

Designation: D7688 – 18

# Standard Test Method for Evaluating Lubricity of Diesel Fuels by the High-Frequency Reciprocating Rig (HFRR) by Visual Observation<sup>1</sup>

This standard is issued under the fixed designation D7688; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

# 1. Scope\*

1.1 This test method covers the evaluation of the lubricity of diesel fuels using a high-frequency reciprocating rig (HFRR).

1.2 This test method is applicable to middle distillate fuels, such as Grades No. 1-D S15, S500, and S5000, and Grades No. 2-D S15, S500, and S5000 diesel fuels, in accordance with Specification D975; and other similar petroleum-based fuels which can be used in diesel engines. This test method also is applicable to biodiesel blends. B5 was included in the round robin program that determined the precision statement.

Note 1—It is not known that this test method will predict the performance of all additive/fuel combinations. Additional work is underway to establish this correlation and future revisions of this test method may be necessary once this work is complete.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use. Specific warning statements are given in Section 7.

1.5 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

#### 2. Referenced Documents

2.1 ASTM Standards:<sup>2</sup>

D975 Specification for Diesel Fuel Oils

- D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
- D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
- D4306 Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination
- D6078 Test Method for Evaluating Lubricity of Diesel Fuels by the Scuffing Load Ball-on-Cylinder Lubricity Evaluator (SLBOCLE)
- E18 Test Methods for Rockwell Hardness of Metallic Materials
- E92 Test Methods for Vickers Hardness and Knoop Hardness of Metallic Materials
- 2.2 SAE Standard:<sup>3</sup>
- SAE-AMS 6440 Steel, Bars, Forgings, and Tubing, 1.45 Cr (0.93-1.05C) (SAE 52100), for Bearing Applications
- 2.3 ISO Standard:<sup>4</sup>

# 3. Terminology

# 3.1 Definitions:

3.1.1 *boundary lubrication,* n—a condition in which the friction and wear between two surfaces in relative motion are determined by the properties of the surfaces and the properties of the contacting fluid, other than bulk viscosity.

3.1.1.1 Discussion—Metal to metal contact occurs and the chemistry of the system is involved. Physically adsorbed or

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.E0 on Burner, Diesel, Non-Aviation Gas Turbine, and Marine Fuels.

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This test method was developed by ISO/TC22/SC7/WG6 and is a part of ISO 12156.

ISO 3290 Roller Bearings, Balls – Dimensions and tolerances

<sup>&</sup>lt;sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>&</sup>lt;sup>3</sup> Available from SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://www.sae.org.

<sup>&</sup>lt;sup>4</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

chemically reacted soft films (usually very thin) support contact loads. As a result, some wear is inevitable.

3.1.2 *lubricity, n*—a qualitative term describing the ability of a fluid to affect friction between, and wear to, surfaces in relative motion under load.

3.1.2.1 *Discussion*—In this test method, the lubricity of a fluid is evaluated by the wear scar, in microns, produced on an oscillating ball from contact with a stationary disk immersed in the fluid operating under defined and controlled conditions.

3.2 Abbreviations:

3.2.1 HFRR-high frequency reciprocating rig

3.2.2 WSD-wear scar diameter

#### 4. Summary of Test Method

4.1 A 2-mL test specimen of fuel is placed in the test reservoir of an HFRR.

4.2 A vibrator arm holding a nonrotating steel ball and loaded with a 200 g mass is lowered until it contacts a test disk completely submerged in the fuel. When the fuel temperature has stabilized, the ball is caused to rub against the disk with a 1 mm stroke at a frequency of 50 Hz for 75 min.

4.3 The test fuel temperature is maintained at 60 °C and the ambient relative humidity is maintained between 30 % and 85 %.

4.4 At the conclusion of the test, the upper specimen holder is removed from the vibrator arm and cleaned. The dimensions of the major and minor axes of the wear scar are measured under  $100 \times$  magnification and recorded.

# 5. Significance and Use

5.1 Diesel fuel injection equipment has some reliance on lubricating properties of the diesel fuel. Shortened life of engine components, such as diesel fuel injection pumps and injectors, has sometimes been ascribed to lack of lubricity in a diesel fuel.

5.2 The trend of HFRR test results to diesel injection system pump component distress due to wear has been demonstrated in pump rig tests for some fuel/hardware combinations where boundary lubrication is believed to be a factor in the operation of the component.<sup>5</sup>

5.3 The wear scar generated in the HFRR test is sensitive to contamination of the fluids and test materials, the temperature of the test fuel, and the ambient relative humidity. Lubricity evaluations are also sensitive to trace contaminants acquired during test fuel sampling and storage.

5.4 The HFRR and Scuffing Load Ball on Cylinder Lubricity Evaluator (SLBOCLE, Test Method D6078) are two methods for evaluating diesel fuel lubricity. No absolute correlation has been developed between the two test methods.

5.5 The HFRR may be used to evaluate the relative effectiveness of diesel fuels for preventing wear under the pre-



FIG. 1 Schematic Diagram of HFRR (not including instrumentation)

scribed test conditions. Correlation of HFRR test results with field performance of diesel fuel injection systems has not yet been determined.

5.6 This test method is designed to evaluate boundary lubrication properties. While viscosity effects on lubricity in this test method are not totally eliminated, they are minimized.

# 6. Apparatus

6.1 *High-Frequency Reciprocating Rig* (*HFRR*),<sup>6,7</sup>—(see Fig. 1) capable of rubbing a steel ball loaded with a 200 g mass against a stationary steel disk completely submerged in a test fuel. The apparatus uses a 1 mm stroke length at a frequency of 50 Hz for 75 min. Complete operating conditions are listed in Table 1.

6.2 *Test Reservoir*, capable of holding a test disk in a rigid manner beneath the test fuel. The temperature of this reservoir, and consequently the test fuel contained in it, is maintained by means of a closely attached electrically controlled heater pad.

6.3 *Control Unit*<sup>6,7</sup> for controlling stroke length, frequency, test reservoir temperature, friction force, electrical contact potential, and test duration, with an electronic data acquisition and control system.

6.4 *Microscope*, capable of  $100 \times$  magnification in graduations of 0.1 mm and incremented in divisions of 0.01 mm.

6.4.1 *Glass Slide Micrometer*<sup>7,8</sup> with a scale ruled in 0.01 mm divisions.

6.5 *Cleaning Bath*, ultrasonic seamless stainless steel tank with adequate capacity and a cleaning power of 40 W or greater.

6.6 *Desiccator*, capable of storing test disks, balls, and hardware.

<sup>&</sup>lt;sup>5</sup> Nikanjam, M., Crosby, T., Henderson, P., Gray, C., Meyer, K, and Davenport, N., "ISO Diesel Fuel Lubricity Round Robin Program," SAE, Paper No. 952372, SAE Fuels and Lubricants Meeting, Oct. 16-19, 1995, Toronto, Canada.

<sup>&</sup>lt;sup>6</sup> The sole source of supply of the apparatus known to the committee at this time is PCS Instruments, 78 Stanley Gardens, London W3 7SZ, England.

<sup>&</sup>lt;sup>7</sup> If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

<sup>&</sup>lt;sup>8</sup> The sole source of supply of the apparatus known to the committee at this time is Catalog No. 31-16-99 from Bausch & Lomb World Headquarters, One Bausch & Lomb Place, Rochester, NY 14604-2701. A certificate of traceability from the National Institute of Standards and Technology is available.

Fluid volume	2 mL ± 0.20 mL
Stroke length	1 mm ± 0.02 mm
Frequency	50 Hz ± 1 Hz
Fluid temperature	60 °C ± 2°C
Relative humidity	between 30 % and 85 %
Applied load	200 g ± 1 g
Test duration	75 min ± 0.1 min
Bath surface area	$6 \text{ cm}^2 \pm 1 \text{ cm}^2$

#### **TABLE 1 Test Conditions**

#### 7. Reagents and Materials

7.1 *Acetone*, reagent grade (**Warning**—Extremely flammable. Vapors may cause flash fire).

7.2 *Compressed Air*, containing less than 0.1 ppmv hydrocarbons and 50 ppmv water. (**Warning**—Compressed gas under high pressure. Use with extreme caution in the presence of combustible material.)

7.3 Gloves, appropriate for the reagents used.

7.4 Reference Fluids:

7.4.1 *Fluid*  $A^9$ —High lubricity reference (**Warning**— Flammable). Store in clean, borosilicate glass with an aluminum foil-lined insert cap or a fully epoxy-lined metal container. Store in dark area.

7.4.2 *Fluid*  $B^9$ —Low lubricity reference (**Warning**— Flammable. Vapor harmful). Store in clean, borosilicate glass with an aluminum foil-lined insert cap or a fully epoxy-lined metal container. Store in a dark area.

7.5 *Heptane*, reagent grade. (Warning—Extremely flammable. Vapors may cause flash fire.)

7.6 *Isooctane*, reagent grade. (Warning—Extremely flammable. Vapors may cause flash fire.)

7.7 2-propanol, reagent grade. (Warning—Extremely flammable. Vapors may cause flash fire.)

7.8 *Test Ball*, (Grade 28 per ISO 3290) of SAE-AMS 6440 steel, with a diameter of 6.00 mm, having a Rockwell hardness "C" scale (HRC) number of 58-66, in accordance with Test Methods E18.

7.8.1 Test balls are an extremely important part of the process and can significantly affect results. An equivalency evaluation was conducted on test balls and test disks and can be found in ASTM Research Report RR: RR:D02-1890.<sup>10</sup>

7.9 *Test Disk,* 10 mm disk of SAE-AMS 6440 steel machined from annealed rod, having a Vickers hardness "HV 30," in accordance with Specification E92, a scale number of 190-210, turned, lapped, and polished to a surface finish of less than  $0.02 \ \mu m R_a$ .

7.9.1 Test disks are an extremely important part of the process and can significantly affect results. An equivalency evaluation was conducted on test balls and test disks and can be found in ASTM Research Report RR: RR:D02-1890.<sup>10</sup>

NOTE 2—Users are encouraged to request a certificate of conformance

demonstrating that test balls and test disks meet the requirements listed in 7.8 and 7.9.

7.10 *Wiper,* wiping tissue, light-duty, lint-free, hydrocarbon-free, disposable.

#### 8. Sampling and Sample Containers

8.1 Unless otherwise specified, samples shall be taken by the procedure described in Practice D4057 or Practice D4177.

8.2 Because of the sensitivity of lubricity measurements to trace materials, sample containers shall be only fully epoxylined metal, amber borosilicate glass, or polytetrafluorethylene (PTFE), cleaned and rinsed thoroughly at least three times with the product to be sampled before use, as specified under Containers for Lubricity Testing in Practice D4306.

8.3 New sample containers are preferred, but if not available, the Containers for Lubricity Testing section of Practice D4306 gives guidance on suitable cleaning procedures for each type of container.

### 9. Preparation of Apparatus

9.1 Test Disks, (as received):

9.1.1 Place disks in a clean beaker. Transfer a sufficient volume of heptane or 50/50 isooctane/2-propanol into the beaker to completely cover the test disks.

9.1.2 Place beaker in ultrasonic cleaner and turn on for 7 min.

9.1.3 Handle all clean test pieces with clean forceps. Remove the test disks and repeat the above cleaning procedure from 9.1.1 with acetone for 2 min.

9.1.4 Dry and store in desiccator.

Note 3—Drying operations can be accomplished using compressed air jet at 140 kPa to 210 kPa pressure.

9.2 *Test Balls*, (as received)—The test balls are to be cleaned following the same procedure, 9.1.1 to 9.1.4, as for the test disks.

9.3 *Hardware*—All hardware and utensils that come into contact with the test disks, test balls, or test fuel, shall be cleaned by washing thoroughly with heptane or 50/50 isooctane/2-propanol, rinsed with acetone, and dried.

# 10. Test Apparatus Inspection and Verification

10.1 Recommended Calibration Intervals:

10.1.1 *Stroke Length*—Every three months.

10.1.2 Temperature Probes-Every twelve months.

10.2 *Test Apparatus*—Verify test apparatus performance and accuracy at least every 20 tests by testing each reference fluid in accordance with this section. Perform one test with each reference fluid. If the WSD for either fluid is outside the specified limits provided with each fluid by the ASTM Test Monitoring Center, verify that the test is performed correctly, and repeat both reference tests. If necessary, calibrate the HFRR by following the steps in the instrument manual, and then test each of the high and low reference fluids.

# 11. Procedure

11.1 Table 1 summarizes the test conditions.

<sup>&</sup>lt;sup>9</sup> Reference Fluids A and B are available from ASTM Test Monitoring Center, 6555 Penn Ave., Pittsburgh, PA 15026–4489.

<sup>&</sup>lt;sup>10</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1890. Contact ASTM Customer Service at service@astm.org.

11.2 Strict adherence to cleanliness requirements and to the specified cleaning procedures is required. During handling and installation procedures, protect cleaned test parts (disks, balls, reservoir, screws, heater block, and push rod) from contamination by using clean forceps and wearing appropriate gloves.

11.3 Using forceps, place the test disk into the test reservoir, shiny side up. Secure the test disk to the test reservoir and the test reservoir to the test apparatus. Ensure the unit's temperature probe is properly placed in the reservoir. Ensure the relative humidity in the test laboratory is between 30 % and 85 %. (**Warning**—relative humidity is an important parameter. Performing the test outside of the relative humidity limits will affect the lubricity result.)

11.4 Using forceps, place the test ball into the upper specimen holder and attach the holder to the end of the vibrator arm. Ensure the holder is horizontal before fully securing the unit.

11.5 Using a pipette, place 2 mL  $\pm$  0.2 mL of the test fuel into the test reservoir.

11.6 Set the test parameters according to Table 1.

11.7 Lower the vibrator arm and suspend a 200 g weight from the arm. Start the test.

11.8 At the completion of the test, lift up the vibrator arm. Remove the upper specimen holder.

11.9 Rinse the test ball (still in the holder) in cleaning solvents and wipe and dry thoroughly with a tissue.

11.10 Remove the test reservoir and properly dispose of the fuel.

11.11 Place the test ball holder under the microscope and measure the wear scar diameter in accordance with Section 12.

# 12. Measurement of the Wear Scar and ards/sist/a8632b0

12.1 Turn on the microscope light and position the test ball under microscope at  $100 \times$  magnification.

12.2 Focus the microscope and adjust the stage such that the wear scar is centered within the field of view.

12.3 Align the wear scar to a divisional point of reference on the numerical scale with the mechanical stage controls. Measure the major axis to the nearest 0.01 mm. Record the readings on the data sheet.

12.4 Align the wear scar to a divisional point of reference on the numerical scale with the mechanical stage controls. Measure the minor axis to the nearest 0.01 mm. Record the readings on the data sheet.

12.5 Record the condition of the wear area if different from the reference standard test, that is, debris color, unusual particles or wear pattern, visible galling, and so forth, and presence of particles in the test reservoir.

Note 4—Refer to Annex A1 for guidance to determine the boundaries of the wear scar.

# 13. Calculation

13.1 Calculate the wear scar diameter as follows:

$$VSD = \left[ (M+N)/2 \right] \cdot \left[ 1000 \right]$$

where:

WSD = wear scar diameter,  $\mu m$ ,

M =major axis, mm, and

N = minor axis, mm.

# 14. Report

14.1 Report the following information:

14.1.1 Major axis and minor axis to the nearest 0.01 mm, and wear scar diameter to the nearest  $10 \,\mu$ m.

14.1.2 Description of the test fuel and date sample taken.

14.1.3 Record the batch number of the test specimens.

14.1.4 Date of testing.

14.1.5 Report the test method number, D7688.

# 15. Precision and Bias<sup>11,12</sup>

15.1 *Precision*—The precision was developed using fuels representing a range of lubricity levels as well as a practical mix of common types of fuels, such as Grade No. 1-D, Grade No. 2-D, additized, and a biodiesel blend. The precision data were developed in a 2008 cooperative testing program involving ten testing laboratories from the United States, Canada, and South Africa. There were six distinct fluids and each laboratory received four samples of each fuel to conduct replicate testing both with the microscope and the digital camera. The fluids were blind coded so that replicate samples were not known to the operator. A randomized test sequence was provided and each laboratory was requested to use the same operator and equipment for all 24 samples.

15.1.1 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 the following value in only one case in twenty:

Repeatability =  $70 \ \mu m$ 

15.1.2 The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following value in only one case in twenty:

### Reproducibility = 90 $\mu$ m

15.2 *Bias*—The procedure in this test method has no bias because lubricity is not a fundamental and measurable fluid property and thus is evaluated in terms of this test method.

#### 16. Keywords

16.1 boundary lubrication; diesel fuel; friction; HFRR; lubricity; wear

<sup>&</sup>lt;sup>11</sup> Nikanjam, M., Rutherford, J., "Improving the Precision of the HFRR Lubricity Test," SAE Paper No. 2006-01-3363.

<sup>&</sup>lt;sup>12</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1718.