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## Standard Guide for In-Service Lubricant Viscosity Measurement<sup>1</sup>

This standard is issued under the fixed designation D8185; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

### 1. Scope\*

1.1 *Significance and Determination of Viscosity*—The purpose of this guide is to provide sufficient knowledge for a person with some technical background in lubrication or condition monitoring from which they can determine the best choice for measuring viscosity of an in-service oil. Such information from this guide should enable the user to engage in productive discussions with colleagues, service providers, managers, and service personnel about obtaining and using information on and from viscosity. There are a number of different approaches to viscometric measurement, and this guide is intended to be a helpful resource in selecting the most appropriate viscometric approach to gain information for the in-service fluid.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system are not necessarily exact equivalents; therefore, to ensure conformance with the standard, each system shall be used independently of the other, and values from the two systems shall not be combined.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

<sup>1</sup> This guide is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.96.01 on In-Service Lubricant Viscosity Testing Practices and Techniques.

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### 2. Referenced Documents

#### 2.1 ASTM Standards:<sup>2</sup>

- D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
- D446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers
- D2983 Test Method for Low-Temperature Viscosity of Automatic Transmission Fluids, Hydraulic Fluids, and Lubricants using a Rotational Viscometer
- D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
- D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
- D4378 Practice for In-Service Monitoring of Mineral Turbine Oils for Steam, Gas, and Combined Cycle Turbines
- D4683 Test Method for Measuring Viscosity of New and Used Engine Oils at High Shear Rate and High Temperature by Tapered Bearing Simulator Viscometer at 150 °C
- D5133 Test Method for Low Temperature, Low Shear Rate, Viscosity/Temperature Dependence of Lubricating Oils Using a Temperature-Scanning Technique
- D5293 Test Method for Apparent Viscosity of Engine Oils and Base Stocks Between –10 °C and –35 °C Using Cold-Cranking Simulator
- D5478 Test Methods for Viscosity of Materials by a Falling Needle Viscometer
- D6224 Practice for In-Service Monitoring of Lubricating Oil for Auxiliary Power Plant Equipment
- D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance
- D6304 Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

\*A Summary of Changes section appears at the end of this standard

- D6616 Test Method for Measuring Viscosity at High Shear Rate by Tapered Bearing Simulator Viscometer at 100 °C
- D6896 Test Method for Determination of Yield Stress and Apparent Viscosity of Used Engine Oils at Low Temperature
- D7042 Test Method for Dynamic Viscosity and Density of Liquids by Stabinger Viscometer (and the Calculation of Kinematic Viscosity)
- D7110 Test Method for Determining the Viscosity-Temperature Relationship of Used and Soot-Containing Engine Oils at Low Temperatures
- D7279 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids by Automated Houillon Viscometer
- D7483 Test Method for Determination of Dynamic Viscosity and Derived Kinematic Viscosity of Liquids by Oscillating Piston Viscometer
- D8092 Test Method for Field Determination of Kinematic Viscosity Using a Microchannel Viscometer
- 2.2 SAE AIR Standard:<sup>3</sup>
- SAE AIR 5704 Field Viscosity Test for Thickened Aircraft Anti-icing Fluids

### 3. Definitions and Terms

3.1 For definitions of terms used in this guide, refer to Terminology D4175.

#### 3.2 What is Viscosity?

3.2.1 Viscosity is commonly recognized as the ease or difficulty with which a fluid flows—that is, its fluidity. Often it is very evident that temperature has a strong effect on fluidity—viscosity always increases with decreasing temperature and vice versa.

3.2.2 A fluid’s viscosity arises from the degree of its internal molecular resistance to motion and a fluid flows only under sufficient force whether that force is gravity or some other source. Stirring, pumping, causing fluid to flow in a pipe or lubricating a machine are all examples of shear—applying a force to cause a fluid to move. For a simple example, consider filling a glass of water and a separate glass full of thick used oil, and stir each glassful at the same velocity (shear rate) with an identical spoon held in the same manner. The thicker used oil will require more force to move the spoon than the water, which is consistent with the used oil having a higher viscosity than the water.

3.2.3 Isaac Newton defined viscosity originally as the ratio of the force moving the fluid over the rate at which the fluid moves in response to that force. Fig. 1 helps to visualize this relationship. The edges of the two plates are shown with fluid between. As predicted from Newton’s law regarding viscous flow, when the upper plate is moved under a steady force over the stationary bottom plate, this produces a linear shear gradient through the fluid as shown. Depending on the viscosity of the fluid between the plates, the ratio of the force per area (technically named ‘shear-stress’ and indicated in Eq 1 by  $\tau$ ) causing motion of the upper plate at the shear gradient (termed shear rate and indicated by  $\delta u/\delta y$ ) is given by Newton’s law as:

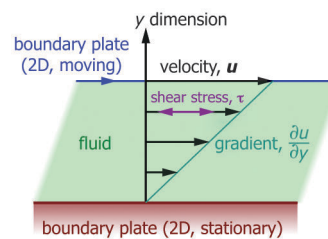


FIG. 1 Depiction of Velocity Gradient in a Flowing Fluid

$$\eta = \tau/\delta u/\delta y \tag{1}$$

3.2.4 Fig. 1 is, of course, a very simple example of fluid flow to clearly show the relationship in Eq 1. However, fluid flow can and does take many much more complex patterns of flow in the process of lubrication and hydraulic service. All of these patterns are expressed by Eq 1 if the fluid is Newtonian in behavior. However, many fluids and lubricants have flow patterns called “non-Newtonian” and these important lubricants will be discussed further on in this guide.

#### 3.3 Two Frequently Used Forms of Viscosity Measurement that Produce Different Values:

3.3.1 In the early years of measuring the viscosity of lubricants, most viscometric measurements of fluids were done using glass capillary viscometers, and these are still used widely today. However, in the mid-1930s, rotational viscometry was commercially introduced and has since become widely used, particularly in high shear rate viscometry, which was introduced particularly for viscometric information on non-Newtonian oils.

3.3.2 However, regarding information on Newtonian oils (which are non-shear rate susceptible), capillary and rotational viscometers—at the same temperature, produce different viscometric values for a very simple reason. The reason is that gravimetric viscometry is also a function of the density of the fluid that causes the fluid to flow through the capillary. Thus, the rate at which the fluid flows through the capillary is not only dependent on a fluid’s viscosity but also on its density. This form of viscometry has been termed “kinematic viscosity.” It was formerly measured and reported in units of centiStokes, cSt. This unit became obsolete in 1976 when worldwide System International, SI, unit of  $\text{mm}^2/\text{s}$  was introduced, (for information  $1.0 \text{ cSt} = 1.0 \text{ mm}^2/\text{s}$ ).

3.3.3 True viscosity is called “dynamic viscosity” and was formerly measured in the units of centipoise, cP. This unit became obsolete in 1976 when the worldwide System International, SI, the unit of  $\text{mPa}\cdot\text{s}$  was introduced, (for information  $1.0 \text{ cP} = 1.0 \text{ mPa}\cdot\text{s}$ ).

3.3.4 With Newtonian fluids, the two viscosity values differed from one another as shown by Eq 2 in which  $\nu$  is the kinematic viscosity,  $\eta$  is the true, dynamic viscosity and  $\rho$  is the fluid’s density at the temperature of measurement.

$$\nu = \eta/\rho \tag{2}$$

Both gravimetric capillary and rotational approaches to measuring viscosity have remained popular and, over time, other viscometric instruments have become available.

3.3.5 However, when selecting a viscosity-measuring technique for an in-service fluid or lubricant, it is critically

<sup>3</sup> Available from SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096, <http://www.sae.org>.

important to clearly understand which unit of measurement has previously been used to characterize its viscosity—that is, whether it is dynamic or kinematic viscosity. Conversion by density according to Eq 2 above may be required to achieve the desired unit of measurement. To repeat, viscosity data must be in identical units before comparison, otherwise incorrect conclusions and false expectations of responses to conditions may result in poor choice of lubricants with a waste of time, effort, and resources or may even result in choices harmful to the device using the fluid or those depending on it.

3.3.6 However, there is another very important factor in the measurement of viscosity, and that is whether the fluid is Newtonian or non-Newtonian and that subject is opened in the following section.

#### 3.4 Newtonian and Non-Newtonian Viscosity and Its Measurement:

3.4.1 As noted earlier, Newton’s definition of viscosity was given in Eq 1, in which the ratio of shear stress to shear rate is constant for a fluid. Many simple fluids such as mineral oils are Newtonian. However, with some more complex fluids or when certain additives are dissolved in some Newtonian fluids, the ratio of shear stress to shear rate is not constant and these fluids are called non-Newtonian. Today, many lubricating oils are non-Newtonian in behavior, and this fact also can affect their ability to lubricate or the energy required to overcome viscous resistance to flow.

3.4.2 Non-Newtonian flow may take many forms in comparison to Newtonian flow, two of which are shown in Fig. 2 compared to Newtonian flow. Shear-thickening by lubricants is rarely shown, however, shear-thinning is very commonly encountered especially in lubricants. This shear-thinning is often called “temporary viscosity loss” or TVL, since on lowering shear rate, the viscosity “lost” returns. The shear rates producing TVL are in the millions of units (called “reciprocal seconds,” denoted frequently by  $1/s$  or  $s^{-1}$ ). Such high values of shear rate are the normal operating level for most lubrication of automotive engines and other machinery. Temporary viscosity loss may reach levels of 30 % or more depending on the lubricant formulation.

3.4.3 The base mineral oils used to manufacture lubricants are essentially Newtonian. For many years, one of the important additives used to formulate lubricants have been so-called “viscosity index (VI) modifiers.” These additives are com-

posed of polymeric molecules which are dissolved in relatively small amounts in the mineral oil base stock to impart desirable viscometric properties at both low and operating temperatures at low shear rates.

3.4.4 Conventional wisdom has held that on dissolution, the volume of these VI modifiers increases greatly and thus impart an accompanying large viscosity increase to the base oil. The polymer volume increases when it is dissolved and is at higher temperature, thus giving a greater increase in viscosity than at lower temperature, but more recent investigations suggest that this phenomenon may occur only with certain polymeric structures, and that for different structures, there is a different mechanism. Using much lower viscosity base oils, this results in formulations that at low shear rates have viscosities similar to those of non-polymer containing lubricants at operating temperatures. However, as noted above, much of this viscosity increase is subject to TVL.

3.4.5 Unfortunately, there is another factor to be considered. At the shear rates and other, non-viscous forces applied in lubrication, the larger of these polymeric additives are vulnerable to mechanical rupture. This causes a significant and unrecoverable loss of lubricant viscosity, a so-called “permanent viscosity loss” or PVL, depending on how vulnerable the dissolved macromolecules are to the forces and conditions to which they are exposed during use. PVL may reach 20 % or more during use of the lubricant.

## 4. Measuring Viscosity

4.1 Some viscosity measurement techniques are performed at specific shear rates and temperature conditions and are designed to simulate the conditions of the device being lubricated, for example, the operating conditions of the lubricant serving an automotive engine. For the automotive engine, Test Method D5293, Test Method D4683, and Test Method D6616 are examples of viscosity measurement techniques that fix shear rates, shear-stresses and temperatures to obtain the resultant viscosity.

### 4.2 Measurement Consistency:

4.2.1 The precision of the measurement method should be well within that required for the changes that can occur with use of the lubricant, otherwise inadequate information may generate needless or erroneous time, effort, and expensive actions because of inadequate information from the viscometric test method chosen. Thus, before choosing a viscometric test, it is wise to review the repeatability,  $r$ , and the reproducibility,  $R$ , of viscosity measurement the particular viscometric method can provide.

4.2.2 As well, programs such as the ASTM Proficiency Testing Programs (PTP) for Petroleum Products and Lubricants can provide valuable insights into a particular viscometric method, such as how well the variability in reported values by the number of laboratories participating in the program for a given method, compares to the reported repeatability ( $r$ ) and reproducibility ( $R$ ) for the viscosity measurement method. Analysis of PTP data can be especially insightful for viscosity measurement methods included in the In-Service Oil Monitoring of Hydraulic Fluids/Oils PTP or the In-service Diesel Lubricating Oil Monitoring PTP. The test oils are actually

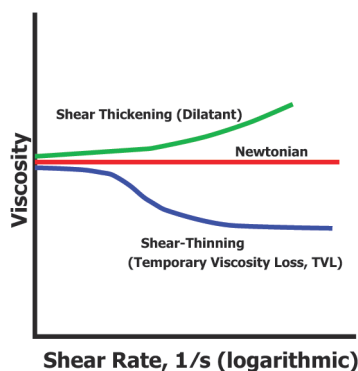


FIG. 2 Newtonian and Two Forms of Non-Newtonian Behavior

in-service oils and can provide insights on measurement variability applied to such in-service oils, rather than fresh oil samples. If variability information is not available, periodic redundant sampling may also provide insight into measurement method repeatability.

**4.3 Specifications and Warranty Testing**—Lubricants are often specified by their viscosity level at a given temperature. For example, in the case for engine oils, the Society of Engineers (SAE) viscosity grade of the fresh, unused oil defines the criteria by which they are identified—as SAE 20, SAE 10W-40, and others. The methods used to determine an SAE grade or lubrication specification can be applied to a used oil to determine if the oil is still within reasonable proximity to the recommended viscosity specification specified by the manufacturer of the equipment. Warranties may require the lubricant meet the requirements of a given specification or the warranty is subject to voiding.

**4.4 Establishing a Monitoring Plan for Viscosity Measurement:**

**4.4.1** Often, a series of in-service viscosity measurements are helpful and informative when taken in a systematic way. Such data can be of interest, for example in comparison to the initial fresh oil value. Or, depending on the equipment, if there is a minimum value below which the viscosity of the fluid should not fall or a maximum above which viscosity should not rise. Documenting a plan for an in-service series of viscosity measurements will likely also improve one's skill in good decision making regarding the information generated by the series.

**4.4.2** Information on specific measurement plans advice can also be found in the following ASTM methods:

**4.4.2.1** Practice **D6224** for monitoring of turbine oils,

**4.4.2.2** Practice **D4378** for mineral turbine oils, and

**4.4.2.3** Guide to ASTM Test Methods for the Analysis of Petroleum Products and Lubricants.<sup>4</sup>

**4.4.3** Measurement plans should be designed to ensure reasonably trouble-free operation of machines and equipment. An effective plan will often include several different pieces of information, starting with the technique to ensure a representative sample. The amount of sample should be sufficient for the planned slate of tests. If the fluid to be analyzed is well circulated in the mechanism, the sample can be taken at any site containing the circulating fluid—for example, the sump of an engine.

**4.4.4** In some cases, lubricant viscosity may be best analyzed under temperatures and shear rates similar to in-service conditions. Frequently, measurements are also made under conditions specified for new oils (for example, 40 °C or 100 °C at atmospheric pressure).

**4.4.5** Regardless of where the samples are obtained and under what conditions they are analyzed, consistent sampling location(s) are important for meaningful interpretation of the results. If multiple sample ports are desired, the location at which the sample is taken should be recorded with analysis values, together with any machine identification, and date and

time. In some cases, it is helpful to have the identification of the person collecting the sample as well.

**4.4.6** For samples likely to contain volatile components/contaminants, sample-containing vessels should be chosen which are readily and completely closable and thus suitable to consistently preserve the sample from changes that may occur between the time the sample is taken and the measurement performed. They should also be sized to hold adequate sample for the measurement method or methods being applied. The measurement plan should define the range of time allowed between sampling and the time a measurement should be made to minimize sample degradation and to provide actionable data as quickly as possible. Ideally this time should be as short as possible.

**4.4.7** The level of analysis at which action should be taken should also be clearly identified as part of the measurement plan, along with what actions should be taken, who should perform them, and the timeliness expected in completing them. In some cases, actions may require additional measurements of other properties. This should be taken into consideration when sizing the sample vessels. The most subjective factor of the sampling plan is the frequency of sampling. This will depend highly on the application, the particular oil type, and take into account the following:

**4.4.7.1** Cost of analyses,

**4.4.7.2** Expected sample degradation rate under normal conditions,

**4.4.7.3** The likelihood of equipment failure, and

**4.4.7.4** Potential cost of a failure if serious degradation is not detected fast enough.

**4.5 Obtaining a Representative Sample:**

**4.5.1** There are inherent limitations when performing any type of sampling, any one of which may affect the information obtained on the sample. For example, a small sample for determining the viscosity of the oil in a pipeline provides a sample from only one particular location. Therefore, when a representative sample cannot be obtained from a single port, a better approach is to collect samples from multiple locations.

**4.5.2** It is important to clearly understand that the objectives of sampling an oil or lubricant are to:

**4.5.2.1** Maintain data integrity,

**4.5.2.2** Maintain proper frequency of sampling,

**4.5.2.3** Set proper targets and alarms for each type of equipment, and

**4.5.2.4** Minimize data obstructions.

**4.5.3** Moreover, another area of planning involves strategic sampling conditions that include such matters as:

**4.5.3.1** Safety considerations,

**4.5.3.2** Sampling location, and

**4.5.3.3** Sampling tools.

(1) Hardware.

(2) Bottles.

(3) Sampling and procedure practices (Practice **D4057**).

**4.5.4** Under all circumstances, it is necessary to follow safe and well-planned sampling procedures for each type of equipment whether they be:

**4.5.4.1** From pressurized or non-pressurized systems.

**4.5.4.2** From small reservoirs (such as bearing housings).

<sup>4</sup> Nadkarni, K., *Guide to ASTM Test Methods for the Analysis of Petroleum Products and Lubricants*, 2007.

4.5.4.3 From a drum or reservoir without a test port.

4.5.4.4 From otherwise difficult or dangerous environments.

4.5.5 All sophisticated oil analysis tools, techniques, and diagnostic processes are meaningless if the oil sample fails to effectively represent the actual condition of the oil in service in the machine. Proper sampling procedures build the foundation of an effective oil analysis program. Without good sampling procedures that obtain a representative quantity of the fluid or lubricant in service, both time and money are wasted, and incorrect conclusions are likely if the data are faulty.

4.5.6 Another caution with sampling is the importance of consistency of the method and the analytical technique. It is not a good idea to trend sample results taken using different sampling methods or different test methods.

#### 4.6 *Temperature of the Viscometric Test and Measurement:*

4.6.1 Viscosity of an in-service oil will change significantly with temperature. Proper viscosity measurement requires the instrument or technique to control the temperature of the sample appropriately during measurement. Some ASTM methods are written for elevated temperature measurement, and others have been specifically developed to evaluate viscosity of the oil at low temperatures. ASTM methods typically indicate the required temperature control limits during the measurement. For some time, 40 °C, 100 °C, and 150 °C have been the three most common above-ambient temperatures at which viscosity data are collected and compared.

4.6.2 When performing different test methodologies, it is important to have an understanding that time to reach the temperature equilibrium at which measurements will be made differs with instrument and technique. For example, the kinematic capillary viscosity test, Test Method **D445** requires the use of clear liquid baths which may need 30 min or more to become constant depending on the temperature of measurement prior to performing a test run. As another example, with the low-temperature dynamic viscosity Test Method **D5133**, the test requires cooling at a rate of 1 °C per hour after stabilizing either the liquid bath or the refrigerated metal dry-bath at –5 °C. Not waiting sufficient time for equilibrium to take place will produce test results outside the expected repeatability/reproducibility stated in the respective test method and operators need to be aware that such poor practice will produce erroneous test results. Obviously, poor temperature control while taking a viscosity measurement will very likely lead to:

4.6.2.1 Inaccurate results,

4.6.2.2 Wrong conclusions, and

4.6.2.3 Poor reproducibility and poor consistency,

4.6.3 Checking the temperature measurements devices, such as the thermometer or PT-100, against a reference on a semi-annual basis (as a minimum) will help to ensure that the viscosity measurements being taken are accurate. Each instrument is likely to have specific procedures for calibrating the temperature measurement and temperature control devices. ASTM methods often detail the procedures for calibrating a device. A laboratory practicing a given form or forms of viscosity measurement should be able to demonstrate proficiency in the performance of the calibrations to the frequency

required by the ASTM method or provide a traceable certificate to the organization which provides the calibration services.

#### 4.7 *Turnaround Time Requirements for Results:*

4.7.1 Different viscosity measurement techniques can require different amounts of time to produce the data. **Table 1** provides insights on the elements that affect turnaround time for each of the viscosity measurement methods. Included in **Table 1** is a brief description of the viscometric measurement technique and the ASTM method number. The information in the *Typical Location* column indicates where the viscosity measurement is most likely to be executed if the nature of measurement is crucial enough to support investment in equipment and facilities to execute the method and personnel are trained to run the test method.

4.7.2 *Elements That Affect Turnaround* column summaries assume that the instrument is properly set-up, calibrated, and ready for sample testing. Such time to execute the test may be extended if calibration and reference procedures are not included unless required by the method to be completed for each test. Time estimates obviously do not include time to ship the samples to a laboratory nor do they include response time of the chosen laboratory if the measurement technique is not immediately available at or associated with the location at which the sample is obtained.

4.7.3 The rate at which data is collected must be balanced with the information that is required for the information desired. An in-service oil analysis plan that utilizes the most appropriate measurement technique and allows for the results to be turned around in the appropriate time will achieve the best results with the minimum delay in appropriate response to the data's information. Make sure that the laboratory you choose is qualified, ISO 9001 or 17025 certified, and experienced in running the test method.

4.8 *Availability of Certified Reference Fluids*—Reference fluids are an important tool that a laboratory must use to ensure that the viscosity measurement equipment and procedures of the technicians are both correct and consistent. When developing an in-service viscosity measurement program, it may be advantageous to understand the reference fluids available and how they can be used by a laboratory to ensure consistent results. Either the viscometer manufacturer or the ASTM committee responsible for viscosity reference material producer can give assistance with selection of appropriate reference materials for a particular application. Reference materials are generally classified as certified and non-certified. Here we will describe these and how they can be used.

#### 4.8.1 *Standard Viscosity Reference Fluids:*

4.8.1.1 Special standardized fluids are necessary for:

(1) Viscometer calibration.

(2) Verification of a viscometer's calibration or performance.

(3) Or in the case of non-Newtonian behavior, to appraise an instrument's response to certain test conditions.

4.8.1.2 Certified standard viscosity reference materials should be sourced from the instrument manufacturer, the ASTM or other group responsible for the viscometric test method, or an ISO 17025 or Guide 34 accredited laboratory

**TABLE 1 Elements Affecting Turnaround for Each Viscosity Measurement Method**

Description	ASTM Method	Temperature	Typical Location	Elements that Affect Turnaround
Capillary Viscosity	<b>D445</b> Kinematic Viscosity	Typical 40 °C and 100 °C, wide range possible; –40 °C to 150 °C	Oil Analysis Laboratory	<ul style="list-style-type: none"> <li>• If the measurement is made by an automatic unit or manual procedure.</li> <li>• Temperature of measurement as a sample must equilibrate at the test temperature. Standard temperatures such as 40 °C and 100 °C are more likely to be ready on an automated unit or have a bath ready at temperature for a manual measurement.</li> </ul>
Low Temperature Viscosity	<b>D2983</b> Dynamic Viscosity	–55 °C to 20 °C	Oil Analysis Laboratory	<ul style="list-style-type: none"> <li>• This method requires the sample to equilibrate to temperature for 16 h before measurement.</li> </ul>
TBS HTHS Viscosity 150 °C	<b>D4683</b> Dynamic Viscosity	40 °C to 200 °C	Oil Analysis Laboratory	<ul style="list-style-type: none"> <li>• Non-standard temperatures will require a calibration procedure</li> </ul>
Scanning Brookfield Technique	<b>D5133</b> Dynamic Viscosity	–5 °C to –40 °C is typical, can be run to –70 °C	Oil Analysis Laboratory	<ul style="list-style-type: none"> <li>• Test method requires cooling at 1 °C per hour from –5 °C to –40 °C. Test will take at least 48 h to complete.</li> </ul>
Cold Cranking Simulator	<b>D5293</b> Dynamic Viscosity	–5 °C to –30 °C, depending on viscosity grade	Oil Analysis Laboratory	<ul style="list-style-type: none"> <li>• Test takes 1 h to 2 h to complete.</li> </ul>
Falling Needle Viscometer	<b>D5478</b> Dynamic Viscosity	–40 °C to 350 °C	Field, Oil Analysis Laboratory	<ul style="list-style-type: none"> <li>• Field measurements take 2 min to 3 min.</li> <li>• Tests completed in the lab take 2 min to 3 min after temperature equilibration of approximately 5 min to 10 min.</li> </ul>
TBS HTHS Viscosity 100 °C	<b>D6616</b> Dynamic Viscosity	100 °C, see <b>D4683</b>	Oil Analysis Laboratory	<ul style="list-style-type: none"> <li>• Method takes 5 min for results when unit is calibrated and ready to test.</li> </ul>
Yield Stress and Apparent Viscosity	<b>D6896</b> Dynamic Viscosity	–15 °C to –40 °C, depending on viscosity grade	Oil Analysis Laboratory	<ul style="list-style-type: none"> <li>• Depends on W grade, and the temperature of the test. Lower temperatures can require 48 h to 72 h to complete the test.</li> </ul>
Dynamic Viscosity Stabinger	<b>D7042</b> Dynamic Viscosity	–60 °C to 135 °C	Oil Analysis Laboratory	<ul style="list-style-type: none"> <li>• Test results take 1 min to 3 min, depending on precision mode selected.</li> <li>• For samples containing ferrous materials, a magnetic particle trap can be used with to improve performance by removing larger fragments.</li> </ul>
Houillon Kinematic Viscosity	<b>D7279</b> Kinematic Viscosity	20 °C to 120 °C	Oil Analysis Laboratory	<ul style="list-style-type: none"> <li>• A typical test takes 2 min to 3 min to complete. Sample must equilibrate to test temperature.</li> </ul>
Oscillating Piston Dynamic Viscosity	<b>D7483</b> Dynamic Viscosity	–40 °C to 190 °C	Oil Analysis Laboratory	<ul style="list-style-type: none"> <li>• A typical test takes 3 min to 25 min, depending on sample viscosity and temperature differential at the time the new sample is introduced to the viscometer.</li> </ul>
Microchannel Viscometer	<b>D8092</b> Kinematic Viscosity	40 °C	Field	<ul style="list-style-type: none"> <li>• Test takes approximately 1 s per centistoke.</li> </ul>

capable of manufacturing the certified standard viscosity reference material. Some methods specify qualifications of the source for these reference materials. Calibration of various viscometers are defined in their ASTM test methods. Calibration of automated viscometers may be defined in the corresponding ASTM method or the manufacturer's operating manual. A calibration interval for the viscometer may be suggested in its ASTM test method and should be further determined by each laboratory. A calibration verification interval should also be determined to ensure accuracy of the measurement results.

4.8.1.3 Calibration verification of a viscometer by use of a standard is measured by the instrument and the resultant value compared to the certified value of the standard. If the two values agree within an acceptable tolerance as specified by the ASTM Test Method, then the viscometer can be put into use. If the two values do not agree within the acceptable tolerance, then the viscometer or its manual should be checked for help or the manufacturer contacted as needed. (For manual glass capillary viscometers, see Test Method **D445**, subsection 9.2 (Certified Viscosity Reference Standards) and Annex A4 (Calculation of Acceptable Tolerance Zone (Band) to Determine Conformance with a Certified Reference Material).) The certified reference materials are typically manufactured for an intended use and the data corresponds to the appropriate temperature and viscosity range.

#### 4.8.2 Non-certified Reference Fluids:

4.8.2.1 Suitable reference fluids can be chosen and used in a laboratory for a daily or periodic verification of an instrument and these measurements can become part of a statistical quality control (SQC) system for a laboratory. This system would track changes or drifts in the measurement system. If the measured result for a SQC sample does not fall within a defined tolerance, then the viscometer can be calibrated again or the cause of the error found and repaired. The tolerance should be based on the ASTM test method's precision. Non-certified viscosity reference fluids are also available from accredited laboratories. However, many companies use their own base stock or formulated engine oils as the non-certified reference material. When choosing a fluid to use, some important characteristics are:

- (1) The material must be homogeneous,
- (2) Stable over time,
- (3) Free of contaminants, and
- (4) Of sufficient volume such that the material will be available for an extended time.

4.8.2.2 The viscometer manufacturer should be able to assist with selection of an appropriate material for the desired application. Some viscometers are made to measure shear sensitive fluids or low-temperature sensitivity to gelation, and therefore a fluid with similar non-Newtonian characteristics is preferred. For example, some reference materials are fully formulated motor oils and can be used to verify that the applied shear rate corresponds to the method. Practice **D6299** is an

available resource for this further information on this topic. Both certified and non-certified viscosity reference materials should be considered to ensure the accuracy and consistency of the viscosity measurements of the in-service fluids.

### 5. Fluid Rheology; the Flow Behavior of Fluids

5.1 Previous sections of this guide have presented a somewhat simple view of flow behavior of fluids. Understanding of the property of viscosity of a fresh lubricant is not simple, and this property becomes more complex with use of the lubricant. Moreover, the conditions under which the lubricant is used can add further complexity to its viscous behavior. This section extends the degree of understanding of how lubricant viscosity affects and is affected by its hours of use in an effort to remove some of the complexity.

#### 5.2 Newtonian Fluid Behavior:

5.2.1 As previously noted, the viscosity of a simple fluid was defined initially by Newton as the ratio of the force moving the fluid to the rate at which the fluid moves. This ratio is apparent in a restatement of Eq 1 where Newton’s Equation is stated as:

$$\eta = \tau / \dot{G} \quad (3)$$

where:

- $\eta$  = is the viscosity (most often expressed in milliPascal seconds, mPa·s, or centipoise, cP),
- $\tau$  = is the force moving the fluid (called shearstress, expressed experimentally in whatever units of force are applied) and
- $\dot{G}$  = is the rate at which the fluid moves in response to the force (called shear rate and expressed in terms of reciprocal seconds, 1/s or s<sup>-1</sup>).

5.2.2 As stated earlier, an important point is that a fluid having Newtonian properties will, at constant temperature, have the same viscosity no matter what the shear-stress or resultant shear rate is. That is, since Eq 3 is a linear equation, a plot of Eq 3 should be a straight line with the slope equal to viscosity and a zero intercept. This is shown in Fig. 3 for a

Viscometric Response of a Newtonian Lubricant Showing Linear Shear Stress and Rate Behavior As Well as Evident Temperature Dependence

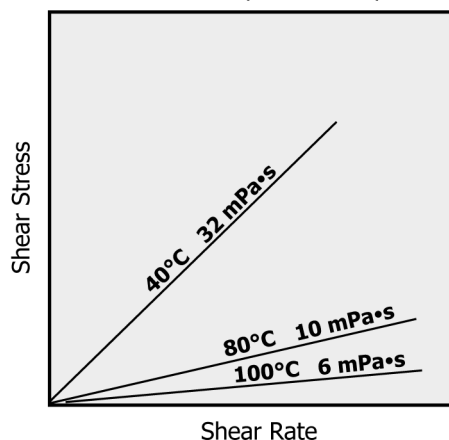


FIG. 3 Viscometric Response of a Newtonian Lubricant

fresh Newtonian engine oil measured at three temperatures as an example. Of further interest in Fig. 3 is the evident effect of temperature on viscosity. Not only does the viscosity of a fluid increase with decreasing temperature but, as suggested by the slopes of the engine oil at the three temperatures, the effect of the temperature is exponential.

5.2.3 That the principles of Newton’s equation remain constant to the physical limits of high shear rate viscometry has been shown by studies made, for example, over a very large shear rate range by the tapered bearing simulator (TBS) viscometer. A plot of the shear rate versus the shear-stress on several Newtonian mineral oils in the TBS viscometer over a wide range of shear rates is shown in Fig. 4. The plot again emphasizes the linearity and zero intercept associated with Newton’s concept of viscosity shown in Eq 3 when analyzing the viscosity of Newtonian fluids.

#### 5.3 Non-Newtonian Fluids and Their Complex Behavior:

5.3.1 Many simple fluids, such as basic mineral oils, are Newtonian. However, with some more complex fluids or when certain additives are dissolved in some Newtonian fluids the ratio of shear stress to shear rate is not constant, and these fluids are called non-Newtonian. Today, many lubricating oils are non-Newtonian in behavior, and this fact also can affect their ability to lubricate and the energy required to overcome their viscous resistance to flow.

5.3.2 Non-Newtonian flow may take many forms in comparison to Newtonian flow. An important expression of such non-Newtonian flow in lubricants is shown in Fig. 5 compared to Newtonian flow and is termed “Temporary Viscosity Loss” (TVL). Its history is interesting.

5.3.3 Since the 1940s, some of the important additives used to formulate lubricants have been so-called “Viscosity Index (VI) Modifiers.” These additives are composed of very large polymeric macromolecules which, in relatively small concentrations, are dissolved in the mineral oil base stock. These macromolecules impart desirable viscometric properties at both low and operating temperatures at low shear rates.

5.3.4 On dissolution, the molecular size of these VI modifiers increases greatly, and by doing this impart a resistance to flow that results in an accompanying large viscosity increase to the base oil in which they are dissolved as shown in Fig. 6.

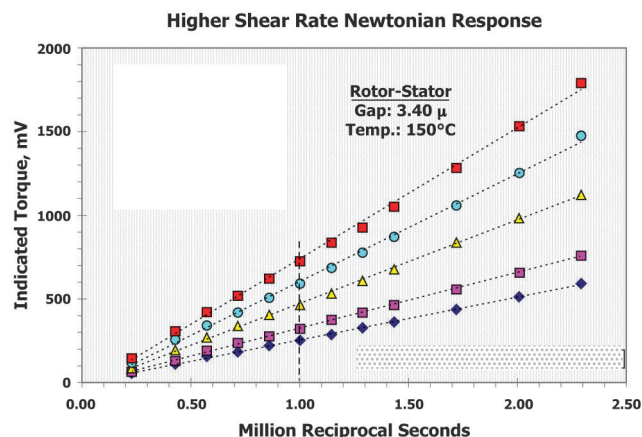


FIG. 4 Higher Shear Rate Newtonian Response

### The Rheology of Oil Containing Dissolved Polymer

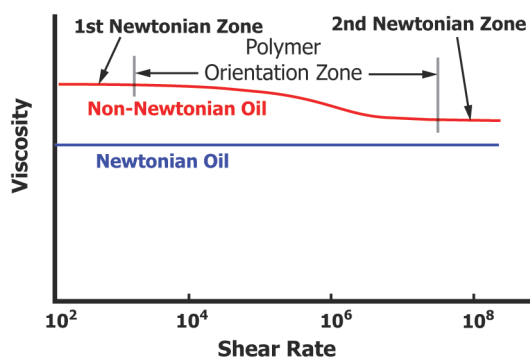


FIG. 5 Example of a Non-Newtonian Oil Showing Classic Temporary Viscosity Loss

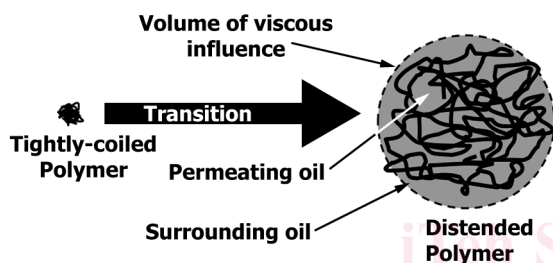


FIG. 6 Distention of the Polymeric Macromolecule in Mineral Oil Solution

Using much lower viscosity base oils results in formulations that at low shear rates have viscosities similar to those of non-polymer-containing lubricants at operating temperatures. This enables the formulator to use lower viscosity base oils and still achieve the same viscosity that higher viscosity oils show at operating temperatures. However, as noted above, much of this viscosity increase is subject to TVL. This approach has since become a well-known practice in lubricant formulations and is very common, for example, in blending so-called “multi-grade engine oils” to meet the Society of Automotive Engineers (SAE) classifications of engine oils such as SAE 10W-30, SAE 0W-20, and so on. Such non-Newtonian behavior and its variation with usage of the lubricant has been the subject of much dialogue and technical publications over the last several decades.

5.3.5 Shear-thinning is very commonly encountered especially in lubricants in which VI improvers are used. The fact that this shear-thinning is often called Temporary Viscosity Loss (TVL) is because on lowering shear rate any viscosity “lost” returns. Shear rates producing TVL are in the millions of reciprocal seconds. Such high values of shear rate are the normal operating level for most lubrication of automotive engines and other machinery. Temporary viscosity loss may reach levels of 30 % or more depending on the lubricant formulation.

5.3.6 Another important reason for high technical interest in the contribution of viscosity to mineral oils by the addition of VI improvers is particularly in regard to engine oils. Unfortunately, at the shear rates and other, non-viscous forces

applied in lubrication, the larger of these polymeric additives are vulnerable to mechanical rupture. This causes a significant and unrecoverable loss of lubricant viscosity—a so-called “Permanent Viscosity Loss” or PVL. The amount of loss is dependent on how vulnerable the dissolved macromolecules are to the forces and conditions to which they are exposed during use. It has been found that PVL may reach 20 % or more during use of the lubricant.

5.3.7 Another factor that can influence the viscosity/shear rate response is the increase of viscosity due to oxidation of the lubricant. Usually, the effect of lubricant oxidation is shown by viscosity increase which may or may not be affected by shear rate.

5.3.8 *Specific to the Falling Needle Viscometer*—For shear thinning fluids, drop a needle with hemispherical ends and extension bar plus external weights (different densities with the same geometry) and measure their terminal velocities by the amount of time taken to travel between two of the measurement lines by using a stopwatch or an automatic sensing device. The viscosities and shear rates can be determined based on the needle velocities, the fluid and needle densities, the needle and system geometries, and gravitational acceleration.

## 6. Care to Avoid Adverse Effects of Contaminants<sup>5</sup>

6.1 *Sampling Procedures*—Sampling procedures must be developed and applied with care particularly when sampling from large containers or sumps of machines. This is especially true when sampling from the bottom of a tank, container, or sump, where heavier particulates and water—both heavier than the oil—will accumulate. Viscosity of the oil sample will obviously be influenced by the water content and particulate content and perhaps even prevent a viscosity measurement from being made depending on the instrument. It is certainly important to choose a viscometric instrument and method to avoid significant error in the effort to obtain meaningful information.

### 6.2 Effect of Solids or Semi-solid Particles in the Sample:

6.2.1 Some viscosity measurement techniques, such as capillary viscometry, can be sensitive to the presence of particulates in a sample and may require the sample to be filtered prior to analysis. Depending on the purpose of the analysis, filtration prior to measurement may be an acceptable practice. Alternatively, obtaining the viscosity of both the unfiltered and filtered oil sample may provide desirable information. On the other hand, it may be of primary interest to analyze the unfiltered sample to learn about the cause or character of the particles.

6.2.2 Once more, it is informative to consider why monitoring oil viscosity is very important. Most lubricated, load bearing moving surfaces are separated by a lubrication film thickness of 10 μm or less. Many of these loaded surfaces in near-contact are prevented from contacting only by the viscosity of the lubricant at that temperature. This viscosity-imposed separation is dependent on the viscosity of the continuously supplied lubricating oil forced into that zone of separation. If

<sup>5</sup> Additional resources available from Trico Corporation, 1235 Hickory St., Pewaukee WI 53072, <http://www.tricocorp.com>.



the lubricant cannot escape the compressive pressure fast enough, the surfaces cannot come into contact because the lubricant cannot be compressed beyond a certain thickness, again dependent upon the lubricant. Especially for systems such as engines or high speed turbochargers, maintaining the correct oil viscosity is quite critical. Any significant decrease of oil viscosity may permit contact of the two surfaces in relative motion and cause damage. In high-pressure hydraulic systems, an increase in viscosity may adversely reduce pumping effectiveness.

6.3 *Water Saturation of the Sample*—Once the saturation point of water for the oil is exceeded, the viscosity of the oil will be less accurate. If it is desired to determine the amount of water in the lubricant, analysis by Karl Fischer, Test Method D6304 is the most accurate test for water up to a concentration of about 1 % in the oil. Over 1 %, Fourier-transform infrared spectroscopy (FTIR) is a more informative but less precise test. See Table 2.

6.4 *Viscometric Suspension of Solids or Semi-solid Particles in the Sample:*

6.4.1 Particulate or soot levels depend on the viscosity of the oil. That is, depending on the densities of both the oil and the particulates, the particulates will either sink or rise to the surface over time. Also, the viscosity of the oil will determine the rate at which particles will settle out or rise. Of course, depending on the viscometric information desired, the oil may be measured with its particles well-distributed by shaking or agitation or by removal through filtration or centrifugation.

6.4.2 A brief summary of potential effects of a viscosity change in an in-service oil:

**Reduction in Viscosity:**

- Loss of oil film resulting in excessive wear.
- Increased mechanical friction causing excessive energy consumption and heat generation.
- Increased sensitivity to particle contamination due to reduced oil film thickness.
- Oil film failure at high temperatures, high loads during start-ups or coast-downs.

**Increase in Viscosity:**

- Excessive heat generation resulting in oil oxidation, sludge and varnish build-up.
- Gaseous cavitation due to inadequate oil flow to pumps and bearings.
- Lubrication starvation due to inadequate oil flow to pumps and bearings.
- Oil whip in journal bearings.
- Excess energy consumption to overcome fluid friction. Poor air release or demulsibility.
- Poor cold start pumpability.

6.4.3 The end results of above effects are shorter oil lifespan, shorter components lifecycle, increased oil consumption, higher power consumption, and reduced machine reliability. It is extremely important for the end user to find the root cause of oil viscosity change and to act accordingly at the earliest possible time to minimize the damage.

6.4.4 A brief summary of potential root causes of a viscosity change in an in-service oil:

**Reduction in Viscosity:**

- Thermal cracking of oil molecules.
- Shear thinning of VI improvers (for multigrade engine oil).
- Fuel dilution.
- Cross mixing with lower viscosity oil.

**Increase in Viscosity:**

- Oxidation.
- Water (emulsion).
- Formation of carbon and oxides that create insoluble soot.
- Antifreeze (Glycol).
- Cross mixing with higher viscosity oil.

6.4.5 Most of the factors, except water ingress, may be rectified by changing the oil. However, further analysis is required to confirm the root cause before any actions are taken. Once the root cause(s) is/are confirmed, remedial action should be taken to prevent the incident from recurring, especially if contamination is identified. It is important to note that sometimes two factors may be acting in opposite directions, resulting in an acceptable viscosity range, for example soot (increases viscosity) and fuel dilution (reduces viscosity) for engine oil. Both factors can be measured separately, and if either of them exceed acceptable limits, this may be detrimental to the machine. Hence, viscosity analysis alone is not sufficient, but extremely important.

6.4.6 In conclusion, as the most important characteristic for lubricating oil, viscosity of in-service oil must be monitored as a routine parameter in oil analysis. However, in order to get the most benefit from oil analysis, other important parameters must be analyzed as well to get the holistic view of oil and machines' condition.

**7. In-Service Oil Viscosity Measurement Techniques**

**Test Method D445**

7.1 *Test Method D445 for Kinematic Viscosity of Transparent and Opaque Liquids*—As previously discussed, kinematic viscosity measurement of in-service lubricants provide a value that is dependent on the ratio of the actual viscosity divided by the fluid density. The use of glass capillary kinematic viscometers presents several issues to consider. This section covers both the manual viscometers and automated kinematic viscometers.

7.1.1 *Factors Affecting Kinematic Viscosity Measurements*—Items discussed here include:

- 7.1.1.1 Sample preparation.
- 7.1.1.2 Samples containing water.
- 7.1.1.3 Samples containing particulate.
- 7.1.1.4 Opaque samples.
- 7.1.1.5 Solvent selection.
- 7.1.1.6 Samples containing volatiles.
- 7.1.1.7 Solvent selection.
- 7.1.1.8 Sample coating of the kinematic capillary viscometer walls.

7.1.2 *Sample Preparation*—Particularly if a sample is non-homogeneous, the sample should first be thoroughly mixed, stirred, or shaken to improve the homogeneity but assurance of homogeneity by such means for any sample is good practice. After mixing, stirring, or shaking, permit the sample to set for a few minutes to permit any bubbles of air to rise and leave, or else the density of the fluid needed to drive the fluid through the capillary will be slightly less and result in a slightly higher

**TABLE 2 Examples of Saturation Levels for Various Oils**

Oil Type	Dissolved (ppm)	Emulsified (ppm)	Free (ppm)
New Hydraulic Fluid	0 – 200	200 – 1000	>1000
Aged Hydraulic Fluid	0 – 600	600 – 5000	>5000
New R&O Oil	0 – 150	150 – 500	>500
Aged R&O Oil	0 – 500	500 – 1000	>1000

kinematic viscosity. Do not use a vacuum on the sample or heat it prior to determining its kinematic viscosity, as this may remove more volatile components of the fluid also resulting in a somewhat higher kinematic viscosity.

### 7.1.3 *Samples Containing Water:*

7.1.3.1 Samples that have a high water content will likely not give repeatable or valid results at temperatures  $\geq 100$  °C due to boiling of the water in the sample. However, it must be considered that any attempt to remove water by heating the sample to or above 100 °C before measuring its kinematic viscosity will also have an effect on removing more volatile components of the sample. Samples of engine oil often have some water and volatiles present as a consequence of their use and different viscometric instrument might be considered for in-service viscosity studies.

7.1.3.2 Emulsions are encountered in lubricants and other mineral-oil-based fluids. A number of these emulsions contain water. Again, kinematic viscometry at 100 °C and above may be subject to error both because of the water content and the size of the emulsion particles. In such cases, it would be wise to contact the manufacturer of the capillary viscometers to obtain guidance for kinematic viscometry.

### 7.1.4 *Filtering Particulates:*

7.1.4.1 If the presence of particles is undesirable for the information desired, particulate filtering may be necessary prior to measuring kinematic viscosity in accordance with Test Method D445. This method specifies glass capillary kinematic viscometers and the capillary size would determine the filtering requirements if one knows the diameter of the particulates. Specification D446 specifies the capillary diameter for many of the manual glass kinematic viscometer tubes. For the automated kinematic viscometers, the manufacturer should be contacted for information regarding the capillary diameter. One of the problems is if the sample is not filtered, it is possible to pull particulate up through the capillary and it can become lodged at the capillary opening, thus changing the flow rate through the capillary. This scenario will cause the reported viscosity to be higher than the actual value.

7.1.4.2 Filtering particulates will prevent partial ‘clogging’ or interference with the flow of the fluid through the capillary. Any particulates, unless very small in comparison to the capillary diameter, will cause kinematic viscosity values to be generated that, on measuring density to convert the value to true dynamic viscosity, will be in some degree of error. Filtering samples containing particles before measuring their kinematic viscosity is desirable, although it will not provide the accurate kinematic viscosity of the particle-containing oil lubricating the mechanism.

7.1.5 *Measuring Opaque Samples*—Reverse-flow manual kinematic viscometers are available for measuring opaque samples, such as highly-oxidized or particle-containing engine oil. Some automated viscometers use thermal detection of the meniscus rather than optical detection, and the opacity of the sample does not offer any obstacle to the sample measurement. Consult the manufacturer to determine the ability to measure opaque samples.

### 7.1.6 *Solvent Selection for Cleaning Kinematic Viscometers:*

7.1.6.1 To clean the kinematic viscometer according to Test Method D445, one must choose a cleaning solvent miscible with the sample as well as a drying solvent miscible with the cleaning solvent and water. Prior to selecting the cleaning solvent, determine the likely contaminants that may be present in the sample. After removing the bulk of the sample by pouring it out or vacuum-extracting it, a suitable solvent can then be selected to adequately remove the remnant sample from the viscometer. Soot, glycol, fuels, water, and metals will potentially be in a sample and need to be removed. While the metals are normally flushed out with the sample; soot, glycol, and water may require different solvent selections than would be needed for new lube oils.

7.1.6.2 For automated kinematic viscometers, verify the solvents are compatible with all wetted components in the viscometer. The manufacturer can provide information for this choice.

7.1.6.3 Verification that the kinematic viscometer has been adequately cleaned after measuring a particular type of sample can be accomplished by measuring a certified reference fluid using two or three measurements using the kinematic viscometer, and then verifying that the repeatability meets the Test Method D445 precision statement for it. Also, verify the kinematic viscosity result agrees with the certified value.

### 7.1.7 *Measuring Samples Containing Volatile Components:*

7.1.7.1 Volatile types of samples can offer several issues. For manual measurements, boiling can occur while measuring at elevated temperatures, such as 100 °C and above, kinematic viscosity, and more volatile components of the oil can be lost. Light ends can also be lost when the sample is drawn into the viscometer tube under vacuum. Moreover, the more volatile components can separate and air pockets or foam can develop within the sample column. Timing in determining kinematic viscosity can be more difficult if bubbles develop at the meniscus.

7.1.7.2 Automated instruments may exhibit the same issues as in manual testing, and the column separation and bubbling can be exasperated by the sample being drawn up through the capillary at the test temperature.

7.1.7.3 Measuring volatile samples at 40 °C may be an alternative to 100 °C measurement.

### 7.1.8 *Sample Coating on the Viscometer Walls:*

7.1.8.1 Some samples may adhere and coat the wall of the glass capillary of the kinematic viscometer over time. The length of time a sample requires to coat the wall of the viscometer tube will vary depending on what is coating it, cleaning effectiveness, and the measurement temperature. Among these coating-forming components adhering to the glass may be additives used to improve the lubrication or engine performance. Also, soot can adhere to the glass or to a coating on the surface. Kinematic viscometer cleaning with chromic acid can be performed to remove such coatings. Alternative products to chromic acid are available and may be used, or sulfuric acid may be used, to clean the viscometer without etching the glass. Glassware detergents are also available and will work to clean the kinematic viscometer. However, some of these are highly alkaline and could etch the glass wall of the capillary and thus change the viscometer