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Standard Test Method for Evaluating Lubricity of Diesel Fuels by the High-Frequency Reciprocating Rig (HFRR)¹

This standard is issued under the fixed designation D6079; 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.

ε¹Note—Editorial changes were made throughout in April 2005.

1. Scope*

- 1.1 This test method covers the evaluation of the lubricity of diesel fuels using a high-frequency reciprocating rig (HFRR). 1.2This test method is applicable to middle distillate fuels, such as Grades Low Sulfur No. 1 D, Low Sulfur No. 2 D, No. 1 D,
- 1.2This test method is applicable to middle distillate fuels, such as Grades Low Sulfur No. 1 D, Low Sulfur No. 2 D, No. 1 D and No. 2 D diesel fuels, in accordance with Specification
- 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; and other similar petroleum-based fuels which can be used in diesel engines. This test method 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 further 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 and health practices and determine the applicable regulatory limitations prior to use. Specific warning statements are given in Section 7.

2. Referenced Documents

2.1 ASTM Standards:²

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 Method for Vickers Hardness of Metallic Materials

2.2 American Iron and Steel Institute Standard: SAE Standard:³

AISI E-52100Chromium Alloy Steel

SAE-AMS 6440 Steel, Bars, Forgings, and Tubing, 1.45 Cr (0.93-1.05C) (SAE 52100), for Bearing Applications

2.3 American National Standards Institute Standard: ISO Standard: 4

ANSI B3.12Metal Balls

ISO 3290 Roller Bearings, Balls – Dimensions and tolerances

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products 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. DOI: 10.1520/D6079-04E01.

² 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.

³ Available from American Iron and Steel Institute (AISI), 1140 Connecticut Ave., Suite 705, Washington, DC 20036.

³ Available from SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://www.sae.org.

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



3. Terminology

- 3.1 Definitions of Terms Specific to This Standard:
- 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 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 <u>disedisk</u> 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

- 4.1A 2-mL test specimen of fuel is placed in the test reservoir of an HFRR and adjusted to either of the standard temperatures (25 or 60°C). The preferred test temperature is 60°C, except where there may be concerns about loss of fuel because of its volatility or degradation of the fuel because of the temperature.
- 4.2When the fuel temperature has stabilized, 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. The ball is caused to rub against the disk with a 1-mm stroke at a frequency of 50 Hz for 75 min.
- 4.3The ball is removed from the vibrator arm and cleaned. The dimensions of the major and minor axes of the wear scar are measured under 100× magnification and recorded.
 - 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 image of the wear scar is captured using the microscope digital camera, and the dimensions of the major and minor axes of the wear scar are measured and recorded.

5. Significance and Use

ASTM D6079-11

- 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.⁵
- 5.3 The wear scar generated in the HFRR test is sensitive to contamination of the fluids and test <u>materials</u> and <u>materials</u>, the temperature of the test <u>fuel</u>, and the <u>ambient relative humidity</u>. 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 prescribed 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). 6.7, (see (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.

⁵ 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.

⁶ The sole source of supply of the apparatus known to the committee at this time is PCS Instruments, 5 Warple Mews, 78 Stanley Gardens, London W3 ORF, 7SZ, England.

⁷ 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, which you may attend.



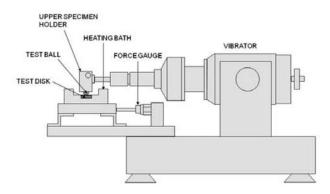


FIG. 1 Schematic Diagram of HFRR (not including instrumentation)

TABLE 1 Test Conditions

Fluid volume	2 \pm 0.20 mL
Stroke length	$1 \pm 0.02 \text{ mm}$
Frequency	$50 \pm 1 \text{ Hz}$
Fluid temperature	25 ± 2°C
	Of
	60 ± 2°C
Relative humidity	> 30 %
Relative humidity	between 30 % and 85 %
Applied load	200 ± 1 g
Test duration	$75 \pm 0.1 \mathrm{min}$
Bath surface area	$6 \pm 1 \text{ cm}^2$
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- 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^{6,7} 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× magnification in graduations of 0.1 mm and incremented in divisions of 0.01 mm.
- 6.4.1 Glass Slide Micrometer, with a scale ruled in 0.01 mm divisions. Microscope, with Digital Camera 6.7 capable of 100× magnification, installed and calibrated according to manufacturer instructions, capable of capturing a crisp image of the wear scar. Camera system resolution should be a minimum of 2048 × 1536 pixels. The measurement system should allow horizontal and vertical measurement devices or cursors to be positioned at the wear scar boundaries with an accuracy of 1 micron.
 - 6.5 Cleaning Bath, ultrasonic seamless stainless steel tank with adequate capacity and a cleaning power of 40 W or greater.
 - 6.6 Desiccator, containing a drying agent, capable of storing test disks, balls, and hardware.

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, elean, lint-free, cotton, disposable, appropriate for the reagents used.
 - 7.4 Reference Fluids:
- 7.4.1 *Fluid A*⁸—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^8 —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 *Test Ball*, (Grade 24 per ANSI B3.12) of AISI E-52100 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, 6.7 (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, and a surface finish of less than 0.05 µm R_a.9

⁸ 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.

⁸ Reference Fluids A and B are available from ASTM Test Monitoring Center, 6555 Penn Ave., Pittsburgh, PA 15026–4489.



7.6Test Disk, 10 mm disk of AISI E-52100.

7.9 Test Disk, $^{6.7}$ —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 μ m R_a .

7.7Toluene, reagent grade (Warning—Flammable. Harmful if inhaled).

7.8.

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 epoxy-lined 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):
- 9.1.1Upon receipt, new discs must be stored under toluene for at least 12 h before cleaning, as described in 9.1.2 through 9.1.5.
- 9.1.2Remove disks from toluene and place dises in a clean beaker. Transfer a sufficient volume of toluene into the beaker to completely cover the test disks.
 - 9.1.3Place beaker in ultrasonic cleaner and turn on for 7 min.
- 9.1.4Handle all clean test pieces with clean forceps. Remove the test discs and repeat the above cleaning procedure from 9.1.2
- 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 discs and repeat the above cleaning procedure from 9.1.1 with acetone for 2 min.

9.1.5

9.1.4 Dry and store in desiccator.

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Note 2—Drying operations can be accomplished using compressed air jet at 140 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 9.1.59.1.4, 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 toluene, dried, and heptane or 50/50 isooctane/2-propanol, rinsed with acetone. Dry acetone, and store in a desiceator. dried.

10. Test Apparatus Inspection and Verification

- 10.1 *Temperature*—Check the temperature control of the test reservoir using a calibrated temperature measuring device. Recommended Calibration Intervals:
 - 10.1.1 Stroke length—every three months.
 - 10.1.2 Temperature probes—every twelve months.
 - 10.2 Frequency—Check the frequency of the vibrator unit with a calibrated frequency meter.
- 10.3Stroke Length—Check the stroke length by measuring the wear sear on the test disk, using a calibrated microscope, after running a test on reference Fluid B. Subtract the width of the wear sear from the length of the wear sear to give the actual stroke length.
 - 10.4Run Time—Check the run time with a calibrated stopwatch (optional).
- 10.5Test Apparatus—Verify test apparatus performance and accuracy at least every 20 tests by testing each reference fluid in accordance with this section. Perform two tests with each reference fluid at the test temperature to which the test apparatus is being verified. If the difference between the two wear sear diameters (WSDs) for either fluid is greater than 80 μm, then further tests or corrective action to verify the test apparatus performance and accuracy will be required. Further tests or corrective action to verify the test apparatus performance and accuracy is also required if the average of the two tests differs by more than 80 μm from the average WSD values provided with Fluid A and Fluid B at 25°C and 60°C. —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.
- 11.2Strict adherence to cleanliness requirements and to the specified cleaning procedures is required. During handling and installation procedures, protect cleaned test parts (disks, balls, reservoir, and fixtures) from contamination by using clean forces and wearing clean cotton gloves.
- 11.3Using 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 thermocouple is properly placed in the reservoir. Ensure the relative humidity in the test laboratory is greater than 30%.
- 11.4Using forceps, place the test ball into the holder and attach the holder to the end of the vibrator arm. Ensure the holder is horizontal before fully securing the unit.
 - 11.5Using a pipette, place 2 ± 0.2 mL of the test fuel into the bath.
- 11.6Set the temperature controller to the desired test temperature (25°C or 60°C, preferably 60°C, see 4.1) and switch on the heater. Set the stroke length to 1 mm. Set the vibration frequency to 50 Hz.
- 11.7When the temperature has stabilized, lower the vibrator arm and suspend a 200-g weight from the arm. Start the vibrator unit.
- 11.8Operate the test for 75 min. At the completion of the test, switch off the vibrator unit and the heater. Lift up the vibrator arm and remove the test ball holder.
- 11.9Rinse the test ball (still in the holder) in cleaning solvents and dry with a tissue. Using a permanent marker, circle the wear sear on the ball.
- 11.10Remove the test reservoir and properly dispose of the fuel. Remove the test disk and wipe it clean. Place the disk in a storage receptacle (plastic bag) marked with the unique test reference.
- 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 ± 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
- 11.12Upon completion of the wear sear measurement, remove the test ball from the holder and place the ball together in storage with the test disk...

12. Measure of the Wear Scar

- 12.1Turn on the microscope light and position the test ball under microscope at 100× magnification.
- 12.2Focus the microscope and adjust the stage such that the wear sear is centered within the field of view.
- 12.3Align the wear sear 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.
- 12.4Align the wear sear 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.
- 12.5Record 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. Measurement of the Wear Scar
 - 12.1 Turn on the microscope light and position the upper specimen holder in the staging area slot at 100× magnification.
 - 12.2 Adjust the stage so that the wear scar in centered in the viewing field.
 - 12.3 Adjust the light intensity to obtain a clearly illuminated image.
 - 12.4 Adjust the microscope stage until the edges of the wear scar come into focus.
 - 12.5 Capture the image using the camera.
 - 12.6 Identify and measure the x axis.
 - 12.7 Identify and measure the y axis.
 - 12.8 Record the measurement results.
 - Note 3—Refer to Annex A1, Measurement of HFRR Wear Scars, for guidance to determine the boundaries of the wear scar.



13. Calculation

13.1 Calculate the wear scar diameter as follows:

 $WSD = (M + N)/2 \cdot 1000$

WSD = (M + N)/2

where:

WSD = wear scar diameter, μ m,

 $M = \text{major axis, } \frac{\text{mm,} \mu \text{m}}{\text{m}}$, and

 $N = \frac{\text{minor axis, mm.minor axis, } \mu\text{m.}}{\text{minor axis, } \mu\text{m.}}$

14. Report

14.1 Report the following information:

14.1.1Major axis and minor axis to the nearest 0.01 mm, and wear scar diameter to the nearest 10 µm.

14.1.2Description of the wear sear area.

14.1.3Fuel temperature.

14.1.4Description of the test fuel and date sample taken.

14.1.5Identification of test specimens.

14.1.6Date of testing.

14.1.7Any deviations from the test conditions given in Table 1.

14.1.1 Major axis and minor axis to the nearest 10 μm, and wear scar diameter to the nearest 10 μm.

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, D6079. Chandards

15. Precision and Bias 9,10

15.1 Precision—The precision was developed for fuels with wear sear diameters between 143 and 772 µm at 25°C (175 and 1000 µm at 60°C). The precision data were developed in a 1995 cooperative testing program involving both United States and European testing laboratories. There were nine distinct fluids and each laboratory was given 18 fluids to test. 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 18 samples. Nine laboratories tested the HFRR at 25°C and ten laboratories tested the HFRR at 60°C. —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 at 25°C=62 μm Repeatability at 60°C=80 μm Repeatability = 50 μm

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 at 25°C=127 μm Reproducibility at 60°C=136 μm Reproducibility = 80 μm

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

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

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

⁹ Reference Fluids A and B are available from ASTM Test Monitoring Center, 6555 Penn Ave., Pittsburgh, PA 15026-4489.

⁹ Nikanjam, M., Rutherford, J., "Improving the Precision of the HFRR Lubricity Test," SAE Paper No. 2006-01-3363.

¹⁰ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1411. RR:D02-1718.