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Petroleum and related products — Determination of the shear stability of polymer-containing oils using a diesel injector nozzle

Pétrole et produits connexes — Détermination de la stabilité au cisaillement de fluides contenant des polymères au moyen d'un injecteur pour moteur diesel



Reference number ISO 20844:2004(E)

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ISO 20844:2004(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 20844 was prepared by Technical Committee ISO/TC 28, Petroleum products and lubricants.

Petroleum and related products — Determination of the shear stability of polymer-containing oils using a diesel injector nozzle

WARNING —The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method to assess the resistance to shear stresses applied to mineral oils, synthetic oils and other fluids containing polymers, when passed through a specified diesel injector nozzle. The shear stability is measured by the change in viscosity of the fluid under test, brought about by the polymer degradation during stress. Under normal circumstances, this International Standard is applied to hydraulic fluids of categories HR and HV as defined in ISO 6743-4 ([1] in the Bibliography) and specified in ISO 11158 ([2] in the Bibliography), but it may also be applied to fire-resistant hydraulic fluids within categories HFA, HFB, HFC and HFD, with modified conditions as specified in ISO 12922 ([3] in the Bibliography).

No formal correlation has been established between the viscosity loss, or the absence of viscosity loss, obtained using the procedures described in this International Standard and that of oils and fluids in actual service. However, it provides standardized conditions for the evaluation of polymer stability under minimized thermal and oxidative stresses. It is normally used by manufacturers of fluids and additives, and users, as a means of ranking existing and potential formulations.

NOTE Changes to properties other than viscosity are specified in some specifications, but these are not covered by the procedures specified in this International Standard.

2 Normative references

The following referenced documents are indispensable for the application 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 3104:1994, Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity

ISO 3170:2004, Petroleum liquids — Manual sampling

3 Terms and definitions

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

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3.1

shear stability

percentage loss in viscosity at 40 °C of an oil or fluid subjected to the mechanical shear stresses specified in this International Standard

NOTE Viscosity measurements at other temperatures may be specified by some users.

3.2

calibration pressure

recorded gauge pressure when the specified calibration fluid is within a viscosity loss range of $2.7 \text{ mm}^2/\text{s}$ to $3.0 \text{ mm}^2/\text{s}$ at $100 \,^{\circ}\text{C}$

4 Principle

A test portion of the oil or fluid under test is pumped through a diesel injector nozzle by means of a diesel fuel pump, at high pressure to exert shear stresses. After a specified number of passes, the viscosity of the stressed oil or fluid is measured and compared to that of the unstressed oil or fluid. The percentage loss in viscosity, corrected by a factor to account for the calibration procedure, is the shear stability of the material under test.

5 Reagents and materials

- **5.1** Calibration fluid, of calibration oil grade RL 34 ¹⁾.
- **5.2 Diesel fuel**, of commercial grade, if required for adjustment of the injector nozzle valve opening pressure (see 8.1).

NOTE Precalibrated injector nozzles are normally used.

6 Apparatus

6.1 Test rig

The test rig is described in Annex A²). It shall be located in a position where the ambient temperature is between 20 °C and 25 °C, and shall be supplied with a supply of circulating cooling water regulated for flow.

6.2 Viscosity measuring apparatus

Viscometers and baths shall conform to ISO 3104. Bath temperatures of at least 40 °C and 100 °C are required.

¹⁾ Reference oil RL 34 may be obtained from Kraft-und Schmierstofftechnik, 6702 Bad Durkheim, Bruchstr 24, Germany. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

²⁾ Test rig components may be obtained from HEA-HMB Elektro-Apparate GmbH, Eidelstedter Weg 255 A, D-25469 Halstenbek, Holstein, Germany. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

7 Samples and sampling

- **7.1** Unless otherwise specified, samples shall be taken in accordance with the procedures specified in ISO 3170 or an equivalent national standard.
- **7.2** Laboratory samples shall be of a minimum volume of 600 ml, and shall be clear of undissolved water and particulate, which may damage the injector nozzle. Filtration, or removal by centrifuge is allowable.

8 Preparation of test rig

- **8.1** If a new uncalibrated nozzle is to be used, adjust the diesel injector nozzle, using diesel fuel (5.2) and a nozzle tester, so that the opening pressure under static conditions is 17,5 MPa. If a precalibrated nozzle is used, set the opening pressure to that specified, which shall be between 17,0 MPa and 18,0 MPa.
- **8.2** Adjust the connecting pipe between the lower reservoir and the pump so that the dead volume between the stopcock and the outlet of the nozzle is 20 ml \pm 0.5 ml.
- **8.3** Connect the cooling water to the cooling vessel and carry out three flushing runs with the material to be tested. Carry out the first two flushing runs with approximately 50 ml of fluid, and the third with approximately 170 ml. Once the test rig has warmed up during the flushing runs, adjust the cooling water rate to give a constant temperature of between 30 °C and 35 °C in the fluid in the lower reservoir.
- **8.4** During the third flushing run, and after a minimum of 3 min running time, set the flow rate to $175 \, \text{ml/min} \pm 5 \, \text{ml/min}$ by means of the regulator screw. Bleed the rig of air by means of the bleed screw during this period. When conditions are stable, check the number of pump strokes per minute at least three times, using the stroke counter and a timer for runs of 30 s, 60 s and 180 s. Record the mean of these results as n.
- **8.5** Drain the fluid from the spray chamber and storage reservoir via the three-way stopcock, leaving the same dead volume of fluid between the stopcock and the nozzle outlet as is specified in 8.2.

9 Calibration

- **9.1** When installed, at the replacement of a nozzle, and at intervals not exceeding three months or 5 000 cycles, calibrate the test rig with calibration fluid (5.1) to obtain a correction factor, F, to be applied to the test results. The calibration fluid should be supplied with a certified viscosity loss after 30 cycles within the range 2,75 mm²/s and 2,85 mm²/s at 100 °C, and a nominal value of 2,80 mm²/s can be used for the calculation of the calibration factor, F.
- **9.2** If the first calibration run gives a viscosity loss at 100 °C outside the range of 2,50 mm²/s to 3,10 mm²/s, adjust the injector pressure upwards for low values or downwards for high values, but keeping within the range 17,0 MPa to 18,0 MPa, and repeat the test. If adjustment does not result in the viscosity loss being brought within the limiting values, nozzle preconditioning, or nozzle, filter or pump parts replacement, will be necessary.
- **9.3** Calculate the calibration factor, *F*, using the following equation:

$$F = \frac{\lambda_2}{\lambda_3}$$

where

- λ_2 is the nominal or certified viscosity loss at 100 °C after 30 cycles with the calibration fluid (2,8 mm²/s);
- λ_3 is the determined viscosity loss at 100 °C after 30 cycles with the calibration fluid.

10 Procedure

- 10.1 Measure the kinematic viscosity at 40 °C of the oil or fluid to be tested, in accordance with ISO 3104.
- NOTE Some users may specify other, or additional, temperatures of viscosity measurements.
- **10.2** Carry out two separate determinations of the shear procedure (see 10.3 to 10.6).
- **10.3** Place 200 ml of the material to be tested in the lower reservoir, using the graduation marks as a guide, and run the pump with the three-way stopcock open until 50 ml has been displaced and discarded. Close the stopcock.
- NOTE This procedure displaces the dead volume left in the test rig from the flushing procedure.
- **10.4** Set the stroke counter to $250 \times n$ (see 8.4) to perform 250 cycles. Open the three-way stopcocks.
- NOTE Some users specify a lower number of test cycles, or a number related to the material under test.
- **10.5** After approximately ten cycles, check that the temperature of the oil or fluid is maintained at 30 °C to 35 °C. Adjust the cooling water flow rate if necessary.
- **10.6** At the end of the test period, measure the kinematic viscosity at 40 °C of the sheared fluid in accordance with ISO 3104, using the same viscometer tube as was used for the initial measurement (see 10.1).

11 Calculation

11.1 Calculate the shear stability, *SS*, expressed as a percentage change in kinematic viscosity at 40 °C before and after stress, using the following equation:

$$SS = \frac{\lambda_0 - \lambda_1}{\lambda_0} \times 100 \times F$$

where

- λ_0 is the kinematic viscosity at 40 °C of the fluid before shear;
- λ_1 is the kinematic viscosity at 40 °C of the fluid after shear;
- F is the calibration factor (9.3).
- NOTE A low value indicates a high shear stability.

12 Expression of results

- **12.1** Report the average of the two determinations as the corrected result for shear stability to the nearest 0.5 %.
- **12.2** Report the following:
- a) the kinematic viscosity at 40 °C of the unsheared fluid;
- b) the number of cycles;
- c) the calibration factor.

13 Precision

13.1 General

The precision, as determined by statistical examination of interlaboratory test results on hydraulic fluids of categories HR and HV is given in 13.2 and 13.3. The origin of the precision results reported here is from DIN in Germany ³⁾, for shear stability based on 250 cycles, and kinematic viscosity at 40 °C. Precision estimates are available for other materials such as engine oils or hydraulic fluids of category HV, based on different cycle numbers and viscosities at different temperatures.

13.2 Repeatability

The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of the test method, exceed 2,0 % absolute in only one case in 20.

13.3 Reproducibility

The difference between two single and independent test results obtained by different operators working in different laboratories on identical material would in the long run, in the normal and correct operation of the test method, exceed 3,5 % absolute in only one case in 20.

14 Test report

The test report shall contain at least the following information:

- a) a reference to this International Standard;
- b) the type and complete identification of the product tested;
- c) the result of the test (see Clause 12);
- d) any deviation, by agreement or otherwise, from the procedure specified;
- e) the date of the test.

³⁾ DIN 51382:1996 ([4] in the Bibliography) gives further details.

Annex A (normative)

Test rig

A.1 General

The test rig is shown schematically in Figure A.1, and the numbers in parentheses in this text refer to the key in Figure A.1. In principle, the test rig shall consist of a fluid reservoir, a double plunger pump with an electric motor drive, an atomization chamber complete with diesel injector nozzle and holder, and a fluid cooling vessel with controlled-flow circulating water. The parts are described in more detail in Clause A.2.

A.2 Test rig components

A.2.1 Fluid reservoir

The reservoir (7) has an internal diameter of 45 mm ± 2 mm and a capacity of approximately 250 ml. It shall be graduated in 25 ml intervals. Inside the reservoir is fitted a distributor plate (4) of 40 mm diameter to reduce the tendency of fluid channelling. The temperature sensor is fitted such that the temperature is recorded 10 mm to 15 mm above the entrance to the drain tube opening. The outlet is equipped with a three-way stopcock (6) of a cone type with a nominal 8 mm bore size. Plastic tubing (9) connects the stopcock to the pump inlet.

NOTE A watch glass with serrated edges is acceptable as a distributor plate.

A.2.2 Double plunger pump

This consists of a Bosch PE 2 A 90C 300/3 S2266 two-cylinder injection pump (11)⁴⁾, equipped with a stroke counter (13) fitted with an automatic cut-off, venting (bleeding) screw (14) and flow-rate adjusting screw (10). The pump is driven by a 1,1 kW three-phase electric motor (12) rated at a speed of 925 r/min ± 25 r/min. The outlet of the injection pump is connected to the atomization chamber using high-pressure steel tubing.

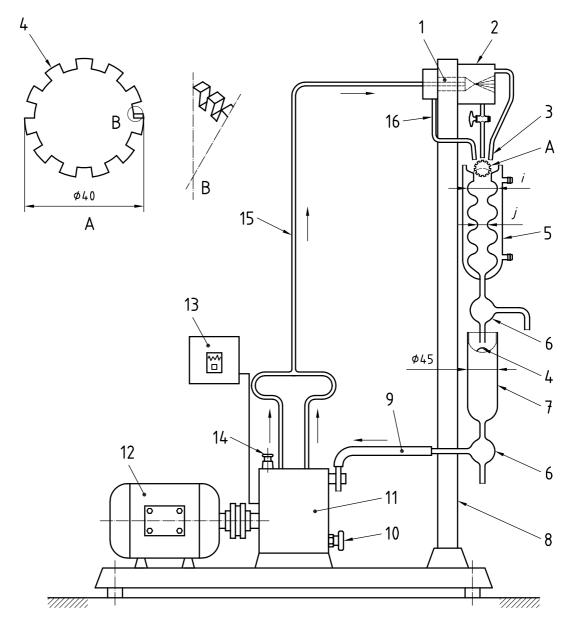
A.2.3 Atomization chamber

The atomization chamber [(2) in Figure A.1] is illustrated in detail in Figure A.2, together with the injector nozzle assembly.

The chamber is designed so that the fluid under test exits the nozzle into fluid to minimize foam generation, and is fitted with an outlet tube [(3) in Figure A.1], and a drain tube [(16) in Figure A.1] fitted with a two-way stopcock. The injector nozzle is a Bosch DN 8 S 2 pintle nozzle injector held in a Bosch KD 43 SA 53/15 nozzle holder 4), which includes a filter cartridge.

⁴⁾ This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

Dimensions in millimetres



Key

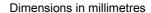
- 1 injector nozzle
- 2 atomization chamber
- 3 outlet of atomization chamber
- 4 distributor plate
- 5 cooling vessel
- 6 three-way stopcock
- 7 fluid reservoir
- 8 support stand

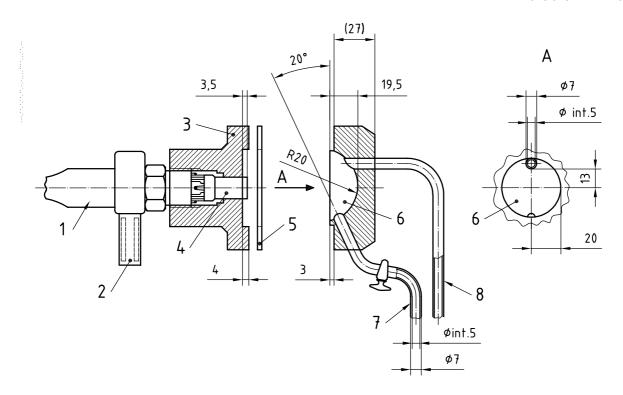
- 9 connecting pipe to pump (plastic tubing)
- 10 pump setting (flow-rate adjusting) screw
- 11 pump
- 12 electric motor
- 13 stroke counter and cut-off
- 14 pump venting (bleeding) screw
- 15 high-pressure tube
- 16 return line for overflow (drain tube)

Figure A.1 — Schematic of test rig

A.2.4 Cooling vessel

The glass vessel (5), with exterior cooling jacket, is constructed so that the heat transfer surface of the jacket is approximately spherical. The exterior diameter of the jacket [(i) in Figure A.1] is approximately 50 mm, the internal diameter [(j) in Figure A.1] approximately 35 mm, with a length of approximately 180 mm. A distributor plate (4), similar to that in the fluid reservoir, is positioned in the upper portion of the cooling vessel to ensure contact between the fluid and cooling surface. The discharge from the cooling vessel is through a three-way stopcock (6). The jacket is supplied with an adjustable flow of cold water.





Key

- seal $(50 \times 80 \times 2)$ 1 nozzle holder 2
- filter cartridge 6 atomization chamber 3 threaded flange 7 drain tube
- spray nozzle outlet tube

Figure A.2 — Atomization chamber and nozzle assembly

Bibliography

- [1] ISO 6743-4:1999, Lubricants, industrial oils and related products (class L) Classification Part 4: Family H (Hydraulic systems)
- [2] ISO 11158:1997, Lubricants, industrial oils and related products (class L) Family H (Hydraulic systems) Specifications for categories HH, HL, HM, HR, HV and HG
- [3] ISO 12922:1999, Lubricants, industrial oils and related products (class L) Family H (Hydraulic systems) Specifications for categories HFAE, HFAS, HFB, HFC, HFDR and HFDU
- [4] DIN 51382:1996, Testing of lubricants Determination of shear stability of lubricating oils containing polymers Method with Diesel injection nozzle, relative viscosity loss due to shear

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