Rheology of Lubricating Greases According to DIN 51810

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Figure 1. The Thermo Scientific HAAKE MARS iQ Rheometer

Introduction

Lubricants reduce the friction between moving parts of machinery, thereby contributing to their reliable and efficient use by reducing wear and energy consumption. Compared to lubricating oils, lubricating greases are soft solids at rest, which deliver long-lasting lubrication without dripping. They consist of a base oil, thickeners to adjust mechanical stability, and additives for example to reduce wear and/or corrosion. With increasing shear, the properties of greases increasingly resemble the properties of their base oil. The rheological characterization of lubricating greases therefore mainly focusses on their viscosity under shear and their mechanical stability at rest.

The German industry standard DIN 51810 describes methods to perform rheological measurements on lubricating greases with a rotational/oscillatory rheometer and to evaluate the results. This report describes the methods of part 1, 2, and 4, and shows example data measured with a Thermo Scientific™ HAAKE™ MARS™ iQ Air Rheometer (Figure 1) equipped with a Peltier temperature module. Part 3 is not covered here, since it is based on a different measuring principle.

All Thermo Scientific™ HAAKE™ RheoWin™ jobs, which have been used for the measurements done for this report, are part of the HAAKE RheoWin Job Library. The results shown in this report are based on measurements of the same lithium soap grease.

Part 1: Shear Viscosity

Part 1 describes the measurement of the shear viscosity performed using a cone-plate measuring geometry C25/1° at a temperature of 25 °C. This method is suitable for NLGI grade 000 to 2 greases. These grades have been defined by the National Lubricating Grease Institute (USA) to classify greases according to their hardness with higher grade numbers representing harder greases.

After sample loading, adjusting the measuring gap, and reaching thermal equilibrium in the sample, a preshear of 100 s-1 over 1 min was applied to "erase" the effects of sample handling and sample loading. After a rest time of 2 min the shear rate was increased linearly from 0 s-1 to the shear rate selected for the measurement within 1 min.

For NLGI grades 000 to 1, a constant shear rate of 1000 s-1 is defined. For NLGI grade 2 the shear rate is reduced to 500 s-1. This constant shear rate was maintained for 5 min (Figure 2).

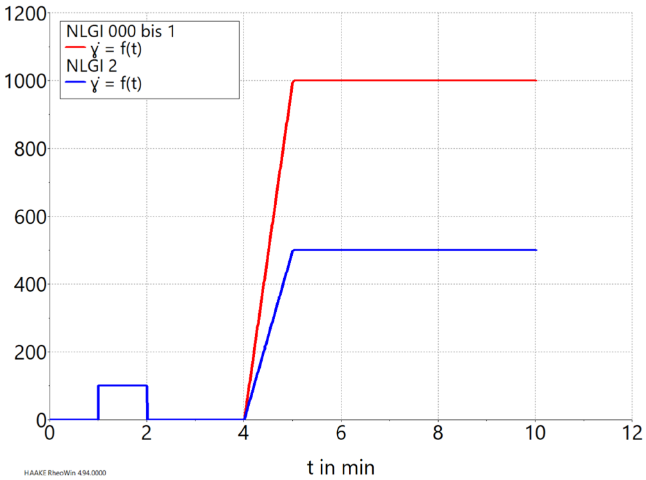

Figure 2. Shear rate profiles according to DIN 51810-1 for NLGI 000 to 1 (red) and NLGI 2 (blue)

For the evaluation, only the data from the 5 min of constant shear at the end of the measurement was used (Figure 3).

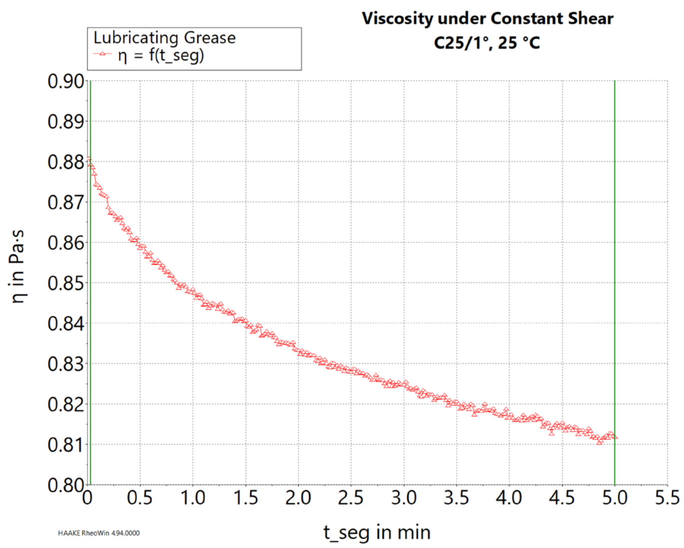

Figure 3. Shear viscosity of a Lithium soap grease during 5 min of constant shear at 25 °C

The viscosity at the beginning ηb is determined 2 s after the start of the constant shear. The last value at 300 s is the final viscosity ηf. Based on these 2 values, the relative change in viscosity ηrel was calculated according to this equation:

$$\eta_{rel} = \frac{\eta_b - \eta_f}{\eta_b} \cdot 100 \quad [\%]$$

Based on the data shown in Figure 3 the following results were calculated:

η_b = 0.879 Pas

η_f = 0,812 Pas

η_{rel} = 7.6 %

Apart from the absolute viscosity values η_b and η_f, η_{rel} contains important information about the shear stability of a lubricating grease—for example, if it will remain stable under shear and in place.

Part 2: Flow Point

Part 2 describes the rheological determination of the flow point with a 25 mm plate-plate measuring geometry with a measuring gap of 1 mm at 25 °C and -40 °C. This method is suitable for NLGI grade 0 to 2 grade greases. This DIN standard was transferred to ISO 13227.

The measurement at -40 °C requires extra care and time since sample loading and gap setting happen at around room temperature to avoid condensation and ice formation on the measuring surfaces. Afterwards, the sample temperature was reduced to -40 °C with a cooling rate of 0.4 °C/min followed by an equilibration time of 30 min.

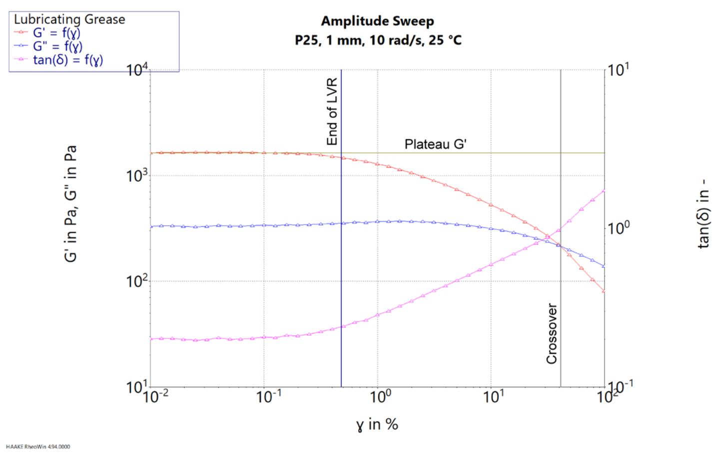

Figure 4. Example for the results of an amplitude sweep on a Lithium soap grease at 20 °C plotted over the deformation γ in %. The lines indicate the crossover point, the end of LVR and the plateau of G'.

The amplitude sweep can be run with controlled deformation (CD) or controlled stress (CS). In most cases, the CD mode is preferred to maintain better control over the measurement independent from the sample's properties. The evaluation focuses mainly on the calculation of characteristic stress values from the results. For this purpose, DIN 51810-2 recommends plotting the results as a function of the stress. Since the HAAKE RheoWin Software can calculate these stress values independent of the parameter on the X-axis, plotting the data as a function of the deformation is possible as well as shown in Figure 4.

The main evaluation parameter is the so-called flow point τ_f, the stress at the crossover of the storage and the loss modulus (G' and G'') in the non-linear range. Several other evaluation variables have been proposed, of which the so-called yield point τ_y—the stress at the end of the linear viscoelastic range (LVR)—is the most important. With these two values, the calculation of the flow transition index τ_f/τ_y is possible. The smaller this value becomes, the more brittle the grease is.

The evaluation of the data from Figure 4 shows that the sample tested was a soft grease with a small yield stress and a broad transition range:

$\tau_f = 123.8$ Pa

$\tau_y = 7.3$ Pa

$\tau_f/\tau_y = 16.9$

Part 4: Consistency of lubricating greases

Part 4 describes a method for determining the consistency of metal saponified lubricating greases and their assignment to an NLGI grade based on rheological data collected at 25 °C with an oscillatory rheometer using a cone-plate measuring geometry C25/1°. This method has been developed for NLGI-grade 00 to 3 greases. This DIN standard was transferred to ISO 13511.

Traditionally, the NLGI grade is determined with a worked penetration test according to ASTM D217, which requires 500 ml sample per test. The sample size for a single measurement with the C25/1° is only 0.08 ml. Sample preparation is therefore less time consuming, especially since working the sample before the measurement is not required for this method.

The rheological measurement is a CD amplitude sweep with the deformation starting at 0.01% and increasing to 1000 % with 10 points per order of magnitude at a constant angular frequency of 10 rad/s.

Based on the results, the HAAKE RheoWin software calculates the effective shear rates ($\dot{\gamma}_{eff}$) and the effective shear stresses (τ_{eff}) for each datapoint. For an oscillatory measurement, the shear rate ($\dot{\gamma}$) and the shear stress (τ) are calculated for the amplitude of the oscillation. The effective values of both parameters are calculated analogue to the approach used in electrical engineering by multiplying the amplitude values by a factor of $1/\sqrt{2} \approx 0.71$.

τ_{eff} is plotted as a function of $\dot{\gamma}_{eff}$ and a linear function is fitted to the last 4 datapoints. i.e., the ones with the highest values (see Figure 5).

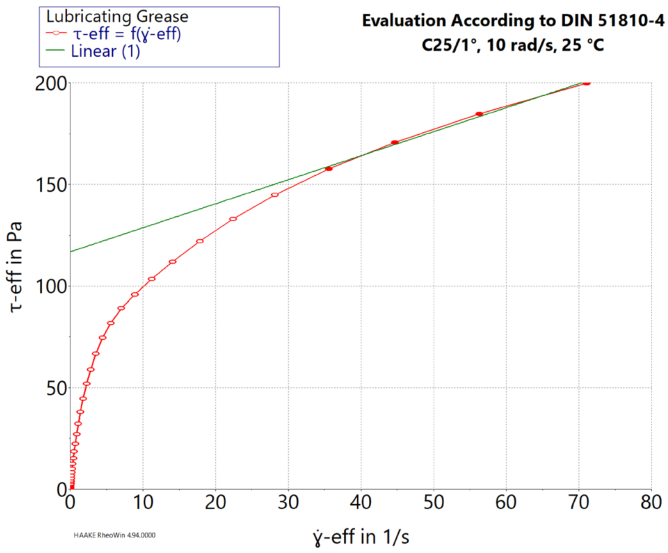

Figure 5. Example for the evaluation according to DIN 51810-4 with the HAAKE RheoWin software. The green line has been fitted to the last 4 datapoints and its y-intercept was calculated.

The y-intercept of the linear fit function is converted into a consistency value using the following formula:

Consistency [0.1 mm] = -59.666 * ln(y-intercept) + 680.89

The calculated consistency is comparable to a worked penetration and therefore can be used to determine the NLGI grade. For the Lithium soap grease, the evaluation shown in Figure 5 results in the following values:

y-intercept = 116.8 Pa

consistency [0.1 mm] = 397 × 0.1 mm

NLGI grade 00 to 0

Conclusions

The rheological methods of DIN 51810 allow to quantify the viscosity, the stability, and the consistency of lubricating greases. For the latter, significantly less sample volume is required for the rheological determination method compared to the traditional penetration method. All measurements have been performed on a HAAKE MARS iQ Air Rheometer using predefined HAAKE RheoWin test protocols from the software's method library. The user-friendly design of rheometer and software make the HAAKE MARS iQ Air Rheometer the perfect tools for the characterization of lubricating greases.

Literature

ASTM D217-21a: Standard Test Methods for Cone Penetration of Lubricating Grease

DIN 51810-1:2017-04: Testing of lubricants - Testing rheological properties of lubricating greases - Part 1: Determination of shear viscosity by rotational viscosimeter and the system of cone/plate

DIN 51810-2:2017-04: Testing of lubricants - Testing rheological properties of lubricating greases - Part 2: Determination of flow point using an oscillatory rheometer with a parallel-plate measuring system

DIN 51810-3:2016-08: Testing of lubricants - Testing of rheological properties of lubricating greases - Part 3: Determination of flow point with inclining rod method

DIN 51810-4:2021-04: Testing of lubricants - Determination of the consistency of metal saponified lubricating greases using an oscillatory rheometer with a cone/plate system

ISO 13227:2025-03 Petroleum products and lubricants - Rheological properties of lubricating greases - Determination of flow point using an oscillatory rheometer with a parallel-plate measuring system

ISO 13511:2025-03 Petroleum products and lubricants - Rheological properties of lubricating greases - Determination of the consistency of greases with metal soap thickener by an oscillatory rheometer with a cone/plate measuring system

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