Engine Oil Viscometry and Viscometers – Fact and Fallacy

By Ted Selby, Savant Group – **Given at STLE Annual Meeting, Detroit, Michigan, USA, May 7, 2013**

Background

Viscosity has always been a critical property of lubricants. It is the source of the hydrodynamic lubrication that separates lubricated surfaces moving past one another under pressure and thus is a major factor in reducing wear. On the other hand, viscosity is also a form of friction – viscous friction – and, as it absorbs energy, viscosity reduces the efficiency of the engine even as it protects it.
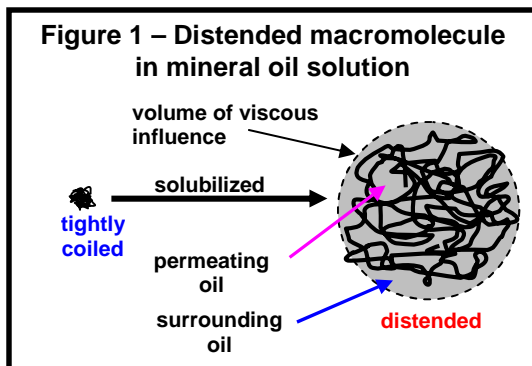
Newtonian Engine Oils

Since viscosity of engine oil is critical to its purpose as a lubricant, so too is the measurement of its viscosity in bench tests simulating the engine environment in which the oil must perform.

In earlier days of automotive history, measurement of engine oil viscosity was relatively simple. That was because, at a given temperature, engine oils had the same viscosity at any shear rate (i.e. rate of flow). Most commonly, viscometers were simple glass capillary tubes placed in a temperature-controlled liquid bath. Such oils were Newtonian and, as said, at a given temperature, an oil's viscosity was the same at all shear rates.

Non-Newtonian Engine Oils

Multi-Grade Engine Oils – However, a number of years ago, a concept was introduced to improve some engine oils' viscosity-temperature behavior over the temperature range of its usage from starting to operation temperatures by incorporating soluble polymeric macromolecules in the oil. These polymers, dissolved in lower viscosity base oils, swell to very large dimensions as shown in Figure 1 and brought the oil to the desired viscosity level at capillary viscometer shear rates. Since that time, many engine oils have been similarly formulated and have been called 'multi-grade' engine oils since they viscometrically span more than one SAE viscosity 'Grade'.
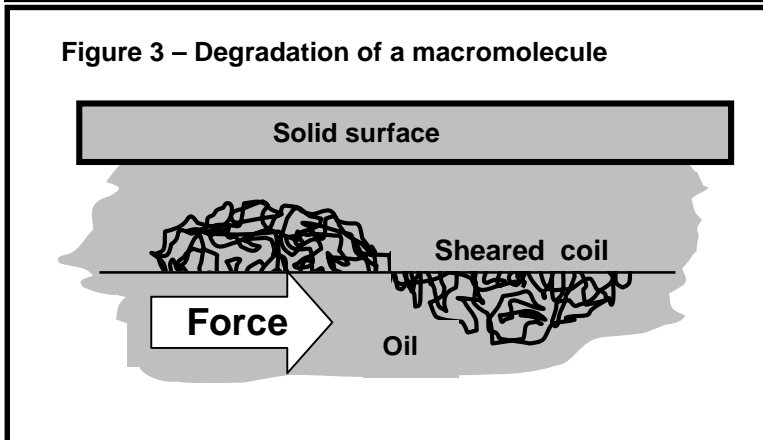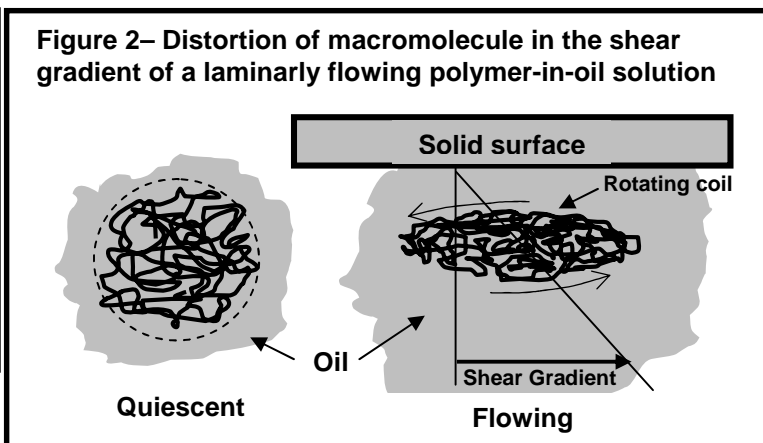

Figure 1 – Distended macromolecule in mineral oil solution

Figure 2– Distortion of macromolecule in the shear gradient of a laminarly flowing polymer-in-oil solution

Figure 3 – Degradation of a macromolecule

Temporary Viscosity Loss – Most multi-grade engine oils are non-Newtonian and decrease in viscosity with increase in the rate of oil flow or shear rate. This is because the 'viscous grip'[*] of the oil into which the dissolved and expanded polymers are 'imbedded', causes distortion and rotation of these macromolecules as shown in Figure 2.

The decrease in viscosity of these non-Newtonian oils is recovered when the shear rate is reduced and the phenomenon is thus called Temporary Viscosity Loss or TVL.

Permanent Viscosity Loss – However, if the oil into which the macromolecules are imbedded is **violently** sheared, some of the imbedded polymer molecules may be suddenly stretched beyond their ability to accommodate the motion of the oil and one or more of the macromolecular chemical bonds may be broken as in Figure 3 (exaggerated for understanding). Consequently, the viscosity of the oil-polymer solution decreases because the viscous contributions of the parts of the original macromolecule are less than the whole polymer molecule before degradation.

In contrast to TVL, this polymer-degradation phenomenon is called Permanent Viscosity Loss or PVL since, in this case, the viscosity lost is not recovered.

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[*] **'Viscous Grip'** is the author's terminology regarding the strength of the association of solvent oil and the dissolved polymer.

Confusion of TVL and PVL – Macromolecular <u>orientation</u> in high-shear-rate <u>laminar</u> flow of TVL shown in Figure 2 and the macromolecular <u>degradation</u> in <u>violent shear flow</u> of PVL shown in Figure 3, are distinctly different forms of viscosity loss. Although so different in their causes, they are easily confused as forms of viscosity loss. Actually, of these two forms of viscosity loss, only TVL is recoverably responsive to change in shear rate. However, if PVL has occurred, and depending on its severity, the degraded polymer will likely still have some TVL response to change in shear rate. In this case, however, a significant decrease in TVL will have been caused by PVL. Thus, it is evident that any device that produces PVL in the effort to measure TVL in multi-grade oil cannot distinguish between the two forms of viscosity loss of polymer-oil solutions.

This confusion has been experienced in a device erroneously introduced as an ultra-high shear rate viscometer. Fortunately, the error was recently corrected by a technical paper[1] showing that use of the instrument was actually producing PVL rather than measuring TVL and it was suggested in the paper that the PVL caused by the instrument might instead have use as a polymer shearing device for the evaluation of the PVL of engine oils.

Acceptable Viscometry for Non-Newtonian Engine Oil

Geometry of the Shearing Zone

Since viscosity of a non-Newtonian oil changes with shear rate (TVL), choice of the two common physical geometries for measuring viscosity – capillary and rotational rotor-in-stator geometries – favors rotational. Matched slightly tapered rotors in stators produce only one shear rate at each rotor-stator gap but many different gaps as the rotor is raised and lowered. In contrast, while capillary viscometry can be used at high shear rates, it can only give an 'averaged' value calculated from an assumed range of shear rates across the capillary[2].

With this in mind, the information following will not include capillary viscometry but only present results from rotational viscometry with the multiple shear rates desired available at different rotor-stator gaps and rotor speeds.

Important Factors

There are three critical factors required in non-Newtonian rotational viscometry:
1. Knowledge of shear rate,
2. Knowledge and control of the test oil temperature during viscometric measurement, and
3. Laminar flow and strict avoidance of rapid imposition of shear on a polymer-containing oil.

Rotational viscometers that are capable of providing all three of these factors are required for acceptable viscometry of both Newtonian and, most important, non-Newtonian fluids.

Shear Rate – If a rotational viscometer is claimed to operate at high shear rate, the device must either be
1. <u>proven</u> to be at the shear rate claimed <u>during operation</u>, or
2. <u>shown</u> to produce the viscosity of a known non-Newtonian reference oil at the high shear rate claimed.

Assumptions of shear rate from rotor-stator measurements are not acceptable support of a high shear rate claims for reasons of both expansion or contraction of the rotor-stator interface even when made of identical metals.

Temperature Control – The effect of shear rate on the polymer molecules dissolved and thus imbedded in multi-grade engine oils requires time for the heat energy generated by high shear rates to come to equilibrium and bring the polymer molecules to the expanded state appropriately possible at that temperature. During this time, the sample temperature must be precisely controlled to produce valid viscometric values.

Avoidance of Polymer Degradation – The dissolved and expanded polymer imbedded in the oil is resistant to degradation unless very strong shearing forces are suddenly imposed on the oil and the polymer is torn between two adjacent layers of the oil being forced to move at very dissimilar rates. In the engine, such conditions are primarily found in gears such as in oil pumps. PVL does not occur even at very high <u>laminar</u> shear rates since adjacent layers of the oil surrounding and permeating the polymer permits the flexible macromolecule to readily adjust its configuration.

Newtonian/Non-Newtonian Engine Oil Viscometry

High Shear Rate Rotational Viscometry

Tapered Bearing Simulator Background – In 1982 the first, high shear rate rotational viscometer called the Tapered Bearing Simulator (TBS) viscometer, was made available to laboratories[3]. It was an absolute viscometer that is one capable of determining its operating shear rate without use of reference oil[4]. This viscometer and two others developed somewhat later were used to further expand the SAE Viscosity Classification System, J300.

[1] Holtzinger, J., et al., **"New Method of Measuring Permanent Viscosity Loss of Polymer-Containing Lubricants"**, STLE, Tribology Transactions, 55:5, pp. 631-639, June, 2012.
[2] F. Girshick, **"Non-Newtonian Flow Dynamics in High Temperature High Shear Capillary Viscometers"**, SAE Paper 922288, 1992.
[3] T.W. Selby and D.A. Piasecki, **"Development and Performance of the Tapered Bearing Simulator for High Shear, High Temperature Viscometry"**, Proceedings of the 1982 International Conference on the Viscometry of Automotive Lubricants, pp. 37-47, 1982.
[4] T.W. Selby and D.A. Piasecki, **"The Tapered Bearing Simulator – An Absolute Viscometer"**, SAE Paper 83031, SAE International Congress & Exposition, Detroit, Michigan, February 28 –March 4, 1983
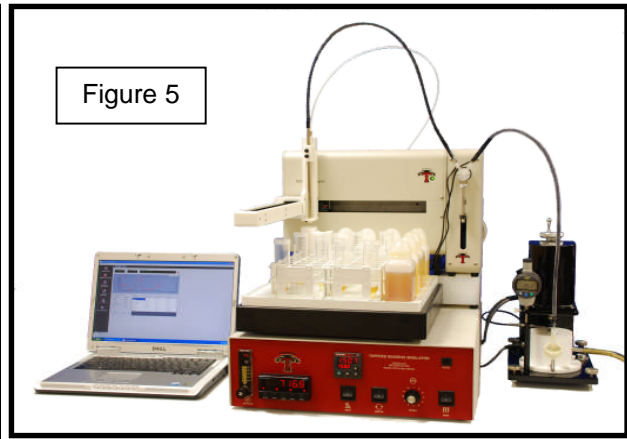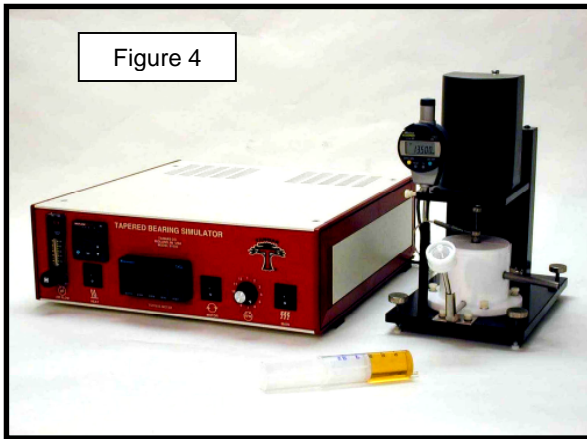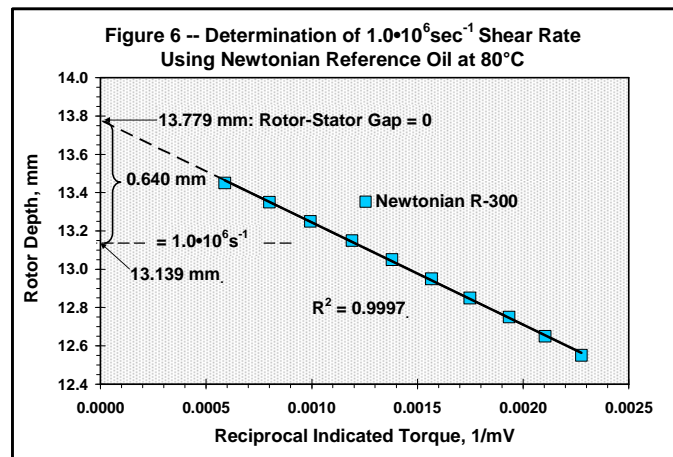
Figure 4

Figure 5

Figure 4 shows a manual TBS while Figure 5 shows a modern fully automated TBS viscometer.

***The TBS and the Three Factors of Viscometric Performance and Precision* –** It is of importance in understanding the following information to appraise the three critical factors involved in higher temperature, very high shear rate viscometry to see how these are met by the Tapered Bearing Simulator viscometer.
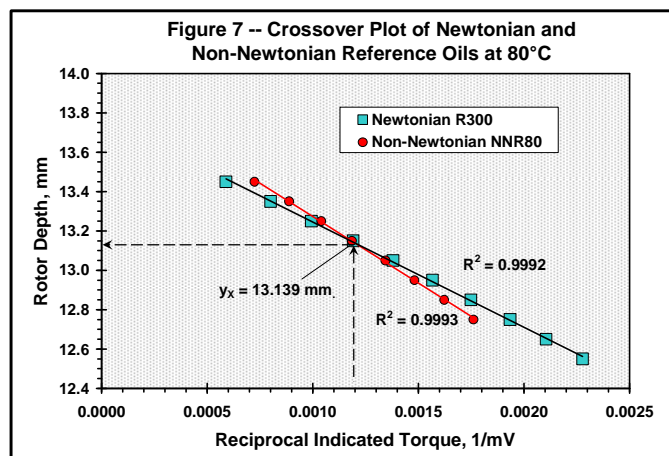
Precise Shear Rate by Absolute Viscometry – The Tapered Bearing Simulator has been shown to be an absolute viscometer[4]. This property permits the shear rate to be determined from the precisely measured slightly tapered rotor depth in the matching stator. The Newtonian equation providing this information is that, using a Newtonian reference oil, rotor depth is directly proportional to the reciprocal of the indicated torque required to spin the rotor. This relationship is shown in Figure 6 where a shear rate of one million reciprocal seconds is determined to be available at 13.139 mm in rotor depth while operating the TBS at 80°C. Other shear rates can be selected by exactly raising and lowering the rotor in the stator during the same test to give a range of viscosities of a non-Newtonian test oil.

It will be noted that the precision of the experimental data is quite good with a correlation coefficient of $R^2 = 0.9997$ – very close to a perfect value of $R^2 = 1.000$.

This procedure can be used at any temperature at which the TBS viscometer is operated.

Precise Shear Rate Using Non-Newtonian Reference Oils – Since the TBS can precisely set its operating shear rate, it can also establish that a non-Newtonian reference oils will have a given viscosity at a given temperature and shear rate. Then, using a Newtonian oil formulated to have the same viscosity at that temperature, this permits setting the rotor of the TBS viscometer at the depth at which the torque values of the Newtonian and non-Newtonian oils are the same thus giving the correct shear rate for operation of the viscometer. This approach is shown in Figure 7. The fully automatic TBS viscometer makes full use of these reference oil sets to maintain the precise rotor position during operation

Such Newtonian/non-Newtonian reference oils sets are presently available[5] for a shear rate of one million reciprocal seconds at temperatures of 80°, 100° and 150°C. Other temperatures and shear rates are available on request.

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[5] Newtonian and non-Newtonian reference oils are available from the Tannas Company, 4800 James Savage Rd., Midland, Michigan 48642, U.S.A. Telephone #: 1-989-496-2309 , E-mail address: ***tannas@savantgroup.com***

Precise Temperature Control – Since viscosity changes exponentially with temperature, the importance of precise temperature control is evident – especially with high shear rate analyses of non-Newtonian oils. Much effort went into the design of the temperature-control system for the TBS and other systems and these efforts have resulted int the grant of patents. Several techniques are employed all of which produce precision within a temperature range providing a variation of ±0.02 centiPoise.
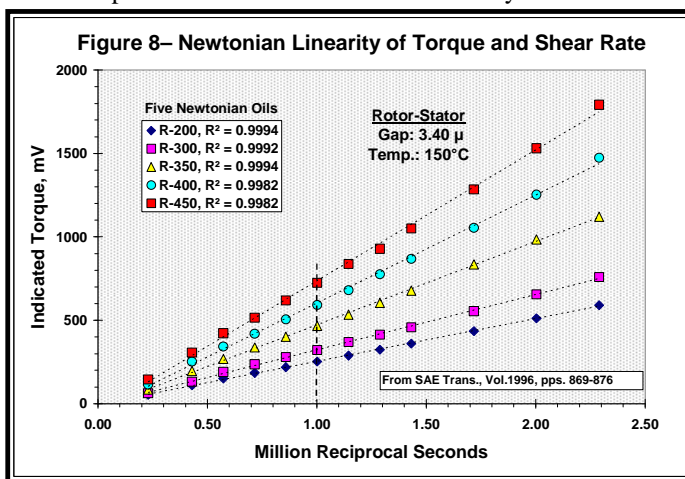
Laminar Flow and Avoidance of Polymer Degradation – For measurement of polymer-containing oils such as those used for multi-grade engine oils it is critical that the test lubricant always be subject to laminar flow in which the surrounding and permeating solvent oil is never subject to sudden, violent shear sufficient to rupture the relatively strong polymer molecular linkages. The flow conditions are thus conducive to the rotating mobility and adjustment of a macro-molecule's linkages without any being compromised.

The success of such viscometry in the TBS viscometer or any other high shear rate viscometer is readily shown by reversing the direction of increasing shear rate and showing that the return viscosity values are the same.

Precision of the TBS Viscometer – An interesting test of the precision of the TBS viscometer is by a multi-shear-rate viscometric measurement of a set of Newtonian oils at several speeds using the same rotor-stator gap. This is shown in Figure 8

The rotor-stator gap was set at 3.40 microns (a little less than $1/10^{th}$ the diameter of a human hair).
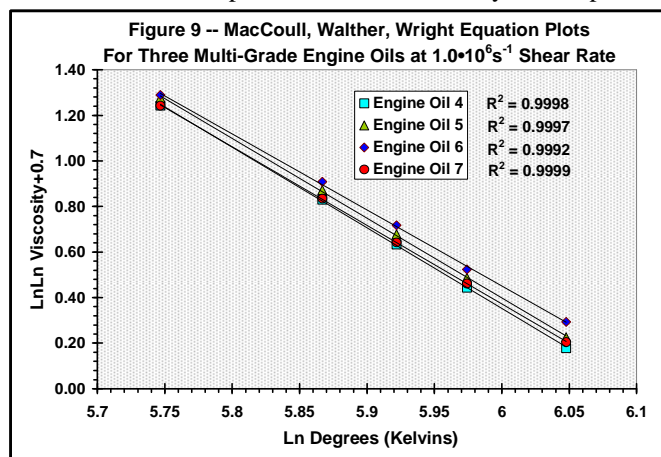
Five Newtonian reference oils were each subjected to 12 different rotational speeds producing known shear rates from $0.25 \cdot 10^6 s^{-1}$ to $2.4 \cdot 10^6 s^{-1}$. The torque values sensed by the rotor's load cell in millivolts at each speed for each oil is plotted versus the shear rate imposed. Each of the reference oils show a very linear relationship between the torque (proportional to shear stress) and the shear rate in support of

Figure 8– Newtonian Linearity of Torque and Shear Rate

Newton's equation. Moreover, each set of points extrapolate to a value near zero with Correlation Coefficients of R^2 of 0.998 or better.

Shear Rate Range of the TBS Viscometer – Studies have been made on the Tapered Bearing Simulator at very high shear rates. Data have been published[6,7,8] showing that shear rates up to 8 million reciprocal seconds are attainable. Precision of this range of shear rate was found to yield Correlation Coefficients of $R^2 = 0.994$ or higher with Newtonian reference oils.

Viscosity-Temperature Relationship at High Shear Rates – In the earlier portion of the last century, an empirical logarithmic equation was generated for accurately estimating kinematic viscosities of Newtonian base oils at temperatures between 100° and 210°F – the two commonly used temperatures of classifying oils[9,10,11] in those days. The equation and technique was later also shown to apply to the kinematic viscosities of non-Newtonian oils as well.

More recently, it was thought worthwhile to try to apply this equation to the viscosity-temperature behavior of non-Newtonian oils at high shear rates using the Tapered Bearing Simulator viscometer. As shown in Figure 9, the MacCoull, Walther, Wright equation also produces a very linear relationship over a temperature range from 40° to 150°C.

Figure 9 -- MacCoull, Walther, Wright Equation Plots For Three Multi-Grade Engine Oils at $1.0 \cdot 10^6 s^{-1}$ Shear Rate

[6] Selby, T.W. and Miiller, G.C., **"Very High Shear Rate Viscometry"**, International Tribology Conference, Yokahama, Japan, October, 31, 1995.

[7] Selby, T.W. and Miiller, G.C., **"Automotive Engine Oil and Very High Shear Rate Viscometry"**, 10th International Colloquium Ecological Conference – Solving Friction and Wear Problems, Esslingen, Germany, January 9-11, 1996.

[8] Miiller, G.C. and Selby, T.W., **"Progress in Super-Shear Viscometry"**, SAE International Spring Fuels & Lubricants Meeting, Dearborn, Michigan, U.S.A., May 6-8, 1996.

[9] MacCoull, N., Lubrication, Texas Co., New York, N.Y., U.S.A., p. 85, 1921.

[10] Walther, C., Erdöl und Teer, Vol. 4, p. 510 1928.

[11] Wright, W.A., Journal of Materials, Vol. 4, No. 1, p.19, 1969.

Conclusions

General Observations

Viscosity is a particularly important property of lubricants and thus a property broadly affecting the civilizations of the world, particularly in the application of automotive engine oils. It is also a deceptively simple-appearing property that can be readily recognized by sight and feel. Experience with Newtonian engine oils – which at a given temperature have the same viscosity at all shear rates – tends to play into a sense of simplicity for those technically interested in measuring and applying viscosity's benefits.

Temporary and Permanent Viscosity Losses

When lubricants such as engine oils are non-Newtonian as a consequence of containing dissolved polymeric molecules, such simplicity of viscometric measurement disappears. Meaningful viscometry must be responsive to the tendency of these flexible dissolved macromolecules to distort and orient in flow under the usual laminar flow conditions in an engine – a condition called temporary viscosity loss (TVL). In making such high shear rate measurements, the viscometer must create laminar flow conditions and impose precise control of temperature on the sample in order to obtain meaningful viscometric data.

However, in measuring TVL, if the instrument used produces a non-laminar, sudden, exceedingly strong shearing force, some of the polymeric macromolecules can be permanently degraded and the fluid lose a part of the polymer's viscous contribution. This permanent viscosity loss will become confused with the temporary viscosity loss normally associated with polymer-oil flow. Thus, the viscometry of non-Newtonian oils, such as multi-grade engine oils, requires viscometers capable of high shear rates, precise temperature control, and establishment and maintenance of laminar flow. The best instruments for such precise measurements are rotational viscometers providing a single shear rate at each setting of the rotor-stator gap when coupled with known shear rates and precisely controlled temperatures.

Fortunately, there are high-shear-rate viscometers proven to be capable of such sensitive measurements. As documented in this and other papers, one of these, the Tapered Bearing Simulator (TBS) viscometer, is also an absolute viscometer. As such, it also produces non-Newtonian calibration oils permitting other, relative viscometers to evaluate non-Newtonian lubricants.

Viscosity-Temperature Relationship of Non-Newtonian Engine Oils

Using the TBS viscometer, it has been found and reported that the empirical MacCoull, Walther, Wright equation which applies to Newtonian engine oils and base oils also applies to non-Newtonian oils at a shear rate of one million reciprocal seconds over a temperature range of 40° to 150°C. This is very helpful in anticipating the viscometric response of such oils at other temperature. To what degree this equation applies at other shear rates remains to be determined.