperature range between 290 and 280°C  |  |  |             |

<https://www.slideshare.net>

Quality control / lot to lot consistency



<http://www.freedigitalphotos.net>

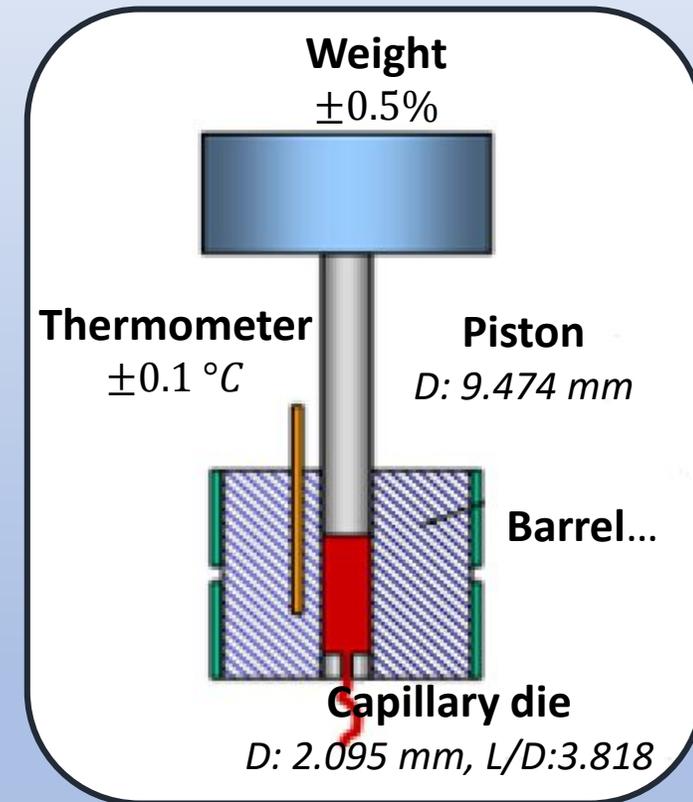
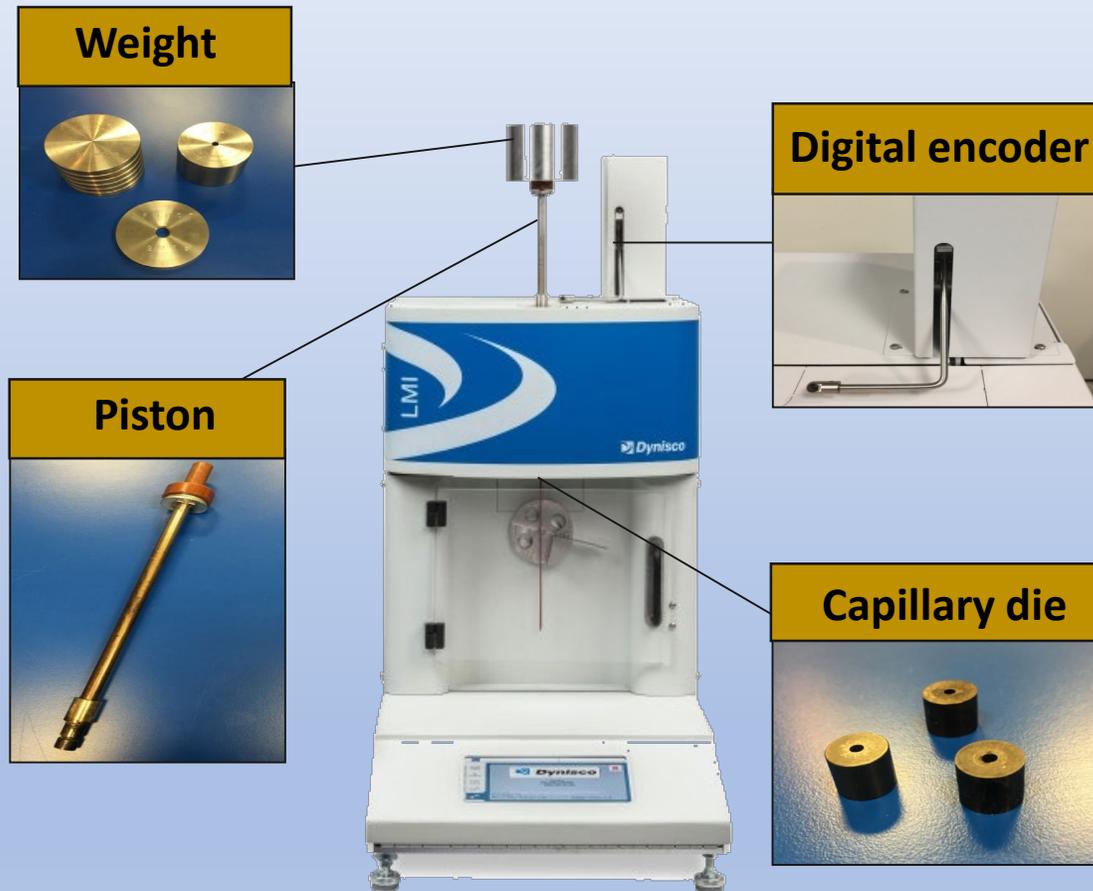
Minimizing waste



<http://westextrusion.com>

<https://home.plasticsnews.com>

# LMI Melt Flow Rate Tester



# Standardized Testing Condition

**ASTM D1238 - 13**

**TABLE X4.1 Suggested Test Conditions for Select Materials**

| Material   | Temperatures | Weights |
|--|--------------|---------|
| Acetals (copolymer and homopolymer)                      | 190          | 1.05    |
|  | 190          | 2.16    |
| Acrylics   | 230          | 1.2     |
|  | 230          | 3.8     |
| Acrylonitrile-butadienestyrene                           | 200          | 5.0     |
|  | 220          | 10      |
|  | 230          | 3.8     |
| Acrylonitrilebutadiene/styrene/polycarbonate blends      | 230          | 3.8     |
|  | 250          | 1.2     |
|  | 265          | 3.8     |
|  | 265          | 5.0     |
| Cellulose esters   | 190          | 0.325   |
|  | 190          | 2.16    |
|  | 190          | 21.6    |
|  | 210          | 2.16    |
| Ethylenechlorotrifluoroethylene copolymer                | 271.5        | 2.16    |
|  | 271.5        | 5.0     |
| Ethylene-tetrafluoroethylene copolymer                   | 297          | 5.0     |
| Polyamide  | 235          | 1.0     |
|  | 235          | 2.16    |
|  | 235          | 5.0     |
|  | 275          | 0.325   |
|  | 275          | 5.0     |
| Perfluoro(ethylenepropylene) copolymer                   | 372          | 2.16    |
| Perfluoroalkoxyalkane                                    | 372          | 5.0     |
| Polycaprolactone   | 80           | 2.16    |
|  | 125          | 2.16    |
| Polychlorotrifluoroethylene                              | 265          | 12.5    |
| Polyetheretharkatone (PEEK)                              | 400          | 2.16    |
| Polyether sulfone (PESU)                                 | 360          | 10      |
|  | 380          | 2.16    |
| Polyethylene   | 125          | 0.325   |
|  | 125          | 2.16    |
|  | 190          | 0.325   |
|  | 190          | 2.16    |
|  | 190          | 5       |
|  | 190          | 10      |
|  | 190          | 21.6    |
|  | 250          | 1.2     |
|  | 310          | 12.5    |
| Polycarbonate  | 300          | 1.2     |
| Polymonochlorotrifluoroethylene                          | 265          | 21.6    |
|  | 265          | 31.6    |
| Polypropylene  | 230          | 2.16    |
| Polyphenyl sulfone (PPSU)                                | 365          | 5.0     |
|  | 380          | 2.16    |
| Polystyrene  | 190          | 5.0     |
|  | 200          | 5.0     |
|  | 230          | 1.2     |
|  | 230          | 3.8     |
| Poly sulfone (PSU)                                       | 343          | 2.16    |
| Polyethylene terephthalate (PET)                         | 250          | 2.16    |
|  | 285          | 2.16    |
| Poly(vinyl acetate)                                      | 150          | 21.6    |
| Poly(vinyl chloride (PVC), rigid compound <sup>a</sup> ) | 190          | 21.6    |
| Poly(vinylidene fluoride)                                | 230          | 5.0     |
|  | 230          | 21.6    |
| Poly(phenylene sulfide)                                  | 315          | 5.0     |
| Styrene acrylonitrile                                    | 220          | 10      |
|  | 230          | 3.8     |
|  | 230          | 10      |
| Styrenic Thermoplastic Elastomer                         | 190          | 2.16    |
|  | 200          | 5.0     |
| Thermoplastic Elastomer-Ether-Ester                      | 190          | 2.16    |
|  | 220          | 2.16    |
|  | 230          | 2.16    |
|  | 240          | 2.16    |
|  | 250          | 2.16    |
| Thermoplastic elastomers (TEC)                           | 230          | 2.16    |
| Vinylidene fluoride copolymers <sup>a</sup>              | 120          | 5.0     |
|  | 120          | 21.6    |
|  | 230          | 2.16    |
|  | 230          | 5.0     |

## ASTM D1238 / ISO 1133 to specify:

- Testing conditions (temperature and weight)
- Testing time
- Sample mass
- Piston travel
- Die dimensions
- Precision

**TABLE 2 Standard Test Conditions, Sample Mass,<sup>A</sup> and Testing Time<sup>B,C</sup>**

| Flow Range, g/10 min | Suggested Mass of Sample In Cylinder, g | Time Interval, min | Factor for Obtaining Flow Rate in g/10 min |
|----------------------|---|--------------------|--|
| 0.15 to 1.0          | 2.5 to 3.0                              | 6.00               | 1.67                                       |
| >1.0 to 3.5          | 3.0 to 5.0                              | 3.00               | 3.33                                       |
| >3.5 to 10           | 4.0 to 8.0                              | 1.00               | 10.00                                      |
| >10 to 25            | 4.0 to 8.0                              | 0.50               | 20.00                                      |
| >25                  | 4.0 to 8.0                              | 0.25               | 40.00                                      |

**TABLE 5 Precision, Procedure B (Values in g/10 min)**

| Material      | Condition | Average | $S_w^A$ | $S_b^B$ | $I^C$  | $I_r^D$ | Number of Laboratories |
|---------------|-----------|---------|---------|---------|--------|---------|------------------------|
| Polyethylene  | 190/2.16  | 0.27    | 0.009   | 0.014   | 0.026  | 0.039   | 8                      |
| Polyethylene  | 190/2.16  | 0.40    | 0.016   | 0.027   | 0.045  | 0.076   | 8                      |
| Polyethylene  | 190/2.16  | 2.04    | 0.040   | 0.094   | 0.112  | 0.256   | 9                      |
| Polyethylene  | 190/2.16  | 43.7    | 0.997   | 1.924   | 2.819  | 5.443   | 8                      |
| Polypropylene | 230/2.16  | 2.25    | 0.052   | 0.214   | 0.1466 | 0.604   | 8                      |
| Polypropylene | 230/2.16  | 7.16    | 0.143   | 0.589   | 0.4051 | 1.666   | 8                      |
| Polypropylene | 230/2.16  | 32.6    | 0.693   | 0.945   | 1.959  | 2.672   | 8                      |
| Polystyrene   | 200/5     | 1.65    | 0.037   | 0.166   | 0.106  | 0.470   | 4                      |
| Polystyrene   | 200/5     | 8.39    | 0.144   | 0.423   | 0.406  | 1.197   | 4                      |
| Polystyrene   | 200/5     | 13.0    | 0.108   | 0.387   | 0.306  | 1.097   | 4                      |

<sup>A</sup>  $S_w$  = within-laboratory standard deviation of the average.  
<sup>B</sup>  $S_b$  = between-laboratories standard deviation of the average.  
<sup>C</sup>  $I$  = 2.83 $S_b$ , and  
<sup>D</sup>  $I_r$  = 2.83 $S_b$ .

# Method A

## Method A Manual Operation

- ❖ Mass measurement of extrudate collected over time
- ❖ Cut-n-weigh method
- ❖ Piston in proper position at end of pre-heating time
- ❖ Results:

$$MFR_{Method A} = \frac{10 M}{t_A}$$

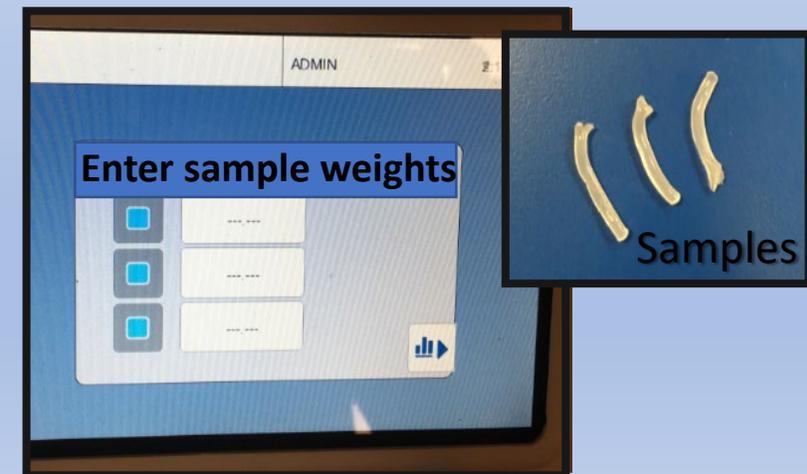
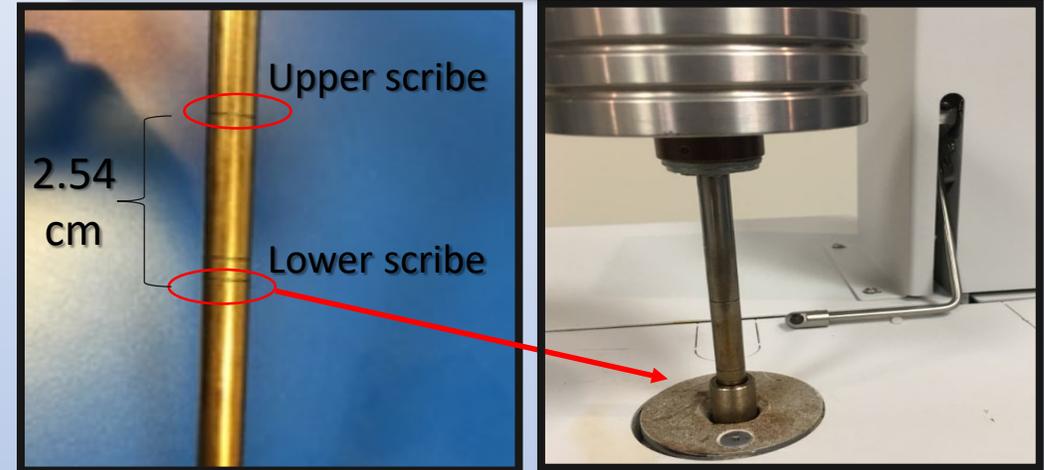
where

$MFR$  ( $g/_{10min}$ ): Melt flow rate

$M$  ( $g$ ): Extrudate mass

$t_A$  ( $min$ ): Cutting time

## Mass measurement for MFR



# Method B

## Method B

### Automatically Timed Flow Rate Measurement

- ❖ Volumetric displacement of polymer melt over time
- ❖ No cutting and weighing (simpler method)
- ❖ More precise for routine analysis
- ❖ Piston in proper position at end of pre-heating time to activate calibrate encoder
- ❖ Results:

$$MVR = \frac{10\pi R^2 L}{t_B}$$

where

$MVR$  ( $cm^3/_{10min}$ ): Melt volume – flow rate

$R$  (cm): Barrel radius (0.477 cm)

$L$  (cm): Length of piston travel (0.635 cm)

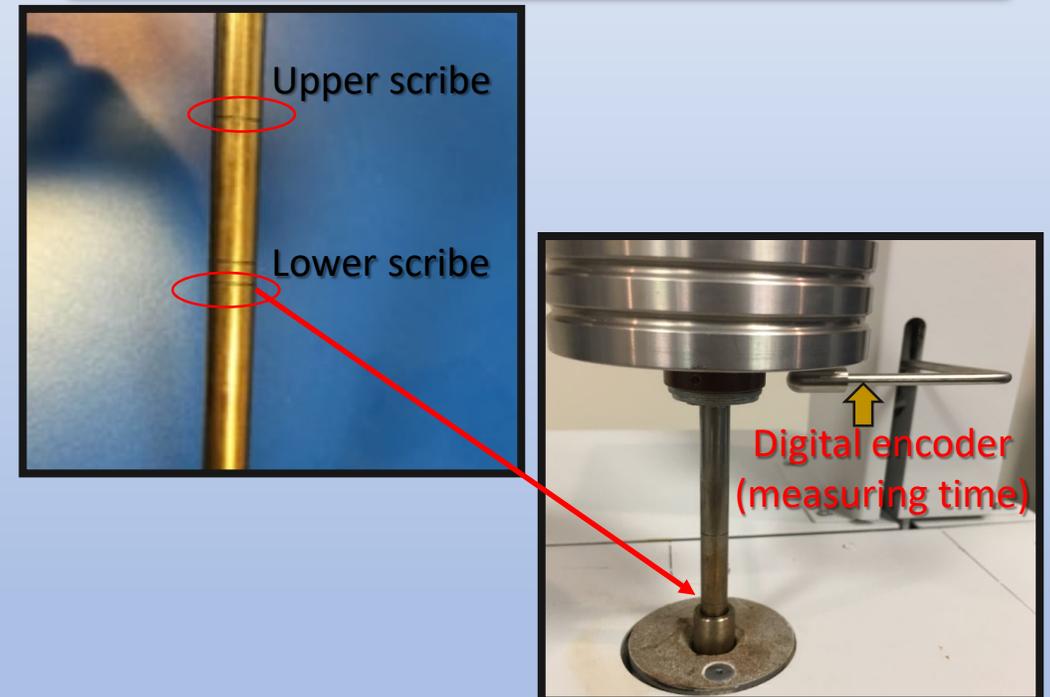
$t_B$  (min): Piston travel time

$$MFR = MVR \times \rho_m$$

where

$\rho_m$  ( $g/cm^3$ ): Mlet density at test temperature

### Volumetric displacement measurements for MVR



# Method A to B Conversion

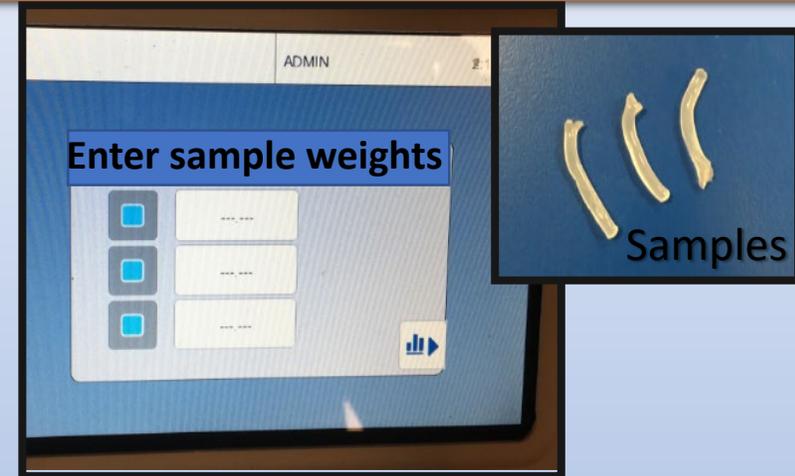
## Method A/B

### For calculation of apparent melt density

- ❖ Measuring melt mass-flow rate (*MFR*) and melt volume-flow rate (*MVR*) on the same charge of sample
- ❖ The ratio of the two values is a measure of the melt density of the polymer in  $g/cm^3$
- ❖ Piston in proper position at end of pre-heating time to activate calibrate encoder
- ❖ Results:

$$\text{Melt density } (\rho_m) = \frac{MFR}{MVR}$$

## Mass measurement for MFR



## Volumetric displacement measurements for MVR



# Method C & Method D

## Method C

### Automatically Timed Flow Rate Measurement for High Flow Rate Polyolefins Using “Half” Die

- ❖ Using a modified die (D: 1.048 mm, L/D: 3.818)
- ❖ For testing POs with a MFR of 75 or greater
- ❖ Improve the reproducibility of results by reducing the flow rate
- ❖ procedures same as Method B with 2.540 cm length of piston travel

## Method D

### Multi-Weight Using Automatically Timed Flow Rate Measurements

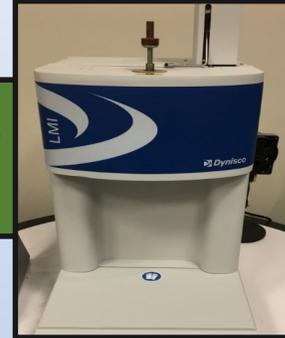
- ❖ Flow Rate Ratio (FRR) test
- ❖ Using two/three different test loads on one charge of material
- ❖ Results:

$$FRR = \frac{MFR_{Higher\ test\ load}}{MFR_{Lower\ test\ load}}$$

- ❖ For comparison of MWD (higher FRR means broader MWD)

# Operation Points

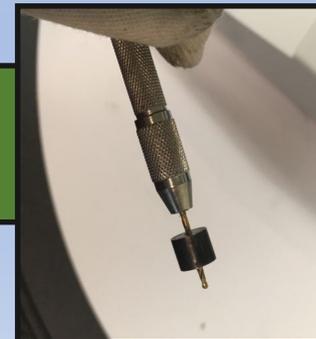
- ❖ Stabilizing the temperature of barrel with piston and die in place for ~ 15 min prior to testing



- ❖ Swabbing out the barrel using cotton patches and barrel cleaning tool



- ❖ Cleaning the die using a proper die cleaning tool



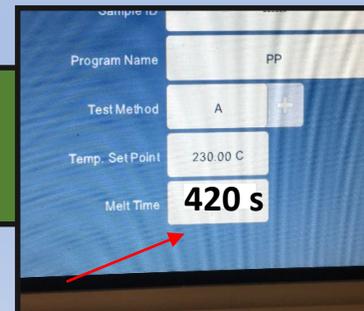
# Operation Points

- ❖ Using a die plug for materials with very high MFR



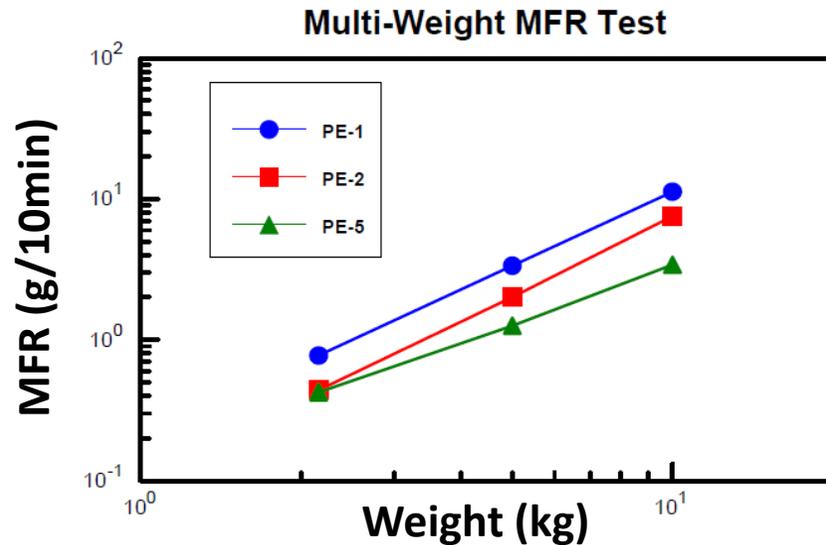
- ❖ Properly drying the hygroscopic samples (e.g. PET, PA, PC, PU, PBT, PEEK, ABS)

- ❖ Sufficient melting time (7 minutes)



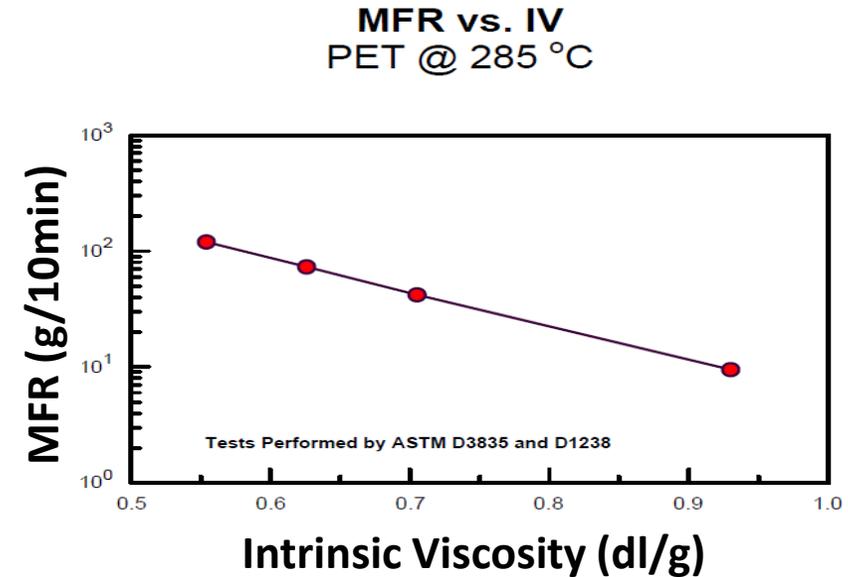
# Other Applications

## Shear thinning of the polymer melt



- ❖ Measuring MFR using various weights
- ❖ Graph of MFR vs Weight
- ❖ Describing the polymer flow behavior
- ❖ Higher slope → more shear thinning
- ❖ For comparing the MWD

## Intrinsic/solution Viscosity of PET

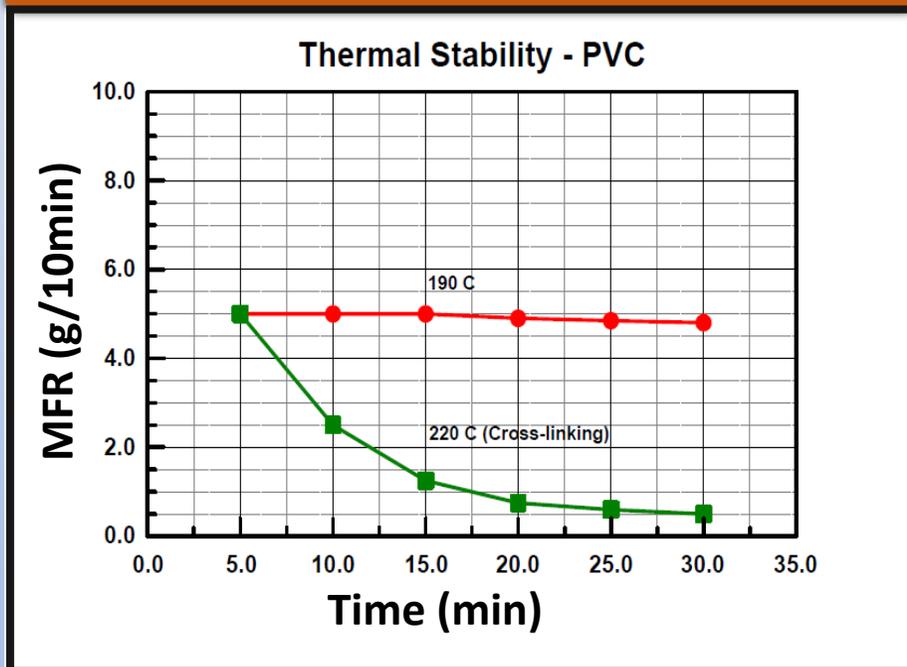


- ❖ Determining of IV of PET at 285 °C by melt rheometer (avoid using of noxious solvents)
- ❖ Both viscosity/MFR and IV are related to the polymer molecular weight<sup>1</sup>. So, they can be correlated to each other!

<sup>1</sup>Fox-Flory and Mark-Houwink relationships

# Other Applications

## Thermal stability



- ❖ resistance of polymer to a change in MFR at the test temperature over specific period of time
- ❖ Can show the presence of moisture or reactive chemicals in polymer.
- ❖ can measure the degradation rate or reactivity of polymer
- ❖ Repeating the test at various temperatures to give “processing window” of the polymer



“Everything Flows”-Heraclitus

<https://www.beautifulworld.com/north-america/united-states/the-wave/>

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**Thank You !**