SIMPLE AFFORDABLE, CONTINUOUS, ON-LINE VISCOSITY MEASUREMENT

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I) Abstract: Viscosity is a fundamental property of polymer flow. Capillary rheometers have been the standard measurement system for determining viscosity for many years. Attempts have been made to adapt these rheometers to on-line situations but their size, complex operation and cost have limited their use. A new on-line rheometer has been developed that is rugged, simple, and inexpensive. This paper shows how the rheometer works and reports test results from a major material supplier that define its consistency, accuracy, and precision.

II) Background: The advantages of measuring the polymer process are well known. In extrusion, for example, motor amps describe the energy required for melting. Pressure measurement indicates output stability. Melt temperature yields the total heat in the polymer. Viscosity—a broad measurement—describes quality.

While the importance of viscosity measurement is recognized, instruments to measure viscosity during processing have not seriously penetrated the market. The reasons why current on-line rheometers have failed to penetrate the market are:

> **1) Size**: On-line rheometers have been far too big. Instruments having the footprint of a small car will not find wide acceptance.
> **2) Expense:** Costly instruments find limited use. An engineering study should not be required to fit a rheometer to an extruder. A rheometer should adapt to the standard Dynisco pressure tap.
> **3) Complexity:** The more complex an instrument, the more likely it is to fail and the more difficult to operate.
> **4) Consistency Only:** Some instruments are only relative measurement devices.

Finally, some “on-line” rheometers are really, “off-line.” For example, when used with reactors, samples are transferred from the cooled product stream back to a small lab extruder. The samples are then remelted and pumped into the rheometer for measurement. Several samples per hour can be processed this way but the process does not measure the original process on-line. Further, the process suffers from a second heat history and a significant time delay.

With these problems in mind, the ViscoM rheometer was developed. The sensing instrument is roughly the size of a Thermos bottle; the instrument adapts to the standard Dynisco pressure hole; the ViscoM rheometer is ‘on-line’ with the process stream, it’s consistent, precise and accurate. The rheometer is affordable.

A few of the many reasons to measure viscosity during processing include:

> **A) Extruder Differences:** It is well known that two supposedly identical extruders behave differently with the same feed stock. Different screws in otherwise identical extruders change the viscosity. Extruder brands, barrel and screw wear, screw speed, screen packs, and extruder size all change the viscosity of the feed stock.
>
> This complicates extrusion enormously. We need to simplify.

B) Quality Control: If the viscosity changes, the quality of the product changes. Constant viscosity is the key measurement we need for quality control.

C) Profile Dies: Die flow is a function of viscosity. This is particularly important in profile dies because the dies are asymmetrical from the material's point of view. For example, a simple square hole die will not produce a square rod because the resistance at the corners is greater than in the middle of the die.

To make a square rod, the resistance to flow in the corners must be reduced to achieve a uniform exit velocity across the die *at a specific viscosity*. Operators need to know the viscosity so that they can retune the process to the die.

D) Mixing: It is well known that dispersive mixing is effective only if the viscosity is high. In twin screw extruders, for example, you need to know the viscosity before and after mixing elements to tune the process conditions and to evaluate the mixing element effectiveness.

E) Additives: Companies that make polymers add materials to the melt stream at many points. Each of these additives is likely to have an effect on viscosity that should be measured to insure consistent quality.

F) Automation: Truly efficient automation of the extrusion process will require a feedback loop to adjust the process variables to control the viscosity.

III) Common Types of Rheometers: A rheometer is a device that measures the resistance to flow of materials such as polymers. It is generally believed that, "only capillary and slit rheometers are suitable"[1] for the measurement of viscosity. Both may be adaptable as an on-line extrusion rheometer but neither has successfully penetrated the marketplace. A good summary of the pro's and con's of these and other rheometers as they apply to extrusion is given by Rauwendaal.[2]

A simple type of rheometer is a coaxial cylinder rheometer. In this rheometer (See Drawing 1), the annular space between the two cylinders is filled with a fluid. The inner cylinder is rotated and the fluid exerts a drag on the cylinder. The shear stress is obtained from the torque required to turn the cylinder. So, given the dimensions of the cylinders and the torque, the viscosity can be determined.[3] Normally, such rheometers are only used for low viscosity fluids but ViscoM has adapted the principle to its rheometer.

IV) Extrusion Rheometer: The geometry of the metering section of an extruder screw is like a coaxial cylinder rheometer (See Drawing 2). If you ignore the presence of the pumping flight, they are the same.

This is the basis of the ViscoM rheometer. See Figures 3 and 4.

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[1] Dealy, John M. Rheometers For Molten Plastics. New York, Van Nostrand Reinhold Company. 1982. P. 75.

[2] Rauwendaal, Chris. Polymer Extrusion. New York: Hanser Publishers. 1986. P. 193-204.

[3] Ibid. P 180-181.

V) Set Up And Test Results (Through Fig. 12): A rheometer must be evaluated in terms of the variables that could affect its performance. In this case, the variables that might affect its performance are pressure (flow) at the inlet, temperature changes at the inlet, time, and vibration from the supporting extruder.

A ViscoM rheometer was installed in a Dynisco pressure transducer hole in the the transfer pipe following a 3/4 inch Kayeness extruder. The extruder generated a process stream that supplied both the ViscoM rheometer and the Kayeness rheometer. This enabled simultaneous measurement of the process stream by the ViscoM and Kayeness so that the values could be compared.

A) Pressure Sensitivity: Like any metering section of an extruder, the ViscoM screw has a dual identity. It is a pump that is restrictive to flow. As a pump, the output is very low—roughly 1 gram per hour per RPM maximum. Because it is a shallow channel, it is resistive to flow. For example, if the screw is stopped, flow decreases to a very small fraction of the normal output.

Flow is a function of pressure. Because a process stream from an extruder, compounder, or other source may vary in pressure, it is important that a rheometer not be affected by pressure. By raising the flow from the extruder that supplies material to the ViscoM rheometer, the sensitivity to pressure can be evaluated. Dowlex was studied at 230C and 280C at extruder speeds between 40 and 80 RPM.

See Figure 5.

There was no discernible effect of pressure during the study of Dowlex polyethylene.

B) Temperature: The next variable to be evaluated is temperature. A process stream from an extruder, compounder or other source may vary in temperature. Since viscosity is specified at a particular temperature, it is important to understand the sensitivity of the rheometer to temperature.

The ViscoM rheometer has two temperature control zones for the barrel and the die. The barrel is, therefore, controlled independently of the process stream. Because the channel depth is so small (typically 0.01 to 0.014 inches), energy transfer is fairly fast. This means that the input temperature can be changed by the rheometer to standard conditions.

The effect of process stream temperature can be seen in Figure 6. At 230°C and short residence times, the apparent viscosity from the ViscoM decreases slightly with increasing temperature. At 280°C, variations in temperature have no apparent effect. That higher temperatures had a smaller effect than lower temperatures is a somewhat surprising result.

C) Precision: Time is another variable to be considered. A rheometer may have to be started and stopped due to upsets in the process stream. How consistent the readings are over a period of time is important. The ViscoM rheometer was started and stopped on the same day and on different days as shown in Figure 7. The ViscoM shows a relative standard deviation of 3 - 4% at 1000 - 2000 Pa•s. Some of the variability is likely to be from the extruder. This is quite adequate for many olefin applications.

D) Vibration/Lateral Force: Because the rheometer is mounted in a process stream, there is a concern about both vibration and lateral force applied to the rheometer. Lateral forces should not exist if the setup is properly done. Vibration of the primary melt stream might be a concern. An orbital belt sander, with the belt removed, was held against the ViscoM rheometer.

The results are shown in Figure 8. The average torque and torque variability increase with vibration and with applied lateral force. Readings quickly returned to original range after removing vibration and lateral force.

It is well known that applied lateral forces, in the absence of vibration, affect the torque reading and, therefore, the viscosity reading. This is because, when the housing is pushed, the screw sensor bends. This bending invariable increases the torque as the screw bends.

Because the experiment as a lateral force and a vibrational force at the same time, it is not clear what the contribution of vibration is. However, it may be telling that the readings all increase in torque. One might expect vibration to be neutral.

E) Accuracy: Because a screw sensor is a cylinder, it is reasonable to ask how closely the viscometer mirrors known information. See Figure 9. The screw rheometer shows a bias at the lower shear rates but good agreement at 100 sec^{-1}. The lower shear rate bias could be the result of the transducer sensitivity or temperature difference. It is also possible that there is a variable contribution from the flight itself.

F) Versatility: Four unfilled materials were also tested. These included two grades of acetyl, nylon 6, and a thermoplastic polyester. The results are in general agreement with existing data. See Figure 10. Additional tests (without on line verification from the capillary rheometer) show agreement with published data for rigid PVC (Fig. 13). Tests with flexible PVC (Fig. 14) show the influence of temperature change to the rheometer. This test is useful to simulate results on a production line without influencing production. An interesting test of electrically conductive carbon black filled material (Fig. 15) showed a surprizing degree of descrimination between carbon loadings of as little as 0.9%. The same material shows the effect of decreasing screw speed (Fig. 16) on viscosity and shows a drop in viscosity correlated with visual evidence of degradation.

G) Torque Transducer: The torque transducer used for these tests was a general purpose transducer designed for high viscosity applications. It has a limited range and, as seen in Figure 11, should not be used below 5% of full scale. Other transducers are available for lower range of operation.

H) Residence Time: Residence time was calculated based on the throughput at 80 RPM screw speed (about 30 grams per hour) using the volume of the screw of about 0.54cc. As seen in Figure 11, residence is about 45 seconds at 80 RPM but can be considerably longer at slower speeds.

VI) Self Renewing Seal: The rheometer construction requires the use of a patented dynamic seal to prevent degradation as the polymer leaves the screw and enters the die. This can be seen in the Figure 12 where two side by side channels have different channel depths.

While there are many ways to make the conveying capacity of the channels different, a powerful variable is channel depth as seen in the following formulae:

$$\Delta P_{max} = \frac{6\pi \cos \theta_b \, N D_b \, \mu l}{\sin \theta \, H^2}$$

Where ΔP= pressure drop, dyne/cm.^{2}, H = channel depth in cm., θ = average helix angle in radians, θ_b= helix angle at barrel surface in radians, N = frequency of screw rotation in revolutions per sec., D_b = inside barrel diameter in cm, μ = viscosity in dyne-sec/cm^2, and l = axial distance, cm.

In this example, the leakage flow between the channels allows renewal of the material in the seal. Flow can be increased through the seal by linking the two channels to increase the flow to the shallow channel.

There will be a viscous drag associated with the material in the dynamic seal and the length cannot be known without knowing the pressure. However, very little pressure is produced because it has about a 60 mil diameter hole and a mere 30 grams per hour flow rate. Given that a 0.01 channel depth in the seal will make an extremely powerful pump, the axial fill length will be very short. As such, it will represent less than 5% of the total torque.

VII) Discussion Of Results: The test results show that the ViscoM rheometer is basically insensitive to process stream. Neither temperature nor pressure has a great effect on the measurement. The measurements themselves show a generally good correlation to standard capillary rheometer techniques. While the ViscoM has slight bias, it is consistent and this makes it reasonable to deal with. Further, it’s noteworthy that no correct factors were applied in this study. However, two corrections factors are being pursued:

A) Mathematical: The first is a mathematical treatment of the flights to reduce the rheometer to a coaxial cylinder viscometer.

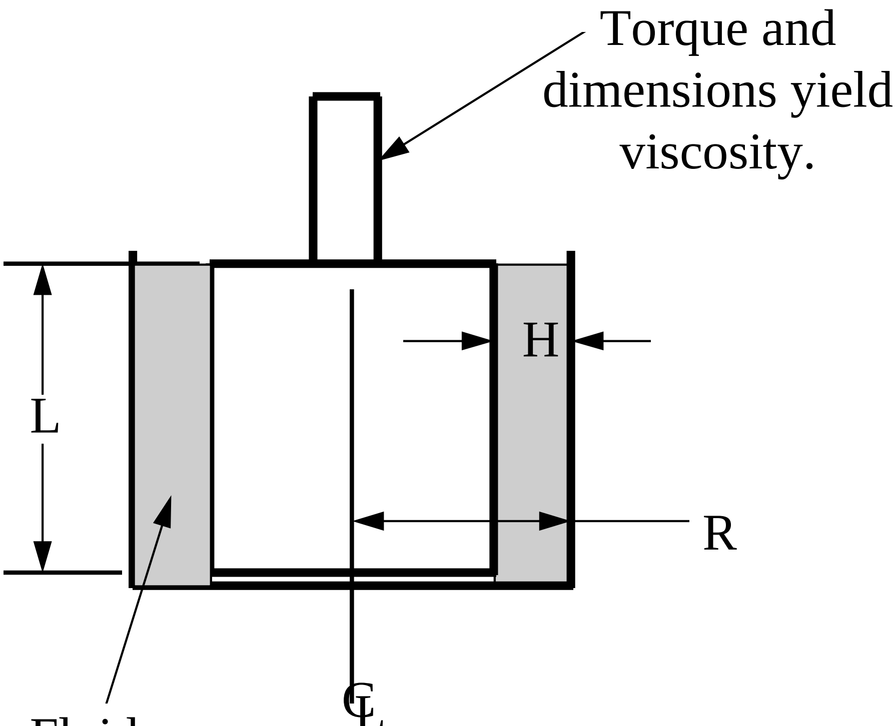
B) Experimental: The second correction is an experimental technique. Imagine that a fully flighted screw were tested. Imagine that this screw then had one half the flights removed. On subsequent testing under the same conditions, the difference in viscosity will be attributed to the flights. Multiplication will give the corrected viscosity. It is not hard to imagine two side by side rheometers making such a correction automatically.

The small size, easy installation and low cost opens up many applications that had not been previously considered. These include multiple local measurements in chemical plants, compounding, foams, and the traditional single screw and twin screw production extruder. Besides the materials in this study, the ViscoM rheometer has processed filled materials such as electrically conductive polyethylene, thermally sensitive materials such as rigid PVC. Where extremely corrosion materials must be processed, the barrels can be made from Hastalloy and the screw's nickel plated.

Because the ViscoM rheometer is an extruder, it enjoys all the advantages of the single screw extruder. These include simple operation, low wear in non filled applications, and low maintenance.

It is important to remember that the ViscoM rheometer will work best when all the requirements of the application are understood. If the requirements for shear rate, residence time, and viscosity range are known, then the proper torque sensor can be selected.

Figure 1:
Coaxial Cylinder Rheometer



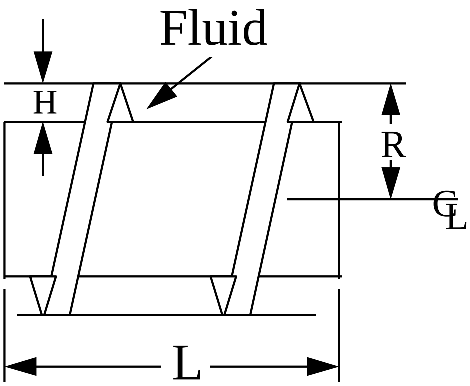


Figure 2:
Screw Rheometer

Figure 3
Rheometer Installed On 1/2 Inch
Randcastle Extruder

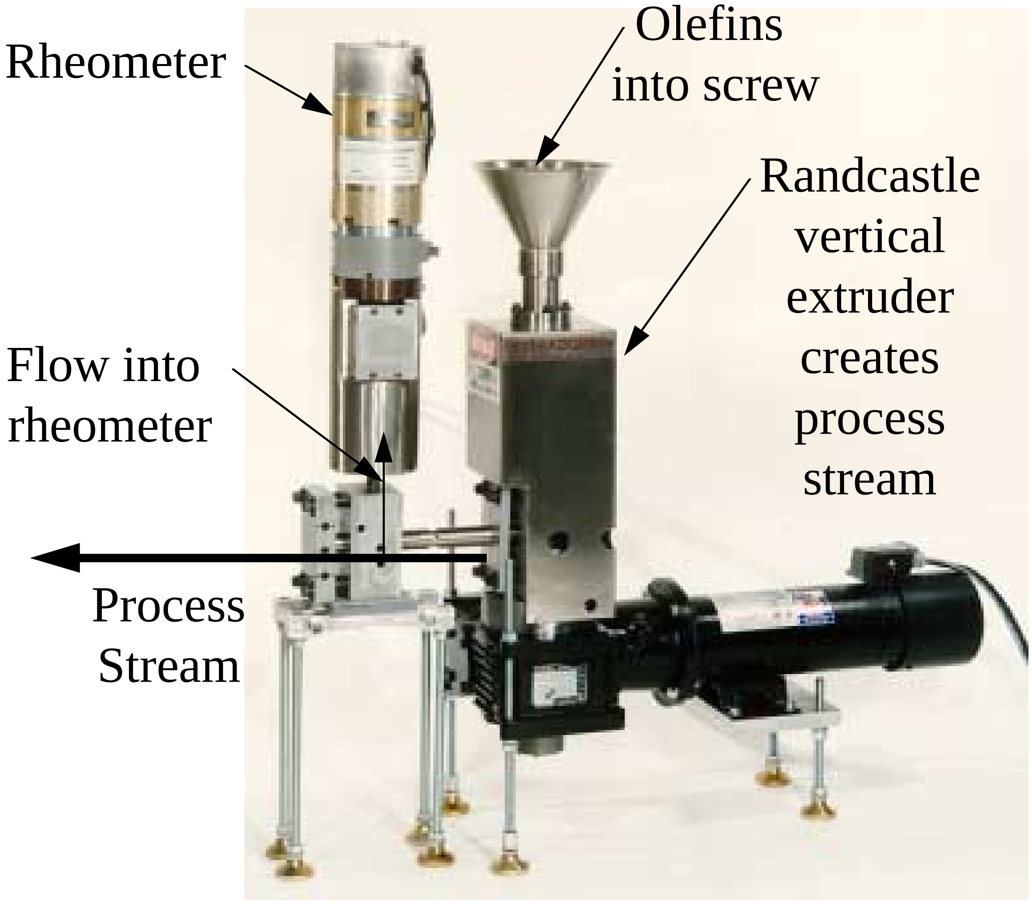
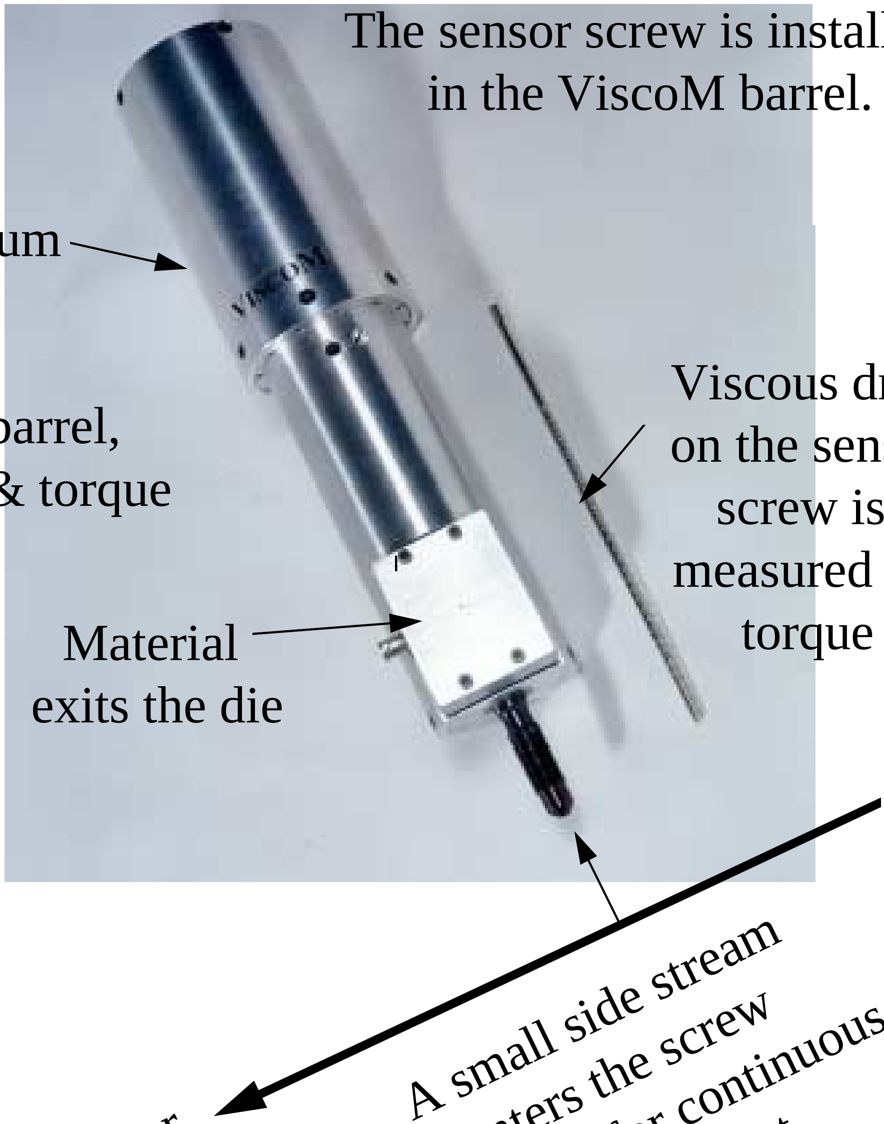


Figure 4

ViscoM Rheometer



The sensor screw is installed in the ViscoM barrel.

Aluminum housing protects motor, barrel, wiring & torque sensor.

Viscous drag on the sensor screw is measured as torque

Material exits the die

A small side stream enters the screw rheometer for continuous measurement.

The polymer process stream

Figure 5

Effect of Process Stream Pressure

Sensor RPM	5 RPM	10 RPM	20 RPM	30 RPM	Average	SD	RSD
40	2022	2052	2154	2154	2095	69	3.3%
50	1813	1867	1933	1936	1887	59	3.1%
60	1640	1704	1745	1776	1716	59	3.4%
70	1519	1589	1603	1597	1577	39	2.5%
80	1388	1470	1507	1480	1461	51	3.5%

	Viscosity (Pa.s)						
Sensor RPM	**5 RPM**	**10 RPM**	**20 RPM**	**30 RPM**	**Average**	**SD**	**RSD**
40	1274	1379	1359	1379	1348	50	3.7%
50	1159	1235	1240	1229	1216	38	3.2%
60	1073	1134	1121	1121	1112	27	2.4%
70	997	1065	1024	1047	1033	29	2.9%
80	930	986	986	966	967	27	2.7%

There is no apparent effect of process stream pressure on the VISCOM.

Figure 6

Effect of Process Stream Temperature

Sensor RPM					Average	SD	RSD
40	2049	2052	2049	2022	2043	14	0.7%
50	1982	1867	1816	1826	1873	76	4.1%
60	1736	1704	1674	1677	1698	29	1.7%
70	1719	1589	1533	1539	1595	87	5.4%
80	1562	1470	1439	1434	1476	59	4.0%

	Viscosity (Pa.s)					
Sensor RPM				**Average**	**SD**	**RSD**
40	1403	1379	1417	1400	19	1.4%
50	1254	1235	1265	1251	15	1.2%
60	1080	1134	1159	1125	41	3.6%
70	1057	1065	1061	1061	4	0.4%
80	981	986	1000	989	10	1.0%

At 230°C and short residence times, the apparent viscosity from the VISCOM decreases slightly with increasing temperature. At 280°C, variations in temperature have no apparent effect.

Figure 7

Precision

	Viscosity (Pas)								
Sensor RPM	**6/30/98**	**7/1/98**	**7/8/98**	**7/8/98**	**7/9/98**	**7/9/98**	**Average**	**SD**	**RSD**
40	2202	2178	2171	2100	2052	2052	2126	66	3.1%
50	1963	1961	1989	1946	1867	1862	1931	54	2.8%
60	1799	1781	1791	1729	1704	1695	1750	46	2.6%
70	1667	1630	1628	1622	1589	1550	1614	40	2.5%
80	1557	1526	1442	1533	1470	1444	1495	49	3.3%

Sensor RPM	**07/23/98**	**07/23/98**	**07/24/98**	**07/24/98**	**07/24/98**	**08/04/98**	**08/04/98**	**08/05/98**	**08/05/98**	**08/06/98**	**Average**	**SD**	**RSD**
40	1458	1434	1396	1369	1383	1359	1362	1505	1342	1379	1399	51	3.7%
50	1298	1300	1292	1229	1319	1205	1205	1328	1202	1235	1261	51	4.0%
60	1184	1187	1191	1109	1162	1114	1109	1150	1107	1134	1145	35	3.0%
70	1088	1094	1088	1016	1053	1034	1018	1053	1032	1065	1054	29	2.8%
80	1034	1008	1012	962	1005	974	957	991	971	986	990	25	2.5%

The relative precision of the VISCOM is ~3 - 4% at 1000 - 2000 Pa•s. Some of the variability is likely to be from the extruder that supplied the material.

Figure 8

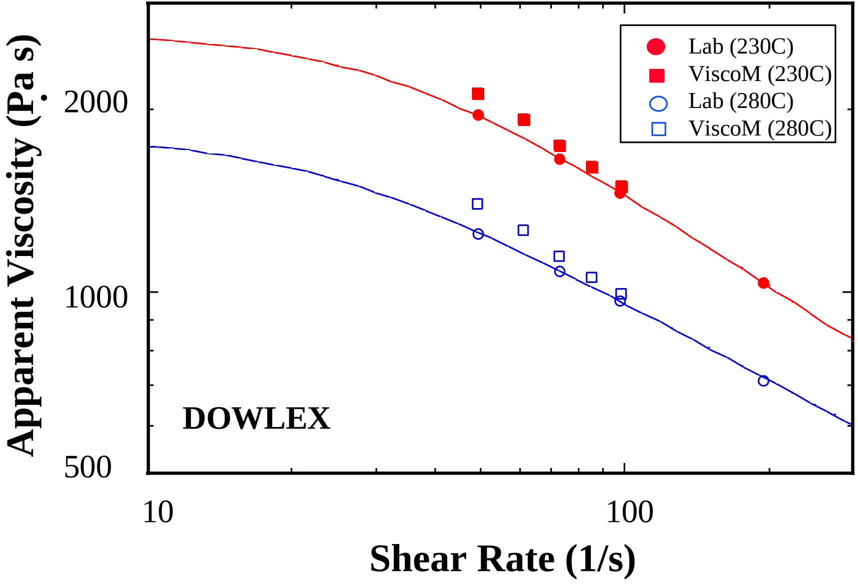
Effect of Vibrations/Lateral Force

	Normal					With Orbital Vibration				
RPM	**Low**	**High**	**Average**	**SD**	**RSD**	**Low**	**High**	**Average**	**SD**	**RSD**
50	4.52	4.56	4.54	0.03	0.62%	5.1	5.26	5.18	0.11	2.18%
60	4.84	4.98	4.91	0.10	2.02%	5.06	5.34	5.2	0.20	3.81%
70	5.34	5.46	5.4	0.08	1.57%	6.72	7	6.86	0.20	2.89%
80	5.66	5.8	5.73	0.10	1.73%	7.3	7.52	7.41	0.16	2.10%

Average torque and torque variability increase with vibration and with applied lateral force. Readings quickly returned to original range after removing vibration.

Figure 9

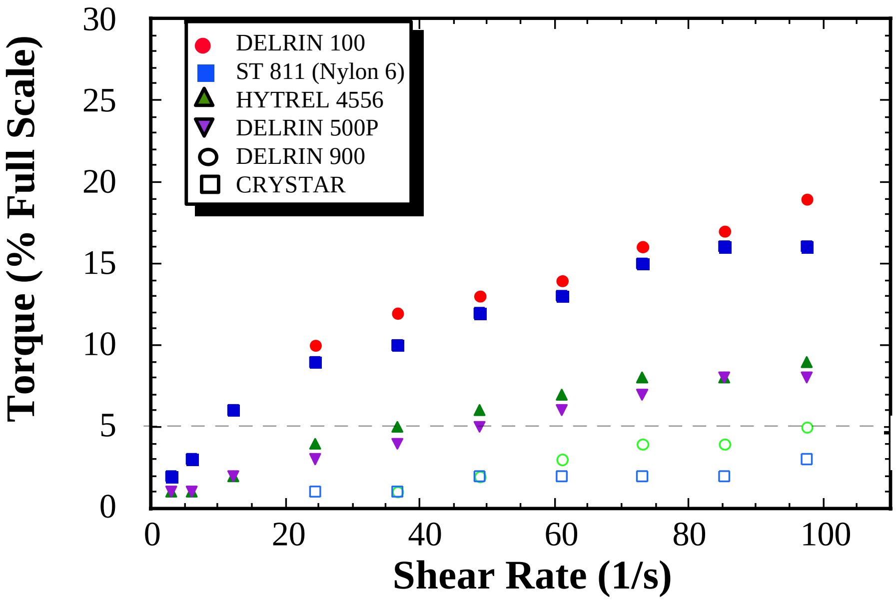
Accuracy



Good agreement between lab and VISCOM at 100 sec^{-1}. Bias at lower shear rates could be a result of transducer sensitivity or temperature differences.

Figure 10

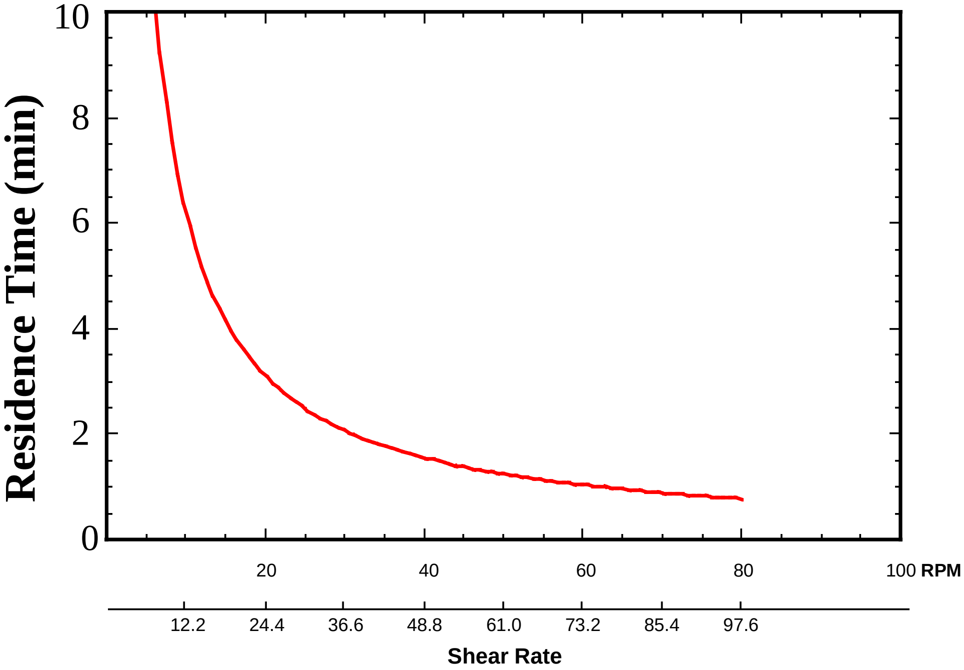
Torque Transducer



Shear rate/torque transducer must be sized correctly.

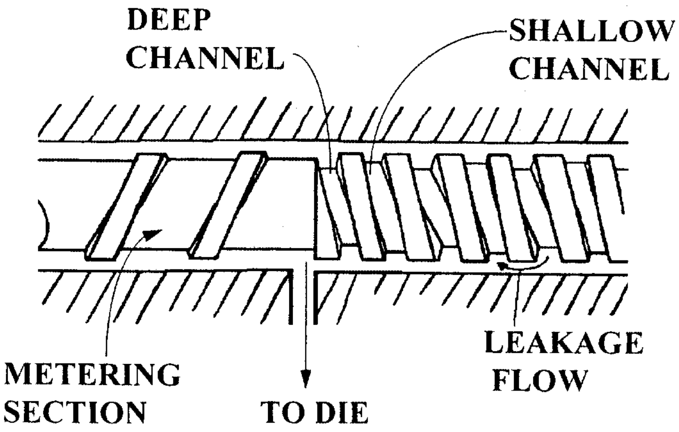
Figure 11

Residence Time



Screw should be designed for desired shear rate at
at short residence times.

Figure 12



The patented dual channel seal prevents degradation that could affect the torque.

Figure 13
Various Rigid PVC's Plotted

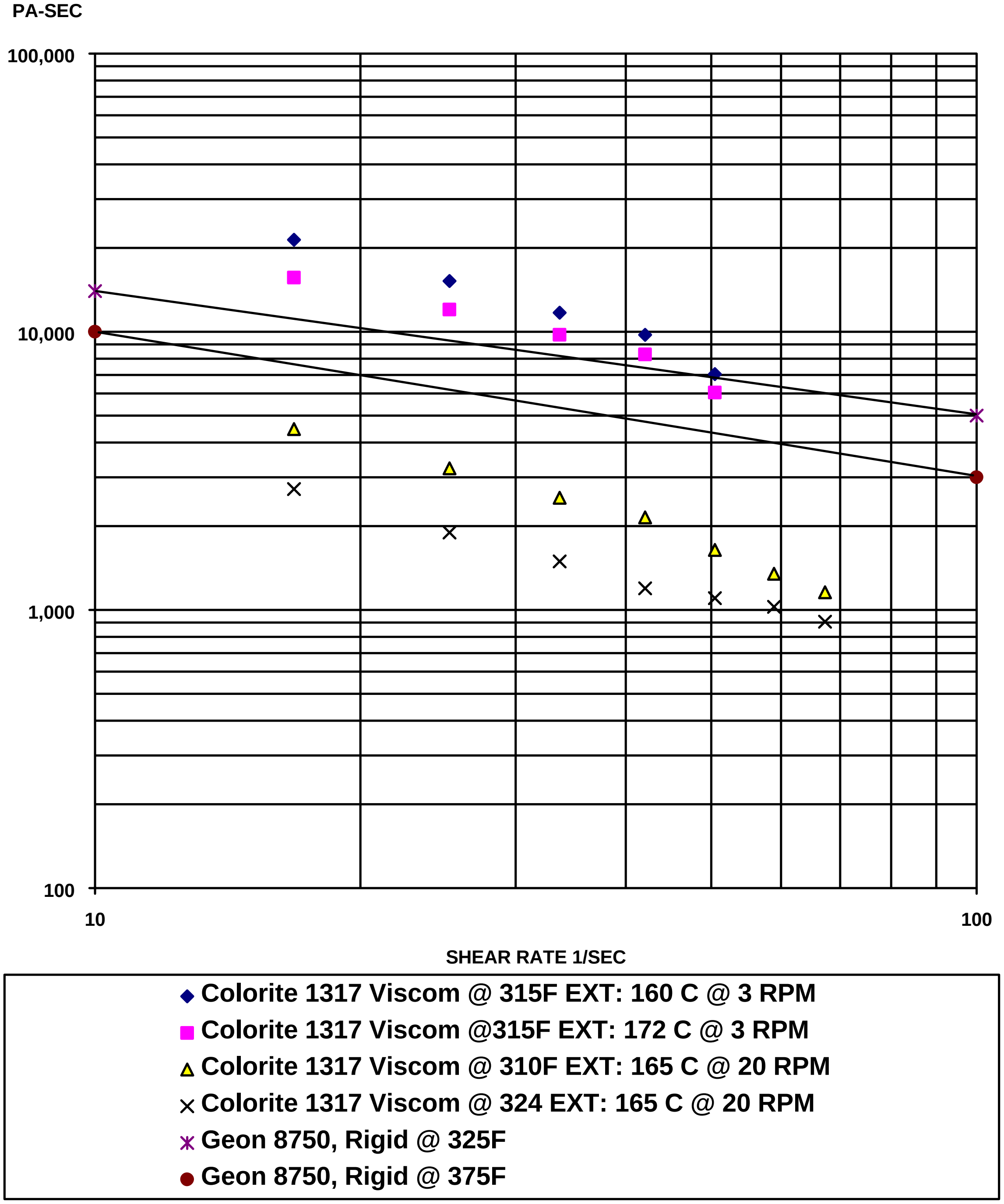


Figure 14: Viscosity VS Sensor Temperature

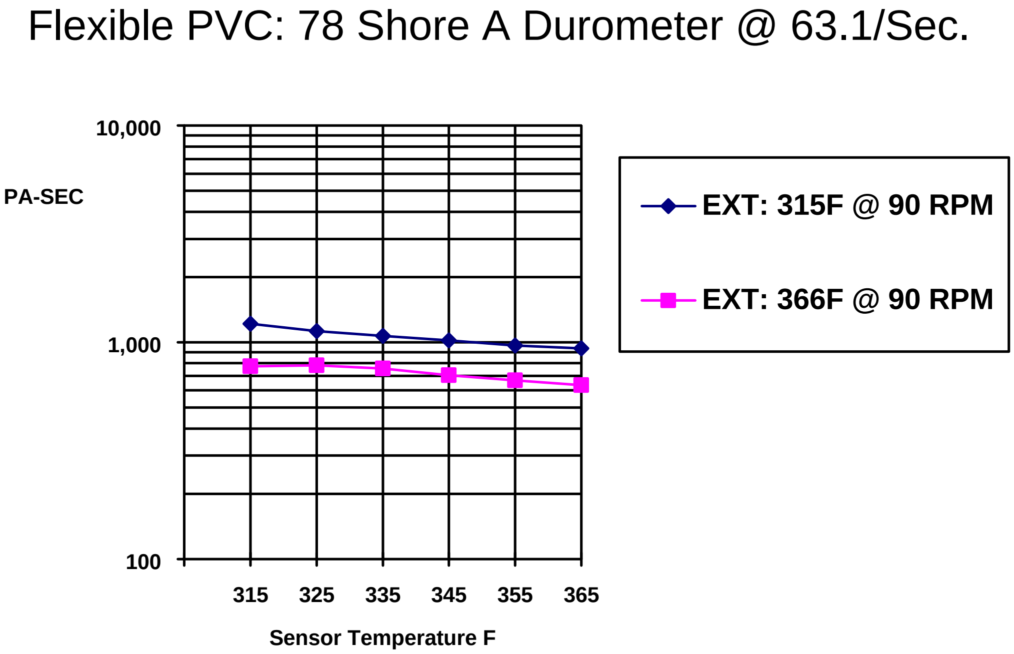
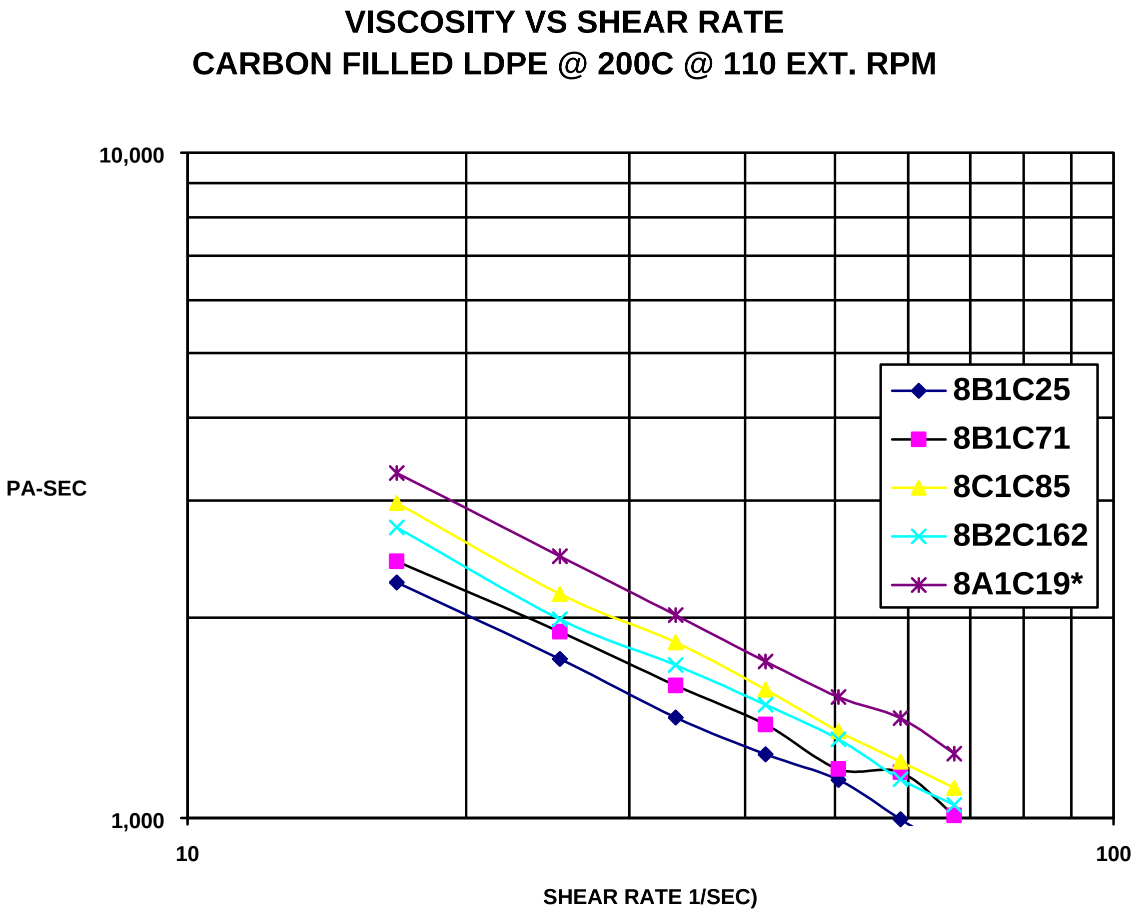

Flexible PVC: 78 Shore A Durometer @ 63.1/Sec.
PA-SEC
10,000
1,000
100
315
325
335
345
355
365
Sensor Temperature F
EXT: 315F @ 90 RPM
EXT: 366F @ 90 RPM

Figure 15

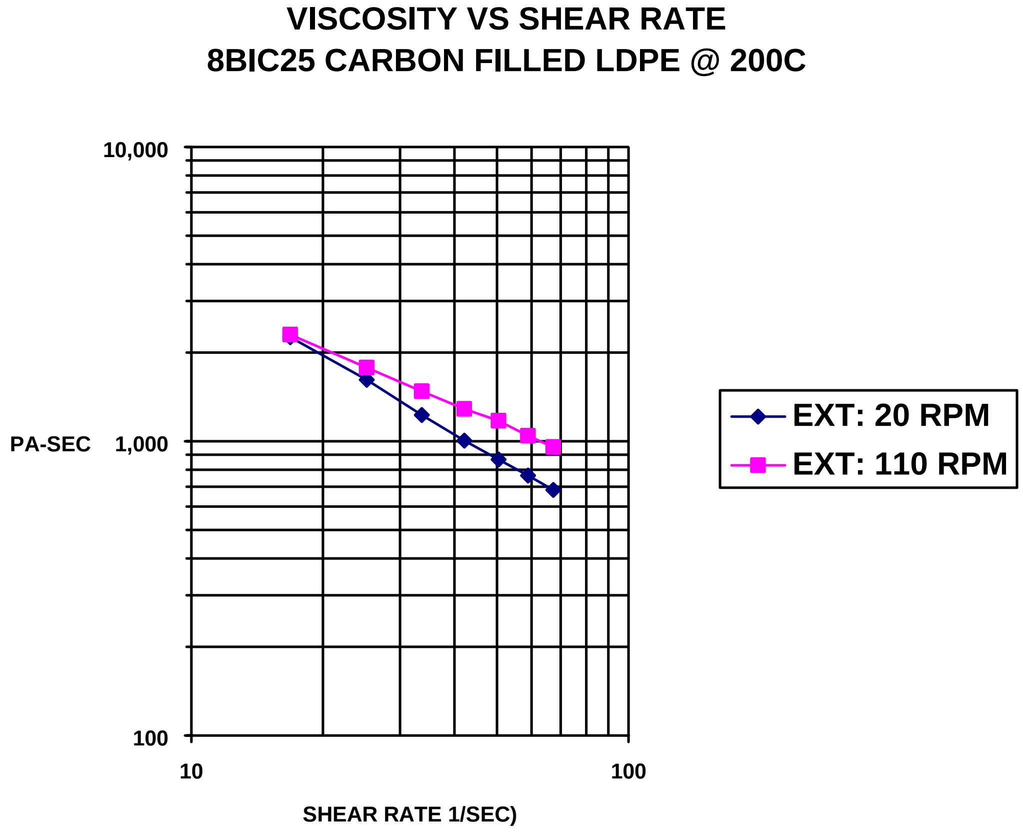

VISCOSITY VS SHEAR RATE
CARBON FILLED LDPE @ 200C @ 110 EXT. RPM

Viscom Sensor Speed RPM @ 200C		20	30	40	50	60	70	80
	1/SEC	16.82	25.23	33.65	42.1	50.47	58.9	67.29
8B1C25	PA-SEC	2,259	1,734	1,416	1,247	1,141	996	888
8B1C71		2430	1904	1581	1381	1185	1171	1009
8C1C85		2,972	2,171	1,836	1,558	1,350	1,215	1,110
8B2C162		2,732	1,989	1,697	1,480	1,313	1,143	1,047
8A1C19 *		3,303	2,474	2,018	1,719	1,520	1,412	1,249

Testing conducted blind. Percentages of carbon black by volume:

8B1C25:	29.0%
8B1C71:	30.9%
8B2C162:	33.6%
8C1C85:	34.5%
8A1C19:	35.8%

Figure 16



Viscom Sensor Speed RPM @ 200C	20	30	40	50	60	70	80

Avg. Inch Pounds Torque: Ext @ 20 RPM (PA-SEC)	7.79 2,259	8.38 1,614	8.49 1,229	8.67 1,005	8.98 867	9.25 765	9.43 683
Avg. Inch Pounds Torque: Ext @ 110 RPM (PA-SEC)	7.94 2,302	9.22 1,781	10.22 1,480	11.12 1,289	12.17 1,175	12.62 1,044	13.19 955

Viscom Sensor Speed RPM @ 200C	20	30	40	50	60	70	80

Note: At 20 RPM condition, the extrudate appearance was poor compared to 110 RPM showing less shine, roughness, and bubbles—probably indicating residence time degradation.

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