1 Why measure viscosity?

Silicone oils provide a lot of unique properties such as thermal stability, wide operational temperature range, a very high viscosity index, low surface tension and chemical inertness to list only a few of them. They are used e.g. as lubricants, damping oils, hydraulic oils, heat transfer oils and additives in plastics. Nearly all branches of industries use them, e.g. cosmetic, pharmaceutical, textile, food, glass, wood, metalworking industry and many more.

For all these different applications, viscosity plays a major role. The correct viscosity ensures e.g. optimum heat transfer, quick response of brake pedal and pistons or valves within a brake system over a large temperature range, smooth dispersion of the liquid on surfaces, best lubrication properties. A change of viscosity can also indicate a change or degradation of the oil.

Usually, kinematic viscosity and density of silicone oils are specified at 25 °C. Depending on their intended use, viscosity and density are often stated at the temperatures at which the material is used, e.g. at 40 °C or, for brake fluid, even at -40 °C. Furthermore, the viscosity index and for some applications also the refractive index at 20 °C or 25 °C (especially for pharma) are of interest. Some manufacturers also state Saybolt viscosity.

2 Which instrument is used?

For these tests, the viscometer SVM™ 3001 was used. The instrument features a viscosity and a density measuring cell which are filled in one go. SVM™ 3001 measures viscosity according to ASTM D7042 and density according to ASTM D4052 respectively ISO 12815.

Moreover, viscosity index, temperature scans, API calculations of density over temperature as well as the calculation of other parameters like Saybolt viscosity can be selected as output value. SVM™ 3001 can further be combined with a refractometer of Anton Paar’s Performance line, Performance Plus line or Heavy Duty line.

Users can choose from four different precision classes depending on their sample and the precision needs, which also impacts the duration of the measurement.

For single point measurements in a limited temperature range and without density measurement according to ASTM D4052, the SVM™ 2001 may also be sufficient.

3 Which samples are tested?

Six silicone standard oils and three oils for different applications were tested:

<table>
<thead>
<tr>
<th>Sample</th>
<th>Purpose / Substance</th>
</tr>
</thead>
<tbody>
<tr>
<td>RT 5, RT 10, RT 50, RT 100, RT 500, RT 1000</td>
<td>Anton Paar’s viscosity standards Polymethyl silicones</td>
</tr>
<tr>
<td>AK 100 Silicone Fluid</td>
<td>e.g. lubricant for applications at elevated temperatures Polymethyl silicone</td>
</tr>
<tr>
<td>Silicone Oil M3</td>
<td>Low viscosity silicone oil used e.g. as heat transfer fluid in cooling thermostats Polymethyl silicone, stabilised</td>
</tr>
<tr>
<td>Unisilikon TK 002/1000</td>
<td>e.g. lubricant within machinery for food industry Liquid based on methyl silicones</td>
</tr>
</tbody>
</table>
4 Measuring the samples

4.1 Instrument setup

4.1.1 Method settings

Measurements according to ASTM D7042.
- Method: SVM Standard
- Measurement mode: Repeated
- Precision class: Precise
- RDV: 0.10 %
- RDD: 0.0002 g/cm³
- Automatic prewetting: yes

4.1.2 Rotor bearings

To measure silicone based fluids successfully, it is required to exchange the standard ceramic rotor bearings for the PEEK rotor bearings. They are an option for measuring samples with poor lubricity as they provide better self-lubrication and therefore allow a smooth operation. With the standard ceramic rotor bearings, a lot of silicone oils tend to have stability issues in the long term. For replacement refer to the instruction supplied with the PEEK bearings, further to the SVM™ X001 Reference Guide (Customer Maintenance). An adjustment is usually not required, but a calibration measurement is highly recommended.

4.2 Calibration

Use only a calibrated instrument. The calibration shall be performed periodically using certified reference standards. According to ASTM D7042, the reference standards shall be certified by a laboratory, which meets the requirements of ISO/IEC 17025 or a corresponding national standard. Viscosity standards should be traceable to master viscometer procedures. The uncertainty for density standards must not exceed 0.0001 g/cm³. For each certified value the uncertainty should be stated (k = 2; 95 % confidence level). Use one or more standard(s) in the viscosity range of your oil sample(s). If required, apply a calibration correction to improve the reproducibility. To perform the calibration, refer to the SVM™ X001 Reference Guide.

4.3 Sample preparation

If the sample is not freshly drawn from a production line or elsewhere, homogenizing the test specimen might improve the measurement repeatability.

Proceed as follows: Fill approx. 100 mL of sample into a glass beaker; cover it with a laboratory film to avoid contamination and stir the sample on a magnetic stirrer at low speed for approx. 5 minutes.

4.4 Filling

Single-use syringes with a volume of 5 mL are recommended, 10 mL syringes would serve more sample volume for refills, if required.

Ensure that the measuring cells are leak tight, clean and dry.

Fill at least 1.5 mL as first filling. After prewetting refill at least 1 mL or until the sample in the waste hose is free of bubbles. The typical amount for valid results is 4 – 5 mL (including prewetting and refills), where the volume can vary depending on the sample.

4.5 Cleaning

4.5.1 Solvents

 Depending on the type, silicone oils are highly soluble in aromatic hydrocarbon solvents such as toluene, further in aliphatic hydrocarbon solvents such as petroleum benzine, as well as in chlorinated hydrocarbon solvents. Some of them are also soluble in alcohols. If not sure, test the solubility before filling the sample into the cells.

For the tested samples, a mixture of toluene and isopropyl alcohol (50:50) was used as cleaning solvent. As second solvent, heptane showed better drying performance.

In any case, ensure that the used solvent starts boiling at a temperature significantly higher than the measuring temperature. Otherwise insufficient cleaning of the measuring cells may impact the results. Further, the solvent must dry up completely at measuring / cleaning temperature. A single solvent needs to meet the quality “chemically pure” or “for synthesis”. If using more than one solvent, this applies for the finally used (drying) solvent. Typically required solvent volume: 10 – 15 mL / sample.

4.5.2 Cleaning Procedure

- Tap the cleaning button to open the cleaning screen. Observe it during the cleaning procedure.
- Remove the sample from the cells (push through using an air-filled syringe).
- Fill approx. 2 mL of solvent using a syringe and leave the syringe connected.
- Tap the motor speed button to improve the cleaning performance in the viscosity cell. The cleaning screen shows the mixing of solvent and sample residue by change of viscosity. The density value indicates whether the cell is filled properly with solvent. Stop the motor again.
- Move the plunger of the syringe back and forth (motor at filling speed) to improve the cleaning performance both in the density oscillator and in the viscosity cell.

D89IA025EN-A 2 www.anton-paar.com
• Blow air for some seconds through the cells to remove the sample-solvent-mixture.
• Repeat the procedure until the liquid has reached approximately the solvent’s viscosity while the motor is turning at high speed. Silicone oils often need more cleaning effort than petroleum based lube oils, so it may be required to repeat the procedure several times.
• Perform a final flush with a drying solvent to remove any residues.
• Observe the cleaning screen. Dry the measuring cells until the cleaning value turns green and stays steadily green for both cells.
For details see the SVM™ X001 Instruction Manual.

5 Results
For all tested samples, repeat measurements were performed according to ASTM D7042. Based on the valid results (n = 11), the mean value and standard deviation is calculated and displayed in the tables below. Looking at the standard deviation, the samples show a good repeatability.

5.1 Silicone viscosity standard oils

Kinematic viscosity at 25 °C (77 °F):

<table>
<thead>
<tr>
<th>Sample</th>
<th>ASTM D7042 Kin.vis. [mm²/s] (measured)</th>
<th>ASTM D445 Kin.vis. [mm²/s] (reference)</th>
<th>Dev. to D445 %</th>
<th>Max. allowed dev. %</th>
<th>Std. dev. r (2 n) %</th>
</tr>
</thead>
<tbody>
<tr>
<td>RT 5</td>
<td>5.6724</td>
<td>5.6350</td>
<td>0.67</td>
<td>±0.75</td>
<td>0.02</td>
</tr>
<tr>
<td>RT 10</td>
<td>10.880</td>
<td>10.860</td>
<td>0.18</td>
<td>±0.76</td>
<td>0.03</td>
</tr>
<tr>
<td>RT 50</td>
<td>53.362</td>
<td>53.180</td>
<td>0.34</td>
<td>±0.77</td>
<td>0.01</td>
</tr>
<tr>
<td>RT 100</td>
<td>104.22</td>
<td>104.10</td>
<td>0.12</td>
<td>±0.77</td>
<td>0.26</td>
</tr>
<tr>
<td>RT 500</td>
<td>528.93</td>
<td>526.00</td>
<td>0.56</td>
<td>±0.78</td>
<td>0.02</td>
</tr>
<tr>
<td>RT 1000</td>
<td>1064.9</td>
<td>1058.0</td>
<td>0.65</td>
<td>±0.79</td>
<td>0.44</td>
</tr>
</tbody>
</table>

Table 1: Kinematic viscosity of silicone oils at 25 °C

Dynamic viscosity at 25 °C (77 °F):

<table>
<thead>
<tr>
<th>Sample</th>
<th>ASTM D7042 Dyn.vis. [mPa.s] (measured)</th>
<th>ASTM D445 Dyn.vis. [mPa.s] (reference)</th>
<th>Dev. to D445 %</th>
<th>Max. allowed dev. %</th>
<th>Std. dev. r (2 n) %</th>
</tr>
</thead>
<tbody>
<tr>
<td>RT 5</td>
<td>5.1877</td>
<td>5.1540</td>
<td>0.65</td>
<td>±0.75</td>
<td>0.02</td>
</tr>
<tr>
<td>RT 10</td>
<td>10.161</td>
<td>10.140</td>
<td>0.21</td>
<td>±0.76</td>
<td>0.03</td>
</tr>
<tr>
<td>RT 50</td>
<td>50.047</td>
<td>49.880</td>
<td>0.33</td>
<td>±0.77</td>
<td>0.01</td>
</tr>
<tr>
<td>RT 100</td>
<td>100.42</td>
<td>100.30</td>
<td>0.12</td>
<td>±0.77</td>
<td>0.27</td>
</tr>
<tr>
<td>RT 500</td>
<td>501.25</td>
<td>498.60</td>
<td>0.53</td>
<td>±0.78</td>
<td>0.01</td>
</tr>
<tr>
<td>RT 1000</td>
<td>1013.1</td>
<td>1007.0</td>
<td>0.61</td>
<td>±0.79</td>
<td>0.44</td>
</tr>
</tbody>
</table>

Table 2: Dynamic viscosity of silicone oils at 25 °C

The results were obtained with the default full range adjustment, without any calibration correction points and without density adjustment. For details on the adjustment see the SVM™ X001 Instruction Manual.

The absolute deviation between the SVM™ 3001 (ASTM D7042) results and the reference values (ASTM D445) were within the allowed tolerance band. This tolerance band includes the measurement uncertainty of SVM™ of ±0.35 % and the uncertainty of the oil, which was taken from the certificates.

5.2 Ready to use silicone oils
These oils show usually no specification values but a core viscosity at a certain temperature (commonly 25 °C) combined with a certain tolerated deviation. Depending on the oil’s purpose, this is often ±5 to 10 %. So for the measured oil, this tolerance window stated on the data sheet (where applicable) is used as reference. The results are checked whether they are within this tolerance band or not.

Kinematic viscosity at 25 °C (77 °F):

<table>
<thead>
<tr>
<th>Sample</th>
<th>ASTM D7042 Kin.vis. [mm²/s] (measured)</th>
<th>ASTM D445 Kin.vis. [mm²/s] (typical range from data sheet)</th>
<th>Deviations within permitted range</th>
<th>Std. dev. r (2 n) %</th>
</tr>
</thead>
<tbody>
<tr>
<td>AK 100</td>
<td>103.31</td>
<td>95 … 105</td>
<td>yes</td>
<td>0.04</td>
</tr>
<tr>
<td>Silicone Oil M3</td>
<td>3.228</td>
<td>2.7 … 3.3</td>
<td>yes</td>
<td>0.09</td>
</tr>
<tr>
<td>Unisilkon TK 002 1000</td>
<td>1394.0</td>
<td>n.a.</td>
<td>n.a.</td>
<td>0.04</td>
</tr>
</tbody>
</table>

6 Conclusion
SVM™ 3001 in combination with PEEK rotor bearings is perfectly suited for determining the kinematic viscosity and density of silicone oils. Please ensure that equipment and settings are in accordance with this report (see section 4, “Measuring the samples”). Viscosity results are obtained according to ASTM D7042 and show good repeatability.

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