Designation: D 3945 – 93

AMERICAN SOCIETY FOR TESTING AND MATERIALS
1916 Race St. Philadelphia, Pa 19103
Reprinted from the Annual Book of ASTM Standards. Copyright ASTM
If not listed in the current combined index, will appear in the next edition.

An American National Standard

Standard Test Methods for Shear Stability of Polymer-Containing Fluids Using a Diesel Injector Nozzle[1]

This standard is issued under the fixed designation D 3945; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

NOTE—Procedures A and B of this method are in the process of being revised as separate ASTM Test Methods because tests of a series of polymer-containing fluids showed that the two procedures often give different results.

1. Scope

1.1 These test methods measure the percent viscosity loss at 100°C of polymer-containing fluids when evaluated by either of two diesel injector apparatus procedures. Procedure A uses European Diesel Injector Test Equipment and Procedure B uses Fuel Injector Shear Stability Test (FISST) equipment. The viscosity loss reflects polymer degradation due to shear at the nozzle.

NOTE 1—ASTM Test Method D 2603 has been used for similar evaluation of this property. It has many of the same limitations as indicated in the significance statement. No detailed attempt has been undertaken to correlate the results by the sonic and the diesel injector methods. Equipment and replacement parts are no longer available for Test Method D 2603.

NOTE 2—Procedure A of this method uses test apparatus as defined in CEC L-14-A-79, IP 294/76 and DIN 51382. The operational procedure is different on four specific points. Procedure A evaluates hydraulic-type fluids at 30 cycles, with viscosities measured at 100°C. Results are reported for single determinations. No equipment severity correction factor is used to adjust results.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)[2]

D 2603 Test Method for Sonic Shear Stability of Polymer-Containing Oils[3]

3. Summary of Test Method

3.1 The polymer-containing fluid is passed through a diesel injector nozzle at a shear rate that causes the less shear stable polymer molecules to degrade. The resultant degradation reduces the kinematic viscosity of the fluid under test. The reduction in kinematic viscosity, reported as percent loss of the initial kinematic viscosity, is a measure of the shear stability of the polymer-containing fluid.

4. Significance and Use

4.1 Both Procedure A (using European Diesel Injector Test Equipment) and Procedure B (using Fuel Injector Shear Stability Test – FISST – equipment) of this method evaluate the percent viscosity loss for polymer-containing fluids resulting from polymer degradation in the high shear nozzle device. Minimum interference from thermal or oxidative effects would be anticipated. The two procedures exhibit essentially equal percent viscosity loss for each oil used in developing the method. Both procedures also show essentially comparable repeatability and reproducibility.

4.2 These methods are not intended to predict viscosity loss in field service for different polymer classes or for different field equipment. However, it may be possible to establish some correlation for a specific polymer type in specific field equipment.

PROCEDURE A—EUROPEAN DIESEL INJECTOR TEST

5. Apparatus

5.1 The apparatus basically consists of a fluid reservoir, a double-plunger pump with an electric motor drive, an atomization chamber with a diesel injector spray nozzle and a fluid cooling vessel, installed in an area with an ambient temperature of 20 to 25°C (68 to 77°F). Figure A1.1, Annex A1, shows the schematic representation of equipment.[4]

5.1.1 *Fluid Reservoir,* (7) in Fig. A1.1 Annex A1, is open on the top, has a capacity of about 250 cm^3, has a 45-mm (1.772-in.) inner diameter and is calibrated in 25-cm^3 intervals. It is fitted with an internal fluid distributor detailed as Fig. A1.2. A 40-mm (1.575-in.) diameter watch glass with serrated edges is an acceptable distributor plate. The distributor reduces the tendency of fluid channeling. Temperature is measured by a thermometer suspended in the center of the glass container ((7) in Fig. A1.1). The bottom of the thermometer bulb should be 10 to 15 mm above the entrance to the drain tube opening. Other temperature-measuring equipment positioned at the same location may

[1] These test methods are under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and are the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved March 15, 1993. Published August 1993. Originally published as D 3945 – 80. Last previous edition D 3945 – 86[ε1].

[2] *Annual Book of ASTM Standards,* Vol 05.01.

[3] *Annual Book of ASTM Standards,* Vol 05.02.

[4] The complete assembly can be purchased from Hamburger Electro-Apparate Gmb Eidel-estedter Weg 225, Postfach 1244, D-2083 Halstenbek b. Hamburg, W. Germany or in North America, Petrolab, 874 Albany Rd., Latham, NY 12110.

also be used. The outlet is equipped with a three-way cock (*8*) in Fig. A1.1. The three-way cock is of a cone type with a nonexchangeable solid plug with an 8-mm (0.315-in.) nominal bore size. Rubber or plastic tubing, (*10*) in Fig. A1.1, is used to connect the three-way cock to the pump inlet.

5.1.2 *Double-Plunger Injection Pump, 11* in Fig. A1.1. is defined as Bosch PE 2 A 90C300/3S2266.[5] This pump is equipped with a stroke counter, (*15*), venting screw, (*14*), and flow rate adjusting screw, (*12*) in Fig. A1.1.

5.1.3 *Injection Pump* is driven by an electric motor, (*13*) in Fig. A1.1. The electric motor is a three-phase motor with 1.1-kW output rated at a speed of 925 ± 25 rpm.

NOTE 3—This motor runs at 925 rpm on the 50 Hz current prevalent in Europe; it will run at about 1100 rpm on 60 Hz current. The 1100 rpm speed is not acceptable in this procedure. Some suitable means must be taken to ensure the prescribed 925 ± 25 rpm speed to the injection pump. One acceptable method would be use of a 6-to-5 speed reducer.

5.1.4 *Outlet of Injection Pump* is connected to the atomization chamber using high pressure steel tubing. A pressure gage may be installed in this line. The atomization chamber, (*2*) in Fig. A1.1, is defined in more detail in Fig. A1.3. The spray chamber is designed so that the fluid under test exits from the nozzle into a chamber filled with the test fluid, in order to minimize foam generation. A drain tube with the cock is included to minimize contamination from the previous test during the system cleaning steps. The diesel injector nozzle is a Bosch DN 8 S2 type pintle nozzle injector installed in a Bosch KD 43 SA 53/13 nozzle holder. The nozzle holder includes a filter cartridge.[6]

NOTE 4—Take great care to avoid damage to the precision parts of the fuel injection equipment (the plunger and barrel in the pump and the nozzle valve assembly). Service work on the equipment should be performed by a diesel fuel injector pump specialist or with reference to the manufacturer's service manual.[7]

5.1.5 *Fluid Cooling Vessel,* (*5* in Fig. A1.1), is used to maintain the specified temperature of the test fluid, as indicated at the outlet of the fluid reservoir. This vessel is a glass container with exterior cooling jacket constructed so that the heat transfer surface of the jacket is of a spherical nature. The exterior jacket diameter, d_1, is approximately 50 mm (1.969 in.). The interior heat transfer surface, d_2, is approximately 25 mm (0.984 in.) in diameter. The overall length, L, is approximately 180 mm (7.087 in.). A distributor plate, similar in design to the distributor plate in the fluid reservoir, is positioned in the upper portion of the fluid cooling vessel to ensure contact between the fluid and the cooling surface. The discharge from the fluid cooling vessel is through a three-way cock of the same design as used on the discharge of the fluid reservoir. The exterior cooling jacket would be supplied with an adjustable volume of cold water.

6. Reference Fluids

6.1 *Diesel Fuel (No. 2)* is required for adjusting the diesel injector nozzle for the prescribed pressure setting.

6.2 *Calibration Fluid RL 34*[8] must be used to ensure that the equipment is functioning properly. Information on use of Procedure B Calibration Fluid TL-11074 (Section 15.2) by Procedure A is given in Appendix X1.

7. Precautions

7.1 During operation the line between the pump and nozzle, (*16*, in Fig. A1.1), is under a pressure of at least 17.5 ± 0.35 MPa (2550 ± 50 psi). Shut off the pump prior to tightening any fitting that is not properly sealed. Use a safety shield between the high-pressure components and the operator during use of equipment.

8. Sampling

8.1 The test fluid shall be at room temperature, uniform in appearance and free of any visible insoluble material, prior to placing in the test equipment.

8.2 After completing the prescribed number of test cycles drain the sheared oil from the atomization chamber by opening the cock below this chamber. Follow by positioning the three-way cock below the fluid reservoir to discharge the sheared oil into a suitable container for subsequent testing.

9. Calibration of Equipment

9.1 Adjust the diesel injector nozzle holder with nozzle in place with diesel fuel using a nozzle tester[9] so that the valve opening pressure is 17.5 ± 0.35 MPa (2550 ± 50 psi) under static conditions.

9.2 Calibration Fluid RL 34 is used to confirm that the equipment is operating properly and that the shear rate gives the correct kinematic viscosity loss. When carrying out the test in the prescribed manner for 30 cycles, Calibration Fluid RL 34 must experience a reduction in kinematic viscosity of 2.50 to 3.20 cSt (2.50 to 3.20 mm^2/s) at 100°C. If the proper loss in kinematic viscosity is not obtained two possible causes should be evaluated. Confirm that all operating requirements are being met. If these are proper, replace the injector nozzle with a new injector nozzle. Each new injector nozzle used in Procedure A should be run-in for 4 h prior to making the initial reference oil run. The subsequent 10.2 technique is to be used for the reference oil run. If the reference oil meets the viscosity loss specified, test work may be resumed. Otherwise, continue evaluating new nozzles until an acceptable one is in use.

9.3 Determine the residual undrained oil volume of the system. This is the volume of the system between the three-way cock below the fluid reservoir, (*8*) in Fig. A1.1, and the injector nozzle orifice, (*1*).

9.3.1 *Measurement of Residual Undrained Volume:*

[5] The pump can be purchased from Robert Bosch Corp., 2800 South 25th Ave., Broadview, IL 60153 or in North America, Petrolab, 874 Albany Rd., Latham, NY 12110.

[6] The nozzle and nozzle holder can be purchased from Robert Bosch Corp., 2800 South 25th Ave., Broadview, IL 60153 or in North America, Petrolab, 874 Albany Rd., Latham, NY 12110.

[7] Repair Instructions for Diesel Injection Pumps Size A, B, K and Z, Bulletin WJP 101/1 B EP, Robert Bosch GmbH, 2800 South 25th Ave., Broadview, IL 60153.

[8] Calibration Fluid RL 34 may be purchased from Fa. Krüger, Prüfmaschinenbau, Rilkeweg 13, 6831 Oberhausen 2, West Germany or in North America, Petrolab, 874 Albany Rd., Latham, NY 12110.

[9] Suitable sources of supply are: Bacharach Instrument Co., 625 Alpha Drive, Pittsburgh, PA 15238 (Code 65-0030 Bench-Type Nozzle Tester and Code 65-0275 Connector Set) and Robert Bosch Corp., 2800 South 25th Ave., Broadview, IL 60153 (EFEP 60H Nozzle Tester, Part Number 0 681 200 508).