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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

Produits pétroliers et lubrifiants — Propriétés rhéologiques des graisses lubrifiantes — Détermination de la consistance des graisses avec épaississant à base de savon métallique au moyen d'un rhéomètre oscillant de type cône/plan

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Foreword

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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

1 Scope

This document specifies a procedure to determine the consistency of a metal-saponified lubricating grease by an oscillatory rheometer.

This test method is applicable for fresh, as well as used, lubricating greases where only small quantities of the grease are present and the worked penetration that is usually used cannot be determined due to the small quantity.

The determined calibration is only valid for metal-saponified lubricating greases like lithium, lithium-calcium and, also, lithium- and calcium complex.

The method described in this document is applicable for lubricating greases with NLGI grades 00, 0, 1, 2 and 3 according to ISO 6743-99.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 23572, *Petroleum products — Lubricating greases — Sampling of greases*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
shear deformation
shear strain
γ
deformation of the sample generated by tangential deflection

Note 1 to entry: The shear deformation is dimensionless. It is often given in per cent (%), where 100 % = 1.

3.2
loss modulus
G''
component of *shear stress* (3.3) which exhibits a phase shift of $\delta = \pi/2 = 90°$ to the *shear strain* (3.1)

Note 1 to entry: The loss modulus represents the viscous part of the viscoelastic behaviour of the sample and is given in pascals (Pa).

[SOURCE: ISO/DIS 13227:2024, 3.4]

3.3
shear stress
τ
quotient of tangential force to area

Note 1 to entry: The unit of the shear stress is Pascal (Pa).

3.4
shear rate
$\dot{\gamma}$
change in *shear deformation* (3.1) over time

Note 1 to entry: The unit of the shear rate is reciprocal seconds (s^{-1}).

4 Symbols

For the purposes of this document, the symbols listed in Table 1 apply.

Table 1 — Symbols

Symbol	Designation	Unit
τ_{calc}	shear stress, calculated by the loss modulus	Pa
τ_0	shear stress, at shear rate 0 s^{-1}	Pa
$\dot{\gamma}_{calc}$	shear rate, calculated by the shear deformation	s^{-1}
γ	shear deformation	%
G''	loss modulus	Pa
τ	shear stress	Pa
$\dot{\gamma}$	shear rate	s^{-1}

5 Principle

A lubricating grease sample is tested at 25 °C with an oscillatory test using a cone-plate measuring system.

The loss modulus is determined as a function of the shear deformation. The worked penetration and the NLGI grade can then be determined from these values.

6 Reagents and materials

6.1 Solvent, suitable to clean the equipment without any residue.

7 Apparatus

Usual laboratory apparatus and glassware, together with the following shall be used.

7.1 Oscillatory rheometer, air-bearing, equipped with a cone/plate measuring system.

7.2 Cone/plate measuring system, with a cone diameter of 25 mm, Ra roughness of 0,8 µm, an angle of (1,00 ± 0,02)°, and which can be tempered to ±0,1 °C.

7.3 Temperature control unit, allowing the temperature of the sample to be adjusted within ±0,1 °C.

8 Sampling

Sampling shall be conducted in accordance with ISO 23572.

9 Procedure

9.1 Cleaning of the measuring system

Before commencing the test, any residue shall be removed from the cone/plate system with a suitable solvent and afterwards the system shall be dried.

9.2 Tempering

The temperature of the measuring arrangement shall be 25 °C at the start of the test.

9.3 Setting the measuring gap and filling the measuring system cone/plate

After the temperature control has taken place, the zero point (based on the distance between the cone and the plate) shall be set (manually or automatically with the aid of a suitable measuring programme).

The distance shall be set at the measuring temperature, because the gap has a big influence on the measurement results.

Using a spatula, a sufficient amount of grease (with a small excess) shall be applied to the centre of the plate, free of air bubbles. The required amount of grease shall be taken from the operating instructions of the measuring system. The sample shall not be manually distributed on the plate before the cone is lowered, but automatically distributed when the measuring gap is approached. To fill the gap correctly, a measuring gap of 20 % above the final measuring gap shall be approached first. This position is often specified by the device manufacturer, the so-called trimming position. Then the excess lubricant on the edge of the cone/plate shall be wiped off. Afterwards, the measuring gap (as specified by the device manufacturer) shall be approached. The device-specific distance between the cone and the plate has a significant influence on the measurement result and shall therefore be set carefully.

When taking samples, bear in mind that inhomogeneities, impurities and trapped air bubbles can falsify the measurement results. It can therefore be necessary to evaluate the sample in this respect prior to the measurement. Furthermore, in case that the maximum particle size of solid and hard particles is larger than 1/10 of the measuring gap, they can even damage the measuring system.

The rheological behaviour of lubricating greases strongly depends on the previous treatment. Storage over weeks and months can change the lubricating grease. Lubricating greases often become harder if separation of oil has occurred. Also, this can falsify the measurement results.

9.4 Measurement

After properly filling the measuring system with the lubricating grease sample, the measurement programme in Table 2 shall be conducted.

Table 2 — Amplitude test with a given shear deformation (strain sweep)

Rheological parameter	Part 1 Tempering and relaxation	Part 2 Measurement of strain sweep
shear deformation, γ	-	0,01 to 1 000 %
rate of increase of shear deformation	-	logarithmic with 10 points per decade
angular frequency, ω	-	10 rad·s^{-1}
measurement point duration	-	no specification[a]
part duration	10 min	no specification[a]
test temperature, T	25 °C	25 °C

[a] For oscillating tests, the total duration of a measuring part is usually not given by the operator but automatically calculated by the measurement programme.

10 Evaluation

For the evaluation of the measurement data for each recorded measurement point, the shear deformation shall be available in per cent (%) and the loss modulus G'' in pascals (Pa). The data will be transferred to a spreadsheet calculation programme (see Table A.1, column B and column C). A shear rate, $\dot{\gamma}$, in reciprocal seconds (s^{-1}) is then calculated from the shear deformation value in per cent (%). This involves multiplying the shear deformation value in per cent (%) by 0,071 (see column D in Table A.1).

NOTE 1 For a better understanding of the evaluation steps in Clause 10, see Table A.1.

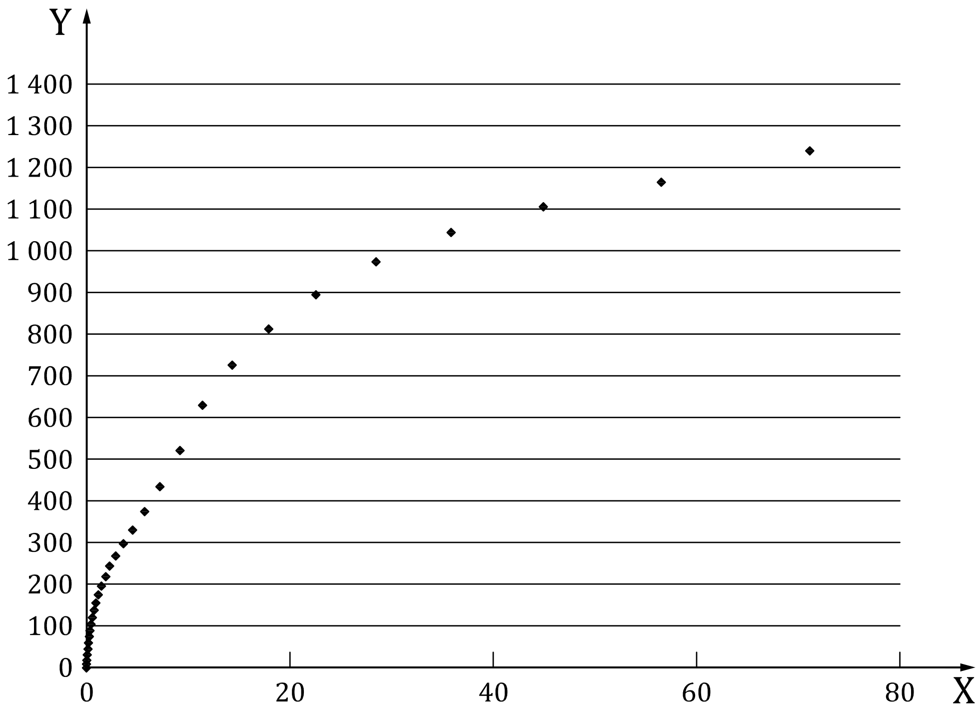
NOTE 2 An explanation and the derivation of the factor 0,071 is shown in Annex A.

At the same time, the elastic component of the shear stress value is calculated by dividing the loss module value by 10 and multiplying this by the shear deformation value and 0,071 (see column E in Table A.1).

Then a diagram (see example in Figure 1), is generated from the calculated data for the shear rate $\dot{\gamma}_{calc}$ in s^{-1} and the shear stress τ_{calc} in Pa (see column D and column E in Table A.1).

NOTE 3 Despite the logarithmic specification in Part 2 of Table 2, linear scaling of the axes is selected in the diagram (Figure 1). This is done solely for the purpose of better optics.

NOTE 4 The derivations of $\dot{\gamma}_{calc}$ and τ_{calc} is shown in Annex A.



Key

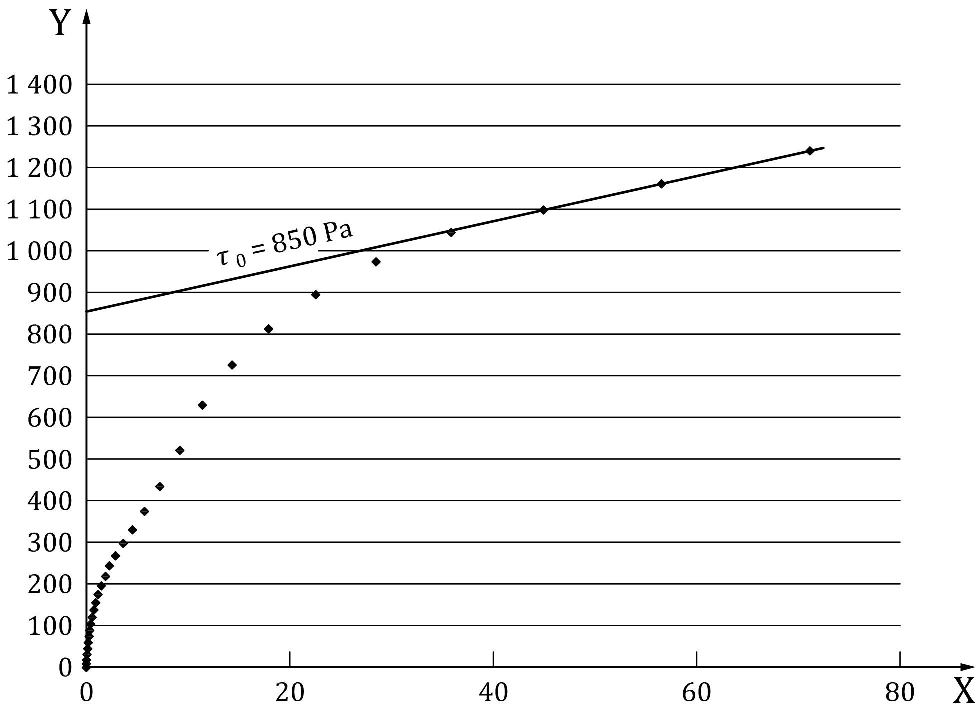
X calculated shear rate, s^{-1}

Y calculated shear stress, Pa

Figure 1 — Graphical representation of the data

After the data has been plotted, a straight line of best fit shall be drawn through the last four data points and the y-value for x = 0 belonging to the straight line of best fit read off. This corresponds to the y-axis intercept of the regression line and is a shear stress value (with the dimension Pa).

The regression line and its y-axis intercept may be inserted manually as shown in Figure 2, or with the help of a computer program (e.g. spreadsheet calculation program) as shown in Figure 3. The determined y-axis intercept is referred to as the τ_0 value.



Key

X calculated shear rate, s^{-1}

Y calculated shear stress, Pa

NOTE The regression line was drawn manually.

Figure 2 — Graphical representation of the evaluation (manual)