INTERNATIONAL STANDARD

ISO 12156-1

Third edition
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Diesel fuel — Assessment of lubricity using the high-frequency reciprocating rig (HFRR) —

Part 1: **Test method**

Carburant diesel — Évaluation du pouvoir lubrifiant au banc alternatif à haute fréquence (HFRR) —

Partie 1: Méthode d'essai

Reference number
ISO 12156-1:2016(E)

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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. www.iso.org/directives

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. 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. 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 22, *Road vehicles*, Subcommittee SC 34, *Propulsion, powertrain and powertrain fluids*, in collaboration with Technical Committee ISO/TC 28, *Petroleum products and related products of synthetic or biological origin.*

This third edition cancels and replaces the second edition (ISO 12156-1:2006), which has been technically revised. Details of the major changes (additions, modifications and deletions) which affect the performance of the products or the technical requirements applicable to the products are provided for information in Annex B.

ISO 12156 consists of the following parts, under the general title *Diesel fuel — Assessment of lubricity using the high-frequency reciprocating rig (HFRR)*:

— *Part 1: Test method*

— *Part 2: Limit*

For the purposes of user feedback and making future improvements to this part of ISO 12156, we encourage you to share your views. Please click on the link below to take part in the online survey. https://www.surveymonkey.com/r/12156-1

Introduction

All diesel fuel injection equipment has some reliance on diesel fuel as a lubricant. Wear due to excessive friction resulting in shortened life of engine components, such as diesel fuel injection pumps and injectors, has sometimes been ascribed to lack of lubricity in the fuel.

The relationship of test results to diesel injection equipment component distress due to wear has been demonstrated for some fuel/hardware combinations where boundary lubrication is a factor in the operation of the component.1)

Test results from fuels tested to this procedure have been found to correlate with many fuel/hardware combinations and provide an adequate prediction of the lubricating quality of the fuel. The correlation of biodiesel blends has been validated through 15 years of field experience and anecdotal data.

This part of ISO 12156 includes content and data, with permission of ASTM International, from ASTM Research Report RR:D02-1718[3] that is cited in ASTM D6079[1] and ASTM D7688.[2]

1) NIKANJAM, Manuch, Teri CROSBY, Paul HENDERSON, Chris GRAY, Klaus MEYER, and Nick DAVENPORT, "ISO Diesel Fuel Round Robin Program," SAE Technical Paper No. 952372, 1995, ISSN 0148- 7191, doi: 10.4271/952372.

Diesel fuel — Assessment of lubricity using the high-frequency reciprocating rig (HFRR) —

Part 1:
Test method

WARNING — Application of this part of ISO 12156 may involve the use of hazardous materials, operations and equipment. This part of ISO 12156 does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this part of ISO 12156 to establish appropriate safety and health practices, and determine the applicable regulatory limitations prior to use.

1 Scope

This part of ISO 12156 specifies a test method using the high-frequency reciprocating rig (HFRR), for assessing the lubricating property of diesel fuels, including those fuels which may contain a lubricity-enhancing additive. It defines two methods for measurement of the wear scar; Method “A” — Digital camera, and Method “B” — Visual observation.

This test method applies to fuels used in diesel engines.

NOTE It is not known if this test method will predict the performance of all additive/fuel combinations, including paraffinic fuels for which no additional correlation testing has been performed. Nevertheless, no data has been presented to suggest that such fuels are not within scope.

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 683-17, *Heat-treated steels, alloy steels and free-cutting steels — Part 17: Ball and roller bearing steels*

ISO 3170, *Petroleum liquids — Manual sampling*

ISO 3171, *Petroleum liquids — Automatic pipeline sampling*

ISO 3290-1, *Rolling bearings — Balls — Part 1: Steel balls*

ISO 4288, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Rules and procedures for the assessment of surface texture*

ISO 5272, *Toluene for industrial use — Specifications*

ISO 6507-1, *Metallic materials — Vickers hardness test — Part 1: Test method*

ISO 6508-1, *Metallic materials — Rockwell hardness test — Part 1: Test method*

ASTM D4306, *Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination*

3 Terms and definitions

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

3.1
boundary lubrication
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

Note 1 to entry: 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.

3.2
lubricity
qualitative term describing the ability of a fluid to affect friction between, and wear to, surfaces in relative motion under load

Note 1 to entry: In this test method, the lubricity of a fluid is evaluated by the wear scar, measured in micrometres, produced on an oscillating ball from contact with a stationary disk immersed in the fluid operating under closely controlled conditions.

3.3
wear scar diameter
WSD
mean diameter of the wear scar produced on the test ball

4 Principle

A sample of the fluid under test is placed in a test reservoir which is maintained at the specified test temperature. A fixed steel ball is held in a vertically mounted chuck and forced against a horizontally mounted stationary steel plate with an applied load. The test ball is oscillated at a fixed frequency and stroke length while the interface with the plate is fully immersed in the fluid. The metallurgies of the ball and plate, test fluid temperature, load, frequency, stroke length, and the ambient air conditions of temperature and humidity during the test are specified. The wear scar generated on the test ball is taken as a measure of the fluid lubricity.

5 Reagents and materials

5.1 Compressed air, if required for drying the equipment (8.1.1 and 8.1.2), supplied at a pressure of 140 kPa to 210 kPa and containing less than 0,1 ml/m^3 hydrocarbons and less than 50 ml/m^3 water.

WARNING — Use with extreme caution in the presence of combustible material.

5.2 Acetone, analytical reagent grade.

WARNING — Extremely flammable. Vapours may cause flash fire.

5.3 Reference fluids.[2)]

WARNING — Flammable.

Two reference fluids, Fluid "A" – High (Good) lubricity reference and Fluid "B" – Low (Poor) lubricity reference, produced in accordance with ISO Guide 34 and ISO Guide 35, shall be used for verifying the performance of the test apparatus. They shall be clearly marked with the HFRR value (WSD) and its expanded uncertainty, expressed in micrometres. Store reference fluids in clean, borosilicate glass with an aluminium foil-lined insert cap or fully epoxy-lined metal container. Store in a dark location.

2) Reference fluids A and B are available from ASTM Monitoring Center, 6555 Penn Avenue, Pittsburgh, PA 15026-4489 USA. This information is given for the convenience of users of this part of ISO 12156 and does not constitute an endorsement by ISO of the products named. Equivalent products may be used if they can be shown to lead to the same results.

5.4 Gloves, appropriate for the reagents used.

5.5 Heptane, reagent grade.

WARNING — Extremely flammable. Vapours may cause flash fire.

5.6 Isooctane, reagent grade.

WARNING — Extremely flammable. Vapours may cause flash fire.

5.7 2-propanol, reagent grade.

WARNING — Extremely flammable. Vapours may cause flash fire.

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

5.9 Toluene, in accordance with ISO 5272.

WARNING — Extremely flammable. Vapours may cause flash fire. May be fatal if swallowed and enters airways. May cause drowsiness or dizziness. Suspected of damaging the unborn child. May cause damage to organs through prolonged or repeated exposure.

6 Apparatus

6.1 Test apparatus,[3)] (see Figure 1), capable of engaging a steel ball loaded against a stationary steel plate with an applied load and oscillating at a fixed frequency and stroke length while the contact interface is fully immersed in a fluid according to the test conditions given in Table 1.

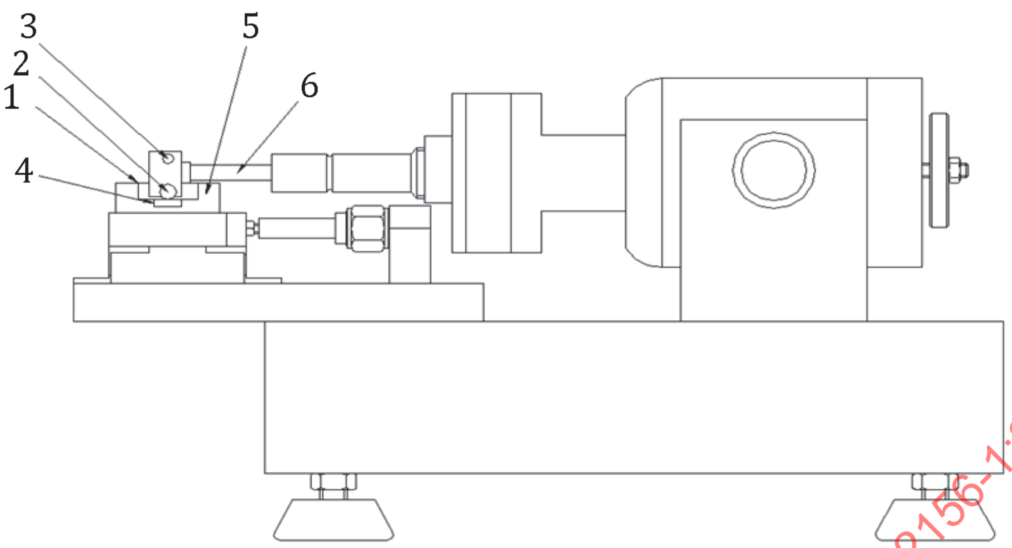
Table 1 — Test conditions

Parameter	Value
Fluid volume, ml	2 ± 0,2
Stroke length, mm	1 ± 0,02
Frequency, Hz	50 ± 1
Laboratory air[a]	See Figure 2
Fluid temperature, °C	60 ± 2
Test mass[b], g	200 ± 1
Test duration, min	75 ± 0,1
Reservoir surface area, mm^2	600 ± 100

[a] Laboratory air conditions as measured between 0,1 m and 0,25 m of the fluid reservoir shall be controlled to the acceptable range of conditions as shown in Figure 2.

[b] Total mass including fixing elements.

3) HFRR units, HFR2, supplied by PCS Instruments, 78 Stanley Gardens, London W3 7SZ, U.K., have been found satisfactory. This information is given for the convenience of users of this part of ISO 12156 and does not constitute an endorsement by ISO of these products. Equivalent products may be used if they can be shown to lead to the same results.



Key

1 fluid reservoir
2 test ball
3 test mass
4 test plate
5 heating bath
6 oscillating motion

Figure 1 — Example of the high-frequency reciprocating rig

The fluid reservoir shall be capable of holding a test plate in a rigid manner and shall also contain the test fluid. The temperature of this reservoir, and consequently the test fluid contained in it, should be achieved by means of an electrically controlled heater pad to which the fluid reservoir is closely attached.

The apparatus control unit for controlling variable parameters shall include provision for electronic data storage and retrieval, and for electronic calibration of the stroke length.

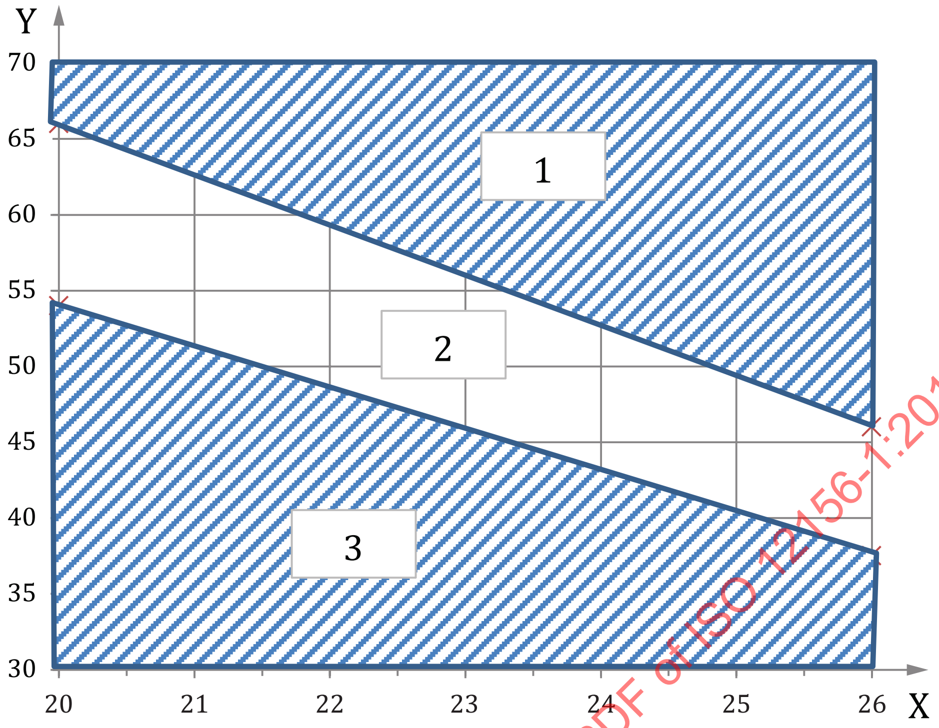
6.2 Test plate,[4] steel ISO 683-17-100Cr6 machined from annealed rod, having a Vickers hardness “HV 30” scale number of 190 to 210 (according to ISO 6507-1). It shall be lapped and polished to a surface finish of Ra < 0,02 µm, measured according to ISO 4288.

6.3 Test ball,[4] 6,00 mm diameter, grade 28 (G28) according to ISO 3290-1 of steel ISO 683-17-100Cr6. It shall have a Rockwell hardness “C” scale (HRC) number of 58 to 66 (according to ISO 6508-1).

6.4 Microscope with digital camera[4] **(Method “A”)**, capable of 100X 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 X 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 µm.

6.5 Microscope[4] **(Method “B”)**, metallurgical type, suitable for measuring the wear scar on the test ball to the nearest 1 µm.

4) Suitable test specimens and microscopes (with or without camera) are available from PCS Instruments, 78 Stanley Gardens, London W3 7SZ, U.K. This information is given for the convenience of users of this part of ISO 12156 and does not constitute an endorsement by ISO of this product. Equivalent products may be used if they can be shown to lead to the same results.



Key

1 unacceptable range of conditions — too moist
2 acceptable range of conditions
3 unacceptable range of conditions — too dry

X air temperature, °C
Y relative humidity, %

Figure 2 — Laboratory air conditions

6.6 Desiccator, containing a drying agent, capable of storing test plates, balls, and hardware.

6.7 Cleaning bath, ultrasonic type, with a seamless stainless steel tank of adequate capacity and a cleaning power of 40 W or greater.

6.8 Time-measuring device, mechanical or electronic, capable of measuring (75 ± 0,1) min.

7 Sampling

7.1 Unless otherwise specified, samples shall be taken by the procedures described in ISO 3170 or ISO 3171.

7.2 Because of the sensitivity of lubricity measurements to trace materials, sample containers shall be only fully epoxy-lined metal or amber borosilicate glass with an aluminium foil-lined insert cap, cleaned and rinsed thoroughly at least three times with the product to be sampled before use, as specified under “Containers for Lubricity Testing” in ASTM D4306.

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

8 Preparation and calibration

8.1 Preparation of apparatus

NOTE Toluene is the preferred solvent; however, users are warned that its use has been limited in some parts of the world for health and safety reasons. Optional solvents, heptane and a 50/50 blend of isooctane and 2-propanol have been proven acceptable in some cases and are included to satisfy the ISO Global Relevance Policy. However, the cleaning efficacy of the optional solvents has yet to be fully tested with fuels containing high amounts of surfactant components.

8.1.1 Test plates and balls

Using clean forceps, place a number of plates (6.2) (shiny side up) and balls (6.3) as received into a clean glass container, and cover with toluene (5.9), or heptane (5.5), or a 50/50 blend of isooctane (5.6) and 2-propanol (5.7). Place the container in the ultrasonic cleaning bath (6.7) and turn on for 7 min. Transfer the plates (shiny side up) and balls into a container of fresh acetone (5.2) and place the container in the ultrasonic cleaning bath (6.7) and turn on for 2 min. Dry plates and balls and place in desiccator (6.6).

NOTE Drying operation can be accomplished using compressed air jet at 140 kPa to 210 kPa pressure.

8.1.2 Hardware

Place the sample holders, screws, and all hardware and utensils that come into contact with the test fluid, in a clean glass beaker and cover with toluene (5.9), or heptane (5.5), or 50/50 isooctane (5.6)/2-propanol (5.7). Place the beaker in the ultrasonic cleaning bath (6.7) for 7 min, then using clean forceps transfer the hardware and test specimens into a beaker of acetone (5.2). Place in the ultrasonic cleaning bath for 2 min. Remove the components, blow dry (5.1), and if not to be used immediately, store in the desiccator (6.6).

8.2 Calibration and correction

8.2.1 Temperature

The temperature control of the fluid reservoir (see Figure 1) shall be checked using a calibrated temperature-measuring device every 12 months.

8.2.2 Frequency

The frequency of the vibrator unit shall be checked with a calibrated frequency meter every three months.

8.2.3 Stroke length

The stroke length shall be checked every three months by electronic self-calibration.

8.2.4 Test duration

The test duration shall be checked with a calibrated timer (6.8) every 12 months.

8.2.5 Test rig performance

The instrument performance shall be checked by running a single test on each of the two reference fluids (5.3). If the WSD is outside the certified range for that reference fluid, two more tests shall be carried out. If either of these tests gives a result which is out of range, the instrumentation and stroke length verification (8.2.1 to 8.2.4) shall be performed. If the result for the poor-lubricity fluid is too low, it may need to be replaced. If necessary, calibrate the test rig by following the steps in the instrument manual, and then test each good and poor reference fluids.

Referencing tests shall be conducted using each reference fluid after every 20 tests or every 90 days, whichever is shorter.

9 Test procedure

9.1 The greatest care shall be taken to adhere strictly to cleanliness requirements and to the specified cleaning procedures. During handling and installation procedures, protect cleaned test parts (plates, balls, fluid reservoir and fixtures) from contamination by using clean forceps and ensure that the specimens (6.2 and 6.3) do not become scratched.

9.2 Using forceps, place the test plate into the fluid reservoir, shiny side up. Secure the test plate to the reservoir and the reservoir to the test rig. Ensure that the temperature-measuring device is properly placed in the fluid reservoir.

9.3 Using 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.

9.4 Using calibrated instruments, measure the ambient temperature and relative humidity within 0,1 m to 0,25 m of the fluid reservoir. To properly manage ambient conditions, the test rig should be operated in a humidity cabinet[5]. If the values of both temperature and humidity do not conform to the requirements of Figure 2, steps shall be taken to correct the ambient conditions before the test may proceed. Record the temperature and relative humidity.

NOTE Relative humidity is an important parameter and the precision has been determined in accordance with the requirements shown in Figure 2. Performing the test outside of the relative humidity limits can affect the lubricity result.

9.5 Using a disposable pipette, place 2 ml ± 0,2 ml of the test fluid in the fluid reservoir.

9.6 Lower the vibrator arm and suspend a 200 g mass from the arm.

9.7 Set the temperature controller, stroke length, and vibration frequency to the values given in Table 1, and initiate the test.

9.8 Operate the test for 75 min. At the completion of the test, lift up the vibrator arm and remove the test ball holder.

9.9 Without removing the ball from the test ball holder; rinse the holder in cleaning solvents and then wipe and dry thoroughly with a tissue.

9.10 Remove the fluid reservoir and properly discard the fluid.

9.11 With the test ball still in the holder; position it under the microscope and measure the wear scar diameter in accordance with Clause 10.

9.12 Upon completion of the wear scar measurement, remove the test ball from the holder and place the ball in storage together with the test plate. It is good practice to retain the specimens for at least 12 months.

5) Humidity Cabinets supplied by PCS Instruments, 78 Stanley Gardens, London W3 7SZ, U.K., have been found satisfactory. This information is given for the convenience of users of this part of ISO 12156 and does not constitute an endorsement by ISO of these products. Equivalent products may be used if they can be shown to lead to the same results.

10 Measurement of wear scar

10.1 General

Contained herein are two methods for measurement of the wear scar; Method "A" — Digital camera, and Method "B" — Visual observation. Method "A" is the preferred method due to its improved precision and ease of use. In case of a dispute, Method "A" is considered the referee method.

10.2 Method "A" — Digital camera

10.2.1 Turn on the microscope light and position the test ball in the staging area slot at 100X magnification.

10.2.2 Adjust the stage so that the wear scar is centred in the viewing field.

10.2.3 Adjust the light intensity to obtain a clearly illuminated image.

10.2.4 Adjust the microscope stage until the edges of the wear scar come into focus; refer to Annex A for guidance if necessary.

10.2.5 Capture the image using the camera.

10.2.6 Measure the scar diameter in the x and y directions to the nearest 10 µm. Record the readings on the data sheet.

10.3 Method "B" — Visual observation

10.3.1 Position the test ball under the microscope at 100X magnification.

10.3.2 Move the test ball to centre the wear scar in the field of view. Adjust the illumination until the edges of the wear scar can clearly be seen; refer to Annex A for guidance if necessary.

10.3.3 Measure the scar diameter in the x and y directions to the nearest 10 µm. Record the readings on the data sheet.

11 Test results

Calculate the wear scar diameter, *WSD*, in micrometres, using Formula (1).

$$WSD = (x + y)/2 \quad (1)$$

where

x is the scar dimension perpendicular to oscillation direction, expressed in micrometres (µm);

y is the scar dimension parallel to oscillation direction, expressed in micrometres (µm).

12 Precision

12.1 General

The precision[6),7)] was developed for fuels with an average wear scar diameter between 200 µm and 700 µm. The samples consisted of six middle distillate fuels, including three neat (without additive) Grades No. 1 D S500, No. 2 D S15 and No. 2 D S500, one biodiesel blend, No. 2 D S15 B5, and two fuels No. 2 D S15 containing two different levels of lubricity enhancing additive, all in accordance with ASTM D975.[4] The precision data were developed in a 2008 cooperative testing program involving 10 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 microscope and with 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. Precision data from each of the two measurement methods are listed separately.

12.2 Repeatability, *r*

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 value given below in only one case in 20.

Method "A" Digital Camera	Method "B" Visual Observation
r = 50 µm	r = 70 µm

12.3 Reproducibility, *R*

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 value given below in only one case in 20.

Method "A" Digital Camera	Method "B" Visual Observation
R = 80 µm	R = 90 µm

13 Test report

The test report shall include the following information:

a) a reference to this part of ISO 12156, i.e. ISO 12156-1;

b) the method of WSD measurement (i.e. Method "A" or Method "B");

c) an unambiguous description of the fluid tested;

d) the x and y wear scar dimensions to the nearest 10 µm;

e) the air temperature and relative humidity at the start and end of the test;

f) the wear scar diameter (WSD) to the nearest 10 µm;

6) NIKANJAM, Manuch and Jim RUTHERFORD, "ISO Diesel Fuel Round Robin Program," SAE Technical Paper No. 2006-01-3363, 2006, ISSN 0148-7191, doi: 10.4271/2006-01-3363.

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

g) an identification of the specimens tested;

h) the date and value obtained for the most recent test on each reference fluid;

i) the date of test.

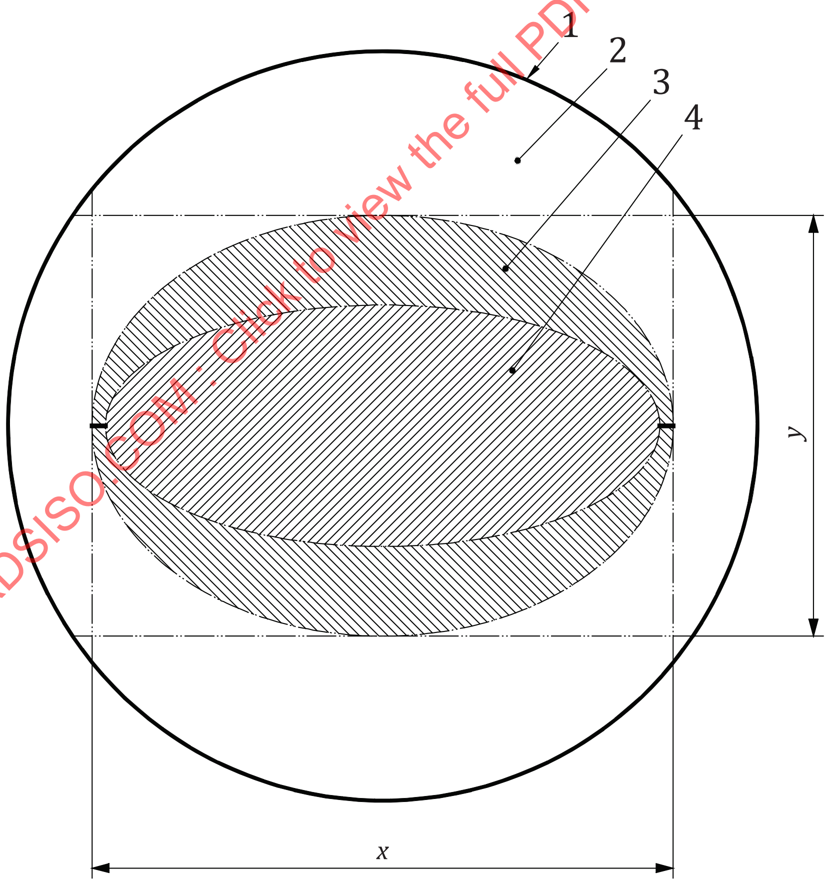
Annex A
(informative)

Measurement of HFRR wear scars

The appearance of the wear scar on the ball can vary with fuel type, particularly when lubricity additives are present. In general, the wear scar appears to be a series of scratches in the direction of motion of the ball, somewhat larger in the x direction than in the y direction.

In some cases, for example when low (poor) lubricity reference fluids are tested, the boundary between the scar and the discoloured (but unworn) area of the ball is distinct, and it is easy to measure the scar size. In other cases, the central scratched part of the scar is surrounded by a less distinct worn area, and there is no sharp boundary between the worn and unworn areas of the ball. In these cases, it can be more difficult to see or measure the true scar shape; as shown in Figure A.1, the overall wear scar comprises the distinct and the less distinct areas.

Photographic examples of various wear scar shapes are shown in Figure A.2, together with an assessment of the overall scar boundary.



Key

1 test ball (not to scale)

2 unworn area

3 less distinct worn area

4 worn area

Figure A.1 — Example of a wear scar with an indistinct boundary