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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

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 28, *Petroleum products and related products of synthetic or biological origin*.

ISO Technical Committee ISO/TC 28 acknowledges permission granted by Co-ordinating European Council to reproduce selected parts of CEC Test Method L-14-93.

This second edition cancels and replaces the first edition (ISO 20844:2004), which has been technically revised with the following changes:

— correction of deficiencies of the former standard and alignment with CEC L-14-93 have been introduced;

— the requirements concerning reagents (Clause 5) has been updated as the former reference fluid is no longer available;

— a requirement on minimum test volume availability (7.2) has been introduced based on field experience;

— the preparation of the test rig (Clause 8 and Annex A) and subsequently the referencing (Clause 9) have been improved and technically revised.

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] and specified in ISO 11158 [2], 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].

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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3104, *Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity*

ISO 3170, *Petroleum liquids — Manual sampling*

ISO 4113, *Road vehicles — Calibration fluids for diesel injection equipment*

3 Terms and definitions

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

3.1
shear stability
mechanically-induced viscosity shear loss at 40 °C of a fluid, expressed as percentage

Note 1 to entry: Viscosity measurements at other temperatures may be specified by some users.

3.2
referencing pressure
recorded gauge pressure when the shear loss in kinematic viscosity at 100 °C of CEC Reference Fluid RL 233 is within the range 2,70 $mm^2·s^{-1}$ to 2,90 $mm^2·s^{-1}$

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 Reference fluid, CEC Reference Fluid RL 233[1)].

5.2 Diesel injection calibration fluid, satisfying the requirements of ISO 4113 for static adjustment of the nozzle opening pressure if required.

NOTE Injector nozzles are conventionally supplied in an un-referenced condition.

6 Apparatus

6.1 Test rig

The test rig is described in Annex A[2)]. 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.

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 A minimum volume of 600 ml of the sample to be tested shall be available. The sample shall be homogeneous and free from visible foreign matter. Particulate matter, which is detrimental to the durability of the injector nozzle, nozzle holder and pump, can be removed by filtration if necessary.

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1) The CEC Secretariat should be contacted at CEC Secretariat, Kellen Europe, Avenue Jules Bordet 142 – 1140 Brussels, Belgium, for further advice and information on the availability of CEC Reference Fluids. 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) The test rig and spare parts for it may be obtained from Hamburg Elektro-Apparate GmbH, Dockenhudener Chaussee 14, D-25469 Halstenbek bei Hamburg, 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.

8 Preparation of test rig

8.1 If a new nozzle is to be used, the opening pressure can be set by either static or dynamic means.

8.1.1 Static method, the opening pressure is determined and adjusted by means of an injector tester under near-static flow conditions. This entails removal of the nozzle holder assembly from the test rig. The fluid used for the adjustment shall be diesel injection calibration fluid satisfying the requirements of ISO 4113[3]. After adjustment and final testing, the nozzle shall be purged as fully as is practicable of residual quantities of the calibration fluid.

8.1.2 Dynamic method, dynamic adjustment of the nozzle opening pressure shall only be attempted during the warm-up run as described in 8.3 using CEC Reference Fluid RL 233.

The rig shall be operational for at least 10 min to achieve a stable operating condition before the pressure is adjusted. The pressure value as indicated on the pressure gauge fitted to the test rig together with the flow rate should be compared to the values recorded for the preceding reference test which failed. Any significant difference between the values is indicative of a failure to achieve a stable operating condition.

Adjustment of the opening pressure is achieved in the same manner as that employed in the static method except that it is conducted with the test rig in operation. Following adjustment of the nozzle opening pressure, the flow rate should be checked and adjusted as necessary. Both values should continue to be monitored throughout the remainder of the warm-up run to ensure a stable operating condition has been achieved. A reference test should then follow to re-validate the test rig.

The opening pressure under dynamic conditions shall be between 13,0 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 ± 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 a 3 minutes running time, set the flow rate to 170 $ml \cdot min^{-1}$ ± 5 $ml \cdot min^{-1}$ by means of the pump-rate adjusting screw. Remove any entrained air from the pressure circuit during this period by means of the vent screw.

The number of cycles may be counted by either a mechanical pulse counter or an electronic revolution counter. In the case of the mechanical system, check the number of pump pulses per minute at least three times using the pulse counter and a timer for runs of 30 s, 60 s, and 180 s. Record the mean of these results as n.

Where an electronic counting system is used, this shall be calibrated at least once annually using traceable standards.

8.5 Drain the fluid from the atomisation 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.

3) Enquiries relating to the availability of diesel injection calibration fluid satisfying the requirements of ISO 4113 should be directed to lubricant distributors or fuel injection service suppliers.

9 Referencing

9.1 The test rig should be referenced according to the procedure detailed in Clause 10 at intervals not exceeding 500 cycles and immediately following any maintenance operation, e.g. the replacement of a nozzle. CEC Reference Fluid RL 233 shall be used for referencing and the reference test shall be of 30 cycles duration. A correction factor, *F*, shall be derived following referencing of the test rig and applied to the test results on candidate fluids. The nominal value for the shear loss in kinematic viscosity at 100 °C of CEC Reference Fluid RL 233 after 30 cycles, λ_2, is 2,80 $mm^2 \cdot s^{-1}$. This value should be used for derivation of the Correction Factor.

9.2 If the first reference run gives a viscosity loss at 100 °C outside the range of 2,70 $mm^2 \cdot s^{-1}$ to 2,90 $mm^2\ s^{-1}$, adjust the nozzle opening pressure as described in 8.1 and undertake a second reference test. In instances where the first reference run does not comply with the required control limits, the second reference test shall comply with the more stringent control limits of 2,75 $mm^2 \cdot s^{-1}$ to 2,85 $mm^2 \cdot s^{-1}$. The flow rate shall be checked following adjustment of the nozzle opening pressure to ensure that it remains in the range 170 $ml \cdot min^{-1} \pm 5\ ml \cdot min^{-1}$.

9.3 Calculate the calibration factor, *F*, using Formula (1):

$$F = \frac{\lambda_2}{\lambda_3} \quad (1)$$

where

λ_2 is the nominal value for the viscosity shear loss of CEC Reference Fluid RL 233 at 100 °C after 30 cycles (2,80 $mm^2 \cdot s^{-1}$);

λ_3 is the determined value for the viscosity shear loss of CEC Reference Fluid RL 233 at 100 °C after 30 cycles.

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 Determine the shear stability of the test fluid by undertaking duplicate test runs according to the procedure described in 10.3 to 10.6 inclusive.

10.3 Place 200 ml of the fluid to be tested in the fluid reservoir (7 in Figure A.1), using the graduation marks as a guide, and run the pump with the three-way stopcock (6 in Figure A.1) in a setting position such that approximately 50 ml is displaced from the apparatus. Upon attainment of the required fluid volume as indicated by the graduation mark on the fluid reservoir (7 in Figure A.1), stop the pump. Discard the displaced fluid portion to waste.

NOTE This procedure displaces the dead volume left in the test rig from the flushing procedure.

10.4 Set the pulse counter or electronic revolution counter to perform 250 cycles and then open both three-way stopcocks (6 and 8 in Figure A.1) to allow the fluid to continuously circulate until the required number of cycles has been completed.

NOTE A lower number of cycles may be adopted by agreement.

10.5 After 10 cycles, the operating temperature shall be within the range 30 °C to 35 °C. The flow of cooling water may be adjusted if required.

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 Formula (2):

$$SS = \frac{\lambda_0 - \lambda_1}{\lambda_0} \times 100 \times F \qquad (2)$$

where

λ_0 is the kinematic viscosity at 40 °C of the fluid before shear, in $mm^2 \cdot s^{-1}$;

λ_1 is the kinematic viscosity at 40 °C of the fluid after shear, expressed in $mm^2 \cdot s^{-1}$;

F is the calibration factor (9.3).

NOTE A low value indicates a high resistance to shear.

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[4)], for shear stability based upon 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 upon 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.

4) DIN 51382:1996[4] gives further details.