1 Introduction

Viscosity is the most important parameter of a lube oil. It is essential to determine a lube's
- Kinematic viscosity at +100 °C, further the
- Kinematic viscosity at +40 °C, in order to calculate the
- Viscosity Index (VI).
In addition, an oil must fulfill several other parameters. Anton Paar provides the SVM™ 3001 Stabinger Viscometer™ for measurement according to ASTM D7042. It is an excellent alternative to conventional capillary viscometry for fast and economical measurement of the low-shear kinematic viscosity over a wide temperature range. This report describes specifically how to test fresh lube oils with the SVM™ 3001 to get data comparable to ASTM D445. The instrument's software is able to calculate the VI from usual viscosity values at 40 °C and 100 °C and from other temperatures according to ASTM D341. Temperature scan, temperature table scan and time scan allow testing oils for temperature dependent changes or changes over time. Scan results are available as graphs, too. All results can be transferred to a PC for further processing via LIMS.

2 Instrumentation

The samples were measured with the SVM™ 3001 viscometer. The instrument features a viscosity and a density measuring cell, which are filled in one go.

Find optional accessories in the SVM™ X001 Product Description List.

2.1 Installation

Refer to the SVM™ X001 Instruction Manual.

3 Measurement of lube oils

3.1 Sample preparation

If the sample is not freshly drawn from a production line or other reservoir, you can improve the repeatability by homogenizing the sample before taking the test specimen. Proceed as follows:
- Fill approximately 100 mL of sample into a glass beaker.
- Cover the beaker with laboratory film to avoid contamination.
- Stir the sample on a magnetic stirrer at low speed for approximately 5 minutes.
3.2 Instrument preparation
The measuring cells must be leak tight, clean and dry.

3.2.1 Instrument settings

Repeated determinations at one temperature:
According to ASTM D7042
- Method: Standard
- Precision class "Precise"
- RDV limit 0.10 %
- RDD limit 0.0002 g/cm³
- 5 determinations
- Automatic prewetting: yes
- Drying time (built-in air pump):
  - at 40 °C: 80 s
  - at 100 °C: 60 s

Automatic Viscosity Index measurement:
According to ASTM D7042
Method: Viscosity Index
Precision class "Precise"
Automatic prewetting: yes
Drying time (built-in air pump):
same as for single point measurement

Note: SVM™ 3001 does not measure repeated determinations in the VI mode. It performs only one single determination at each temperature. ASTM D2270 requires repeated measurements at each temperature. For VI determination according to ASTM D2270, use the double-cell viscometer SVM™ 4001 (separate application report available).

3.3 Calibration
Before measuring the samples, perform a calibration. If required, apply a calibration correction to improve the reproducibility. Use one or more standard(s) in the viscosity range of your lube oil sample(s). This can be a certified standard or a house-internal standard with kinematic viscosity values. In any case, you need reliable kinematic viscosity values at the measuring temperatures.

To perform a calibration (correction), refer to the SVM™ X001 Reference Guide.

3.4 Filling
Use disposable (single-use) syringes. Never use syringes with rubber sealings, as the rubber is chemically not resistant and these syringes tend to suck bubbles.

- Sample volume: typically 5 mL (depending on sample)
- Sample throughput: approx. 10 samples/hour

3.5 Cleaning

3.5.1 Solvents

Solvent quality:
The used solvent needs to dry up completely at the measuring temperature. If using a single solvent, the solvent quality shall be "chemically pure" or "for synthesis". If using two solvents, only the second solvent needs to meet this quality.

Petroleum benzine:
Petroleum benzine (a de-aromatized hydrocarbon solvent, blend of mainly C7, C8, C9 n-alkanes) with a boiling range of 100 °C to 140 °C is the best choice for most oils. This universal solvent can be used over the entire temperature range of SVM™ 3001.

Aromatic solvents:
Some oil samples require toluene or xylene, as they are not (completely) soluble in petroleum benzine. In this case, petroleum benzine is recommended as second solvent for perfect drying of the cells. If petroleum benzine is not available in your country, toluene/xylene as first solvent and n-hexane/n-heptane or a similar hydrocarbon solvent (mixture) as second solvent can be used.

Ethanol, acetone:
Ethanol or acetone as second solvent are not recommended for petroleum based oils. These solvents have a negative influence on the surface wetting behavior for oils. The filling quality of the measuring cells is worse compared to after using hydrocarbon solvents.

3.5.2 Cleaning procedure
For details, see the SVM™ X001 Instruction Manual.

Tip: Open the cleaning screen. Observe it during the cleaning procedure. It gives helpful information on the cleaning and drying status of the cells.

1. Remove the sample from the cells (push through or suck back) using a syringe.
2. Fill approximately 2 mL of solvent using a syringe. The syringe remains connected.
3. Start the motor for a few seconds to improve the cleaning performance in the viscosity cell.
4. Move the plunger of the syringe forth and back when the motor is at filling speed. This improves the cleaning performance both in the density oscillator and in the viscosity cell.
5. Before filling solvent for a new cleaning cycle, remove the sample-solvent-mixture from the cells. Blow air through the cells for some seconds for better removal of the liquid.

6. Ensure to perform a sufficient number of cleaning cycles as the sample is rather highly viscous compared to the solvent.

7. Perform a final flush with fresh solvent to remove any residues. If applicable, flush with a second solvent to improve the drying.

8. Allow a sufficiently long drying time to be sure that the solvent can dry up completely.

Solvent consumption: typically 6 mL (depends on oil and viscosity)

4 Results

This report compares the data of engine oil measured at 40 °C and 100 °C with SVM™ 3001 (ASTM D7042) and with Ubbelohde viscometer (ASTM D445).

<table>
<thead>
<tr>
<th>Engine oil lubricant</th>
<th>D445* mm²/s</th>
<th>D7042** mm²/s</th>
<th>Deviation mm²/s</th>
<th>Deviation %</th>
</tr>
</thead>
<tbody>
<tr>
<td>LU 1409</td>
<td>175.88</td>
<td>175.55</td>
<td>-0.325</td>
<td>-0.18</td>
</tr>
<tr>
<td>LU 1401</td>
<td>121.61</td>
<td>121.97</td>
<td>0.357</td>
<td>0.29</td>
</tr>
<tr>
<td>LU 1309</td>
<td>124.29</td>
<td>124.19</td>
<td>-0.102</td>
<td>-0.08</td>
</tr>
<tr>
<td>LU 1101</td>
<td>113.40</td>
<td>113.15</td>
<td>-0.250</td>
<td>-0.22</td>
</tr>
</tbody>
</table>

Table 1: Comparison of ASTM D7042 and D445 results at 40 °C

<table>
<thead>
<tr>
<th>Engine oil lubricant</th>
<th>D445* mm²/s</th>
<th>D7042** mm²/s</th>
<th>Deviation mm²/s</th>
<th>Deviation %</th>
</tr>
</thead>
<tbody>
<tr>
<td>LU 1409</td>
<td>19.845</td>
<td>19.788</td>
<td>-0.057</td>
<td>-0.29</td>
</tr>
<tr>
<td>LU 1401</td>
<td>16.135</td>
<td>16.083</td>
<td>-0.052</td>
<td>-0.32</td>
</tr>
<tr>
<td>LU 1309</td>
<td>15.287</td>
<td>15.296</td>
<td>0.009</td>
<td>0.06</td>
</tr>
<tr>
<td>LU 1101</td>
<td>15.143</td>
<td>15.113</td>
<td>-0.031</td>
<td>-0.20</td>
</tr>
</tbody>
</table>

Table 2: Comparison of ASTM D7042 and D445 results at 100 °C

* ASTM D445 states a reproducibility (R) of 0.76 % for formulated oils at +40 °C and +100 °C.

** ASTM D7042 states a reproducibility (R) of 0.1087E-01(X + 10.6) % for kinematic viscosity of formulated oils at +40 °C and 0.1087E-01(X + 10.6) % at +100 °C, where X is the result obtained by ASTM D7042.

5 References

- ASTM D7042: Standard Test Method for Dynamic Viscosity and Density of Liquids by Stabinger Viscometer (and the Calculation of Kinematic Viscosity)
- EN ISO 3104: Petroleum products - Transparent and opaque liquids - Determination of kinematic viscosity and calculation of dynamic viscosity
- ASTM D2270: Standard Practice for Calculating Viscosity Index From Kinematic Viscosity at 40 °C and 100 °C

6 Conclusion

The SVM™ 3001 is perfectly suited for determining the kinematic viscosity of lube oils.

6.1 Comparison to conventional viscosity determination

Classically, kinematic viscosity is determined using glass capillary viscometers. The measurement can be performed with either manually filled capillaries and manual timing, manually filled capillaries with automatic timing, or fully automated systems.

SVM™ 3001 Typical glass capillary viscometer

- one measuring cell for all sample types and temperatures
- select the suitable capillary for the sample’s viscosity at the measuring temperature

Measurement according to standard

- ASTM D7042
- ASTM D445

Sample consumption

- typically 5 mL
- approximately 12 to 13 mL

Solvent consumption

- typically 6 to 10 mL
- approximately 50 to 60 mL

Sample throughput

- approx. 10 samples/hour
- manually filled Ubbelohde: approx. 2 samples/hour

Table 3: Comparison of viscometer types

Contact Anton Paar GmbH
Tel: +43 316 257-0
support-visco@anton-paar.com
www.anton-paar.com
Appendix A. Lubricating oils

Lube oils are generally formulated oils. They consist of mineral, semi- or fully synthetic base oil (base stocks) plus a varying number and amount of additives. The quality of a lube oil depends on the base stock and its properties, as well as on the additives. Base stocks alone cannot be used as lube oils, as they lack many of the required properties. Additives are essential to obtain or improve the properties of a lube oil; e.g. defined temperature-viscosity properties, protection against wear and corrosion, keeping the engine clean, holding particles like soot or abrasives in suspension, yield strength under compression and many more. Additives also inhibit or suppress negative properties of base oils such as foaming and ageing.

Explaining the matter in a simplified way, lube oils are produced by blending base oils with additives until the desired properties of the final oil are reached.

Base oils

There are six groups of base oils used for blending lubricating oils.
- Group I – Solvent refined base stocks.
- Group II – Hydro processed base stocks (hydrocrack oils, HC).
- Group III – Unconventional Base Oils (UCBO), severely hydro processed, with high VI.
- Group IV – Polyalphaolefines (PAO), the basis for most traditional synthetic lubricants
- Group V – All base stocks that do not fall into groups I to IV, like alkylated naphthalene, esters, polyalkylene glycols, silicones, polybutenes, ester polyoles. They are mainly used to create oil additives.
- Group VI – Poly internal olefines (PIO) a new group classified in Europe only, similar to PAOs

Additives

There are various additives available to blend oils for different lube requirements. A very important group are viscosity modifiers, which include the VI improvers and pour point depressants.

Depending on requirements, lube oil can contain the following additives:
- Viscosity Index improver (VI-Improver, VII)
- Pour point depressants (PPDs)
- Friction modifiers
- Extreme pressure additives (EP) / Anti wear additives (AW)
- Corrosion inhibitors
- Dispersants
- Detergents
- Antioxidants
- Anti-foam agents
- Seal conditioners

Appendix B. Why Measure Viscosity?

General Overview

For lube oils, different viscosities must be determined. For the finished oil, the viscosity index (VI) is stated.

Kinematic viscosity (low-shear) at 100 °C

While the engine is at operating temperature, the oil has a high temperature. For SAE W-classes a minimum viscosity, for SAE summer classes a viscosity range is specified. Classically, the low-shear kinematic viscosity is measured with glass capillary instruments according to ASTM D445 / ISO 3104. Alternatively, this measurement can be performed with SVM™ 3001 according to ASTM D7042.

Viscosity Index (VI)

The VI shows the influence of temperature on an oil's viscosity. It is calculated from kinematic viscosity at 40 °C and 100 °C according to ASTM D2270. ASTM D7042 is referenced in this standard for determination of kinematic viscosity. Low VI means a considerable change of viscosity with change of temperature. Such an oil is highly viscous at low temperatures and rather liquid at high temperatures. A high VI means the opposite, a small change of viscosity over a wide temperature range. SVM™ 4001 is perfectly suited for VI measurement. It measures kinematic viscosities at 40 °C and 100 °C with repeated determinations and automatically calculates the VI from the obtained results. SVM™ 3001 also provides a measurement mode, which automatically calculates the VI. As measurements at 40 °C and 100 °C are only single point determinations, The VI-mode of SVM™ 3001 is not compliant to ASTM D2270.

To achieve the desired VI, special polymers – so-called Viscosity Index improvers (VI-Improve, VII) – are added to the oil. The polymer molecules are small and coil-shaped when cold. In that state they do not increase the oil's viscosity. With rising temperature, the molecules unfold. Consequently, they reduce the
decrease of viscosity that is caused by the higher temperatures.
Due to the behavior of the polymer molecules, it often makes a difference whether the VI measurement is performed with rising temperatures or with descending temperatures. The viscosity readings and VI results for a rising VI scan (from 40 °C to 100 °C) differ from those of a descending VI scan (from 100 °C to 40 °C). In such a case, it is recommended to perform the measurement and the repeat measurement always in one direction, preferably from lower to higher temperature.

Other viscosity types determined for lube oils
- Cold cranking viscosity
- Pumpability of the oil after engine start (Borderline Pumping Temperature; BPT)
- High temperature high shear viscosity (HTHSV)

Note: These three parameters cannot be measured with SVM™ X001.

Viscosity parameters and typical viscosities
The key viscosity parameter for engine oils is the kinematic viscosity, specified at +100 °C. Further, values for +40 °C and the viscosity index (VI) are stated, as they are also quality indicators.

Viscosity specification of crankcase lubricants according to SAE J300, see Table 4 (excerpt of the full viscosity specification table):

<table>
<thead>
<tr>
<th>SAE Viscosity grade</th>
<th>Low shear rate kinematic viscosity at 100°C [mm²/s, cSt] min.</th>
<th>Low shear rate kinematic viscosity at 100°C [mm²/s, cSt] max.</th>
</tr>
</thead>
<tbody>
<tr>
<td>0W</td>
<td>3.8</td>
<td></td>
</tr>
<tr>
<td>5W</td>
<td>3.8</td>
<td></td>
</tr>
<tr>
<td>10W</td>
<td>4.1</td>
<td></td>
</tr>
<tr>
<td>15W</td>
<td>5.6</td>
<td></td>
</tr>
<tr>
<td>20W</td>
<td>5.6</td>
<td></td>
</tr>
<tr>
<td>25W</td>
<td>9.3</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>4</td>
<td>6.1</td>
</tr>
<tr>
<td>12</td>
<td>5</td>
<td>7.1</td>
</tr>
<tr>
<td>16</td>
<td>6.1</td>
<td>8.2</td>
</tr>
<tr>
<td>20</td>
<td>5.6</td>
<td>9.3</td>
</tr>
<tr>
<td>30</td>
<td>9.3</td>
<td>12.5</td>
</tr>
<tr>
<td>40</td>
<td>12.5</td>
<td>16.3</td>
</tr>
<tr>
<td>50</td>
<td>12.5</td>
<td>16.3</td>
</tr>
<tr>
<td>60</td>
<td>21.9</td>
<td>26.1</td>
</tr>
</tbody>
</table>

Table 4: Crankcase lubricants - viscosity specifications (SAE J300)

For gearbox and drive line oils, the low shear rate kinematic viscosity at 100 °C is specified according to SAE J306. See Table 5 (excerpt of the full viscosity specification table):

<table>
<thead>
<tr>
<th>SAE Viscosity grade</th>
<th>Low shear rate kinematic viscosity at 100°C [mm²/s, cSt] min.</th>
<th>Low shear rate kinematic viscosity at 100°C [mm²/s, cSt] max.</th>
</tr>
</thead>
<tbody>
<tr>
<td>70W</td>
<td>4.1</td>
<td></td>
</tr>
<tr>
<td>75W</td>
<td>4.1</td>
<td></td>
</tr>
<tr>
<td>80W</td>
<td>7.0</td>
<td></td>
</tr>
<tr>
<td>85W</td>
<td>11.0</td>
<td></td>
</tr>
<tr>
<td>80</td>
<td>7.0</td>
<td>11.0</td>
</tr>
<tr>
<td>85</td>
<td>11.0</td>
<td>13.5</td>
</tr>
<tr>
<td>90</td>
<td>13.5</td>
<td>18.5</td>
</tr>
<tr>
<td>110</td>
<td>18.5</td>
<td>24.0</td>
</tr>
<tr>
<td>140</td>
<td>24.0</td>
<td>32.5</td>
</tr>
<tr>
<td>190</td>
<td>32.5</td>
<td>41.0</td>
</tr>
<tr>
<td>250</td>
<td>41.0</td>
<td>--</td>
</tr>
</tbody>
</table>

Table 5: Gearbox lubricants - viscosity specifications SAE J306

All other oils are viscosity classified in ISO 3448. This standard knows 18 viscosity grades (ISO VG) from 2 mm²/s to 1500 mm²/s. Each grade specifies a center viscosity at 40 °C with tolerable maximum deviations of 10 % in both directions: this specifies an acceptable viscosity range at 40 °C. See Table 6 for the ISO viscosity grades (excerpt of full viscosity specification table):

<table>
<thead>
<tr>
<th>ISO Viscosity grade</th>
<th>Center point viscosity at 40°C [mm²/s, cSt]</th>
<th>Low shear rate kinematic viscosity at 100°C [mm²/s, cSt]</th>
</tr>
</thead>
<tbody>
<tr>
<td>ISO VG 2</td>
<td>2.2</td>
<td>1.98</td>
</tr>
<tr>
<td>ISO VG 3</td>
<td>3.2</td>
<td>2.88</td>
</tr>
<tr>
<td>ISO VG 5</td>
<td>4.6</td>
<td>4.14</td>
</tr>
<tr>
<td>ISO VG 7</td>
<td>6.8</td>
<td>6.12</td>
</tr>
<tr>
<td>ISO VG 10</td>
<td>10</td>
<td>9.0</td>
</tr>
<tr>
<td>ISO VG 15</td>
<td>15</td>
<td>13.5</td>
</tr>
<tr>
<td>ISO VG 22</td>
<td>22</td>
<td>19.8</td>
</tr>
<tr>
<td>ISO VG 32</td>
<td>32</td>
<td>28.8</td>
</tr>
<tr>
<td>ISO VG 46</td>
<td>46</td>
<td>41.4</td>
</tr>
<tr>
<td>ISO VG 68</td>
<td>68</td>
<td>61.2</td>
</tr>
<tr>
<td>ISO VG 100</td>
<td>100</td>
<td>90.0</td>
</tr>
<tr>
<td>ISO VG 150</td>
<td>150</td>
<td>135</td>
</tr>
<tr>
<td>ISO VG 220</td>
<td>220</td>
<td>198</td>
</tr>
<tr>
<td>ISO VG 320</td>
<td>320</td>
<td>288</td>
</tr>
<tr>
<td>ISO VG 460</td>
<td>460</td>
<td>414</td>
</tr>
</tbody>
</table>

Table 6: Viscosity classification of oils according to ISO 3448
Although there are many other oil specification standards, the viscosity specifications are more or less related to SAE J300/J306 or ISO 3448, respectively. Manufacturer standards (OEM standards) and military standards sometimes state tighter specification limits.

Lube oil specification standards

For specification details, see the following standards:

- SAE J300 - Viscosity of Automotive Engine Oils
- SAE J306 - Viscosity of Automotive Gear Oils
- ISO 3448 - Industrial Liquid Lubricants - ISO Viscosity Classification
- SAE J2360 - Automotive Gear Lubricants for Commercial and Military Use
- SAE J1423 - Classification of Energy Conserving Engine Oil for Passenger Cars, Vans, Sport Utility Vehicles, and Light-Duty Trucks
- ACEA (Association des Constructeurs Européens d'Automobiles = European Automobile Manufacturer’s Association) European Oil Sequences (2012): Service fill oils for gasoline engines, light duty diesel engines, Engines with after treatment devices and heavy duty diesel engines
- Mil-L-2105D - Military Specification Lubricating Oil Gear, Multi Purpose
- Mil-PRF-2105E - Performance Specification Lubricating Oil Gear, Multi Purpose
- Mil-L-2104F - Military Specification Lubricating Oil, Internal Combustion Engine, Combat/Tactical Service
- AGMA (American Gear Oil Association) 9005-D94 / 9005-E02 Industrial gear lubrication
- Vehicle manufacturer standards (OEM standards), e.g.: MB 229.51, MAN 342, VW 506.01, Renault RN 0710, Chrysler MS-11106, PSA B 712290, Fiat 9.55535-S1, Ford WSS-M2C913-C and many more.