BRITISH STANDARD

BS
2000-450:2000

Methods of test for petroleum and its products — BS 2000-450: Diesel fuel — Assessment of lubricity using the high-frequency reciprocating rig (HFRR) —

Part 1: Test method

(Identical with IP 450-2000)

ICS 75.080

Confirmed
January 2010

National foreword

This British Standard reproduces verbatim IP 450:2000 and implements it as the UK national standard.

The UK participation in its preparation was entrusted to Technical Committee PTI/13, Petroleum testing and terminology, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

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A list of organizations represented on this committee can be obtained on request to its secretary.

The Institute of Petroleum publishes and sells all Parts of BS 2000, and all BS EN petroleum test methods that would be Part of BS 2000, both in its annual publication "Standard methods for analysis and testing of petroleum and related products and British Standard 2000 Parts" and individually.

Further information is available from:

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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.

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.

Test results from fuels tested to this procedure have been found to correlate to many fuel/hardware combinations and provide an adequate prediction of the lubricating quality of the fuel.

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 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. Additional work is underway to further establish this correlation and future revisions of this part of ISO 12156 may be necessary once this work is complete.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 12156. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 4259:1992, *Petroleum products — Determination and application of precision data in relation to methods of test*.

ISO 5272:1979, *Toluene for industrial use — Specifications*.

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

1) To be published. (Revision of ISO 6507-1:1982, ISO 6507-2:1983, ISO 6507-3:1989, ISO 409-1:1982, ISO 409-2:1983 and ISO/DIS 409-3)

ISO 6508:1986, *Metallic materials — Hardness test — Rockwell test (scales A - B - C - D - E - F - G - H - K).*

ISO/IEC Guide 25:1990, *General requirements for the competence of calibration and testing laboratories.*

ISO Guide 35:1989, *Certification of reference materials — General and statistical principles.*

ASTM D-329:1995, *Specification for acetone.*

AISI E-52100, *Chromium alloy steel.*

ANSI B3.12, *Metal balls.*

3 Definitions

For purposes of this part of ISO 12156, the following definitions apply.

3.1 lubricity: A property of the fluid, measured by the wear scar produced on an oscillating ball from contact with a stationary plate immersed in the fluid and operating under closely controlled conditions.

3.2 MWSD: Measured mean diameter of the wear scar produced on the test ball.

3.3 WS1,4: Calculated value of wear scar diameter corrected to the standardized water vapour pressure of 1,4 kPa.

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 reservoir. The metallurgies of the ball and plate, temperature, load, frequency, and stroke length are specified. The ambient conditions during the test are used to correct the size of the wear scar generated on the test ball to a standard set of ambient conditions. The corrected wear scar diameter is a measure of the fluid lubricity.

5 Reagents and materials

5.1 Compressed air, used for drying the equipment, 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 Toluene, in accordance with ISO 5272.

Warning — Flammable. Harmful if inhaled.

5.3 Acetone, in accordance with ASTM D-329.

Warning — Extremely flammable. Vapours may cause flash fire.

5.4 Reference fluids

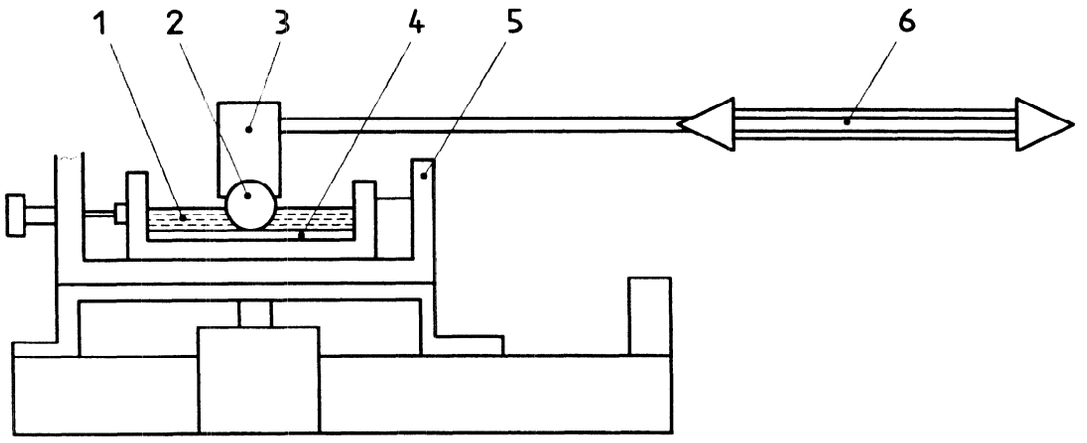
Warning — Flammable.

Two reference fluids shall be used for verifying the performance of the test apparatus. The fluids shall have significantly different lubricity performance, as measured by this International Standard. The fluids shall have certified HFRR values and humidity correction factors (HCF) from a supplier accredited to ISO/IEC Guide 25 and prepared in accordance with ISO Guide 35. They shall be clearly marked with the HFRR value (WS1,4) and its expanded uncertainty, expressed in micrometres, and with the HCF expressed in micrometres per kilopascal. The two reference fluids shall have a minimum difference in HFRR value of 200 µm, as measured by this part of ISO 12156.

NOTE ISOPAR M, which is manufactured by the Exxon Chemical Company and used as CEC Reference Fuel RF-74-T-95, has been found to be satisfactory for the basis of a low-lubricity reference fluid.

The fuel qualified for use in the Caterpillar 1H or 1G single-cylinder tests, fuel conforming to ISO 4113:1988, *Road vehicles — Calibration fluid for diesel injection equipment*, or CEC Reference Fuel RF-90-A-92 have all been found to be satisfactory for the basis of a high-lubricity reference fluid.

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.



Key

1 Fuel bath (reservoir)
2 Test ball
3 Applied load
4 Test plate
5 Heating bath
6 Oscillating motion

Figure 1 — Example of the high-frequency reciprocating rig

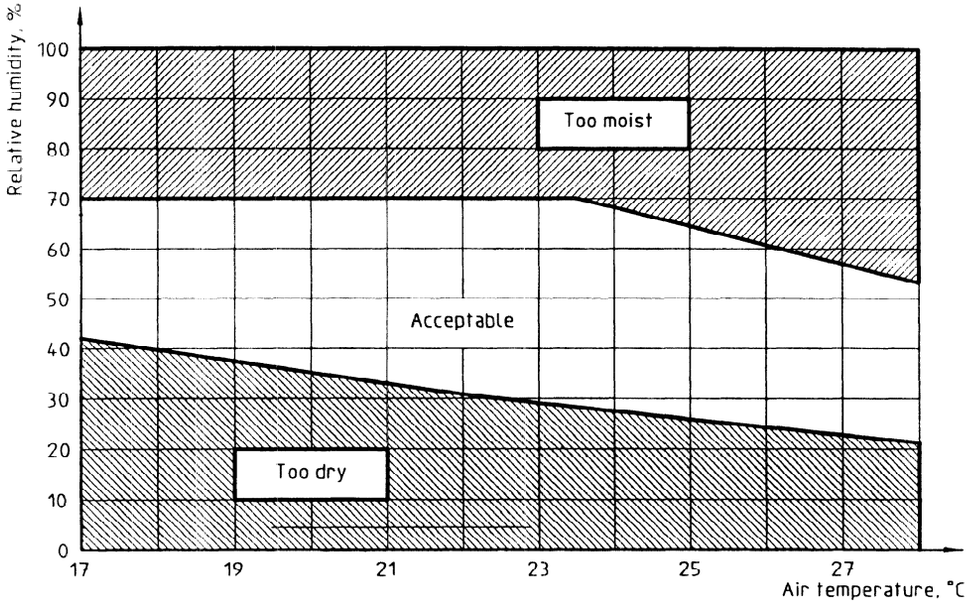


Figure 2 — Laboratory air conditions

6 Apparatus

6.1 Test apparatus

The test apparatus[2] (see figure 1) shall be 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 reservoir 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 [1]	see figure 2
Fluid temperature, °C	60 ± 2
Applied load[2], g	200 ± 1
Test duration, min	75 ± 0,1
Bath surface area, mm^2	600 ± 100
1) Laboratory air conditions as measured within 0,1 m to 0,5 m of test specimen are to be controlled to acceptable range of conditions as shown in figure 2. 2) Total applied load including fixing elements.	

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

The apparatus control unit for controlling variable parameters shall include provision for electronic data storage and retrieval.

6.2 Test plate of AISI E-52100 steel 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.

6.3 Test ball, 6 mm in diameter, grade 28 according to ANSI B3.12 of AISI E-52100 steel. It shall have a Rockwell hardness "C" scale (HRC) number of 58 to 66 (according to ISO 6508) and a surface finish of $Ra < 0,05$ µm.

6.4 Microscope or similar imaging device, capable of × 100 magnification and capable of measuring to within 1 µm.

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

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

6.7 Fuel containers of epoxy lined steel, unless it can be shown that alternative materials give equivalent results.

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

6.9 Test mass of 200 g, including any attaching apparatus for fixing to the vibrator arm.

7 Preparation and calibration

7.1 Preparation of apparatus

7.1.1 Test plates and balls - as received

Using clean forceps, place a number of plates (shiny side up) and balls as received into a clean glass wide-necked jar, and cover with toluene. Leave to soak for a minimum of 12 h, then place the jar in the ultrasonic cleaning bath for 10 min. Transfer the plates (shiny side up) and balls into a jar of fresh toluene.

7.1.2 Hardware

Place the sample holders, screws, and all hardware and utensils that come into contact with the test fluid, together with a plate and ball cleaned according to 7.1.1, in a clean glass beaker and cover with toluene. Place the beaker in the

[2] HFRR units, HFR2, supplied by PCS Instruments, 5 Warple Mews, Warple Way, London W3 ORF, England, 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. Equivalent products may be used if they can be shown to lead to the same results.

ultrasonic cleaning bath for 10 min, then using clean forceps transfer the hardware and test specimens into a beaker of acetone. Place in the ultrasonic bath for 2 min. Remove the components, and, if not to be used immediately, store in the desiccator.

7.2 Calibration and correction

7.2.1 Temperature

The temperature control of the specimen bath (6.1) shall be checked using a calibrated temperature measuring device.

7.2.2 Frequency

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

7.2.3 Stroke length

The stroke length shall be checked by measuring the full length of the wear scar on the test plate, using a calibrated microscope, after running a test on the low-lubricity reference fluid. The mean width of the wear scar is subtracted from the length of the wear scar to give the actual stroke length.

7.2.4 Run time

The run time should be checked with a calibrated timer (6.8).

7.2.5 Test rig performance

The instrument performance shall be checked by running a single test (as described in clauses 7, 8 and 9) on each of the two reference fluids. The certified value of HCF for that reference fluid shall be used in calculating the value of WS1,4.

If WS1,4 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 (7.2.1 to 7.2.4) must be performed. If the result for the low-lubricity fluid is too low it may need to be replaced.

Referencing tests shall be conducted using each reference fluid after every 25 tests or every 10 test days, whichever is shorter.

NOTE During the testing to develop this test method, the MWSD (10.1) for the low-lubricity reference fluid was observed to change during storage. It may need to be replaced regularly and should not be used if older than six months.

8 Test procedure

8.1 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, reservoir, and fixtures) from contamination by using clean forceps and ensure that the specimens do not become scratched.

8.2 Using forceps, place the test plate into the specimen bath, shiny side up. Secure the test plate to the bath and the bath to the test rig. Ensure that the thermocouple is properly placed in the reservoir.

8.3 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.

8.4 Measure the average temperature and relative humidity within 0,1 m to 0,5 m of the specimen bath. If the average values do not conform to the requirements of figure 2, steps must be taken to change the humidity before the test can proceed. Record the average values of temperature and relative humidity.

8.5 Using a disposable pipette, place 2 ml of the test fuel into the bath.

8.6 Lower the vibrator arm and suspend a 200 g weight from the arm, ensuring that the load and suspension strings hang freely.

8.7 Set the temperature controller to the desired test temperature. Set the stroke length. Set the vibration frequency. Initiate the test.

8.8 Operate the test for 75 min. At the completion of the test, switch off the vibrator unit and the heater and remove the suspended weight. Lift up the vibrator arm and remove the upper specimen holder.

8.9 Take a measurement of the average temperature and relative humidity within 0,1 m to 0,5 m of the specimen bath. These measurements shall be in accordance with the requirements of

figure 2 for the test to be valid. Record the average temperature and relative humidity.

8.10 Without removing the ball from the test ball holder, rinse several times using toluene, then several times using acetone, and place the holder in a beaker of fresh toluene. Place the beaker in the ultrasonic bath for 30 s.

8.11 Transfer the holder into a beaker of fresh acetone, and place the beaker in the ultrasonic bath for 30 s. After allowing the test ball holder to air dry, use a permanent marker to circle the wear scar.

8.12 Remove the specimen bath and properly discard the fuel. Inspect the bath and record if any particles are present. Without removing the plate from the specimen holder, rinse several times with toluene, then several times with acetone, and place the holder in a beaker of fresh toluene. Put the beaker in the ultrasonic cleaning bath for 30 s.

8.13 Transfer the holder into a beaker of fresh acetone, and place the beaker in the ultrasonic cleaning bath for 30 s. After allowing the plate to air dry, remove it from the holder and place in a storage receptacle (plastic bag) marked with the unique test reference.

8.14 With the test ball still in the holder, position it under the microscope and measure the wear scar diameter according to clause 9.

8.15 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.

9 Measurement of wear scar

9.1 Turn on the microscope light and position the test ball under microscope at × 100 magnification.

9.2 Focus the microscope and move the test ball such that the wear scar is centered within the field of view. Adjust the illumination until the edge of the wear scar can clearly be seen; refer to annex A for guidance, if necessary.

9.3 Measure the scar diameter in the x and y directions (10.1) to the nearest 1 µm. Record the readings on the data sheet. If the difference between the x and y wear scar measurements, (i.e. $x - y$), is outside the range $^{+100}_{-30}$ µm, check that the scar boundary has been correctly identified.

9.4 Record the condition of the wear area, that is, debris colour, unusual particles or wear pattern, visible galling, etc.

10 Calculations

10.1 Uncorrected mean wear scar diameter (MWSD)

Calculate the mean wear scar diameter, MWSD, in micrometres, as follows:

$$\text{MWSD} = (x + y)/2$$

where

x is the scar dimension perpendicular to oscillation direction, in micrometres;

y is the scar dimension parallel to oscillation direction, in micrometres.

10.2 Initial absolute vapour pressure (AVP_1)

Calculate the initial absolute vapour pressure, AVP_1, in kilopascals, as follows:

$$AVP_1 = \frac{RH_1 \times 10^v}{750}$$

where

RH_1 is the relative humidity at the start of the test, in percent;

$$v = 8{,}017\ 352 - \frac{1\,705{,}984}{231{,}864 + T_1};$$

T_1 is the air temperature at the start of the test, in degrees Celsius.

10.3 Final absolute vapour pressure (AVP_2)

Calculate the final absolute vapour pressure, AVP_2, in kilopascals, as follows:

$$AVP_2 = \frac{RH_2 \times 10^v}{750}$$

where

RH_2 is the relative humidity at the end of the test, in percent;

$$v = 8{,}017\ 352 - \frac{1\,705{,}984}{231{,}864 + T_2};$$

T_2 is the air temperature at the end of the test, in degrees Celsius.

10.4 Mean absolute vapour pressure (AVP)

The mean absolute vapour pressure during the test is given by:

$$AVP = \frac{AVP_1 + AVP_2}{2}$$

10.5 Corrected wear scar diameter (WS1,4)

The corrected wear scar diameter WS1,4, in micrometers, is given by:

$$WS1{,}4 = MWSD + HCF(1{,}4 - AVP)$$

where HCF = 60 for unknown fuel samples.

11 Test report

The test report shall include:

a) reference to this part of ISO 12156;

b) unambiguous description of the fuel tested;

c) x and y wear scar dimensions, and the uncorrected mean wear scar diameter (MWSD) to the nearest 1 µm;

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

e) calculated mean absolute vapour pressure (AVP);

f) corrected mean wear scar diameter (WS1,4) to the nearest 1 µm;

g) description of the wear scar area;

h) identification of the specimens tested;

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

j) date of test.

12 Precision and bias

The precision was developed for fuels with an average wear scar diameter between 360 µm and 600 µm. The precision of this test method was determined by the statistical examination of inter-laboratory test results using ISO 4259.

12.1 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 63 µm in only one case in twenty.

12.2 Reproducibility

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, exceed 102 µm in only one case in twenty.

12.3 Bias

The procedure in this test method has no bias because the value of lubricity can be defined only in terms of a test method.

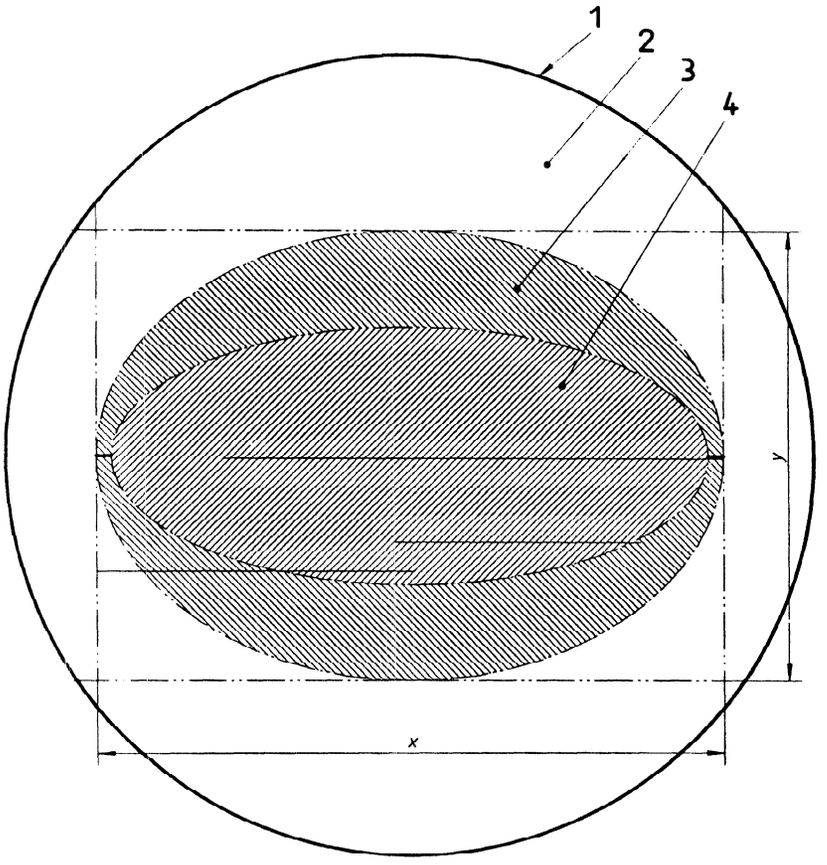
Annex A
(informative)

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-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; see figure A. 1.

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 (reduced diameter)
2 Unworn area
3 Less distinct worn area
4 Worn area

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

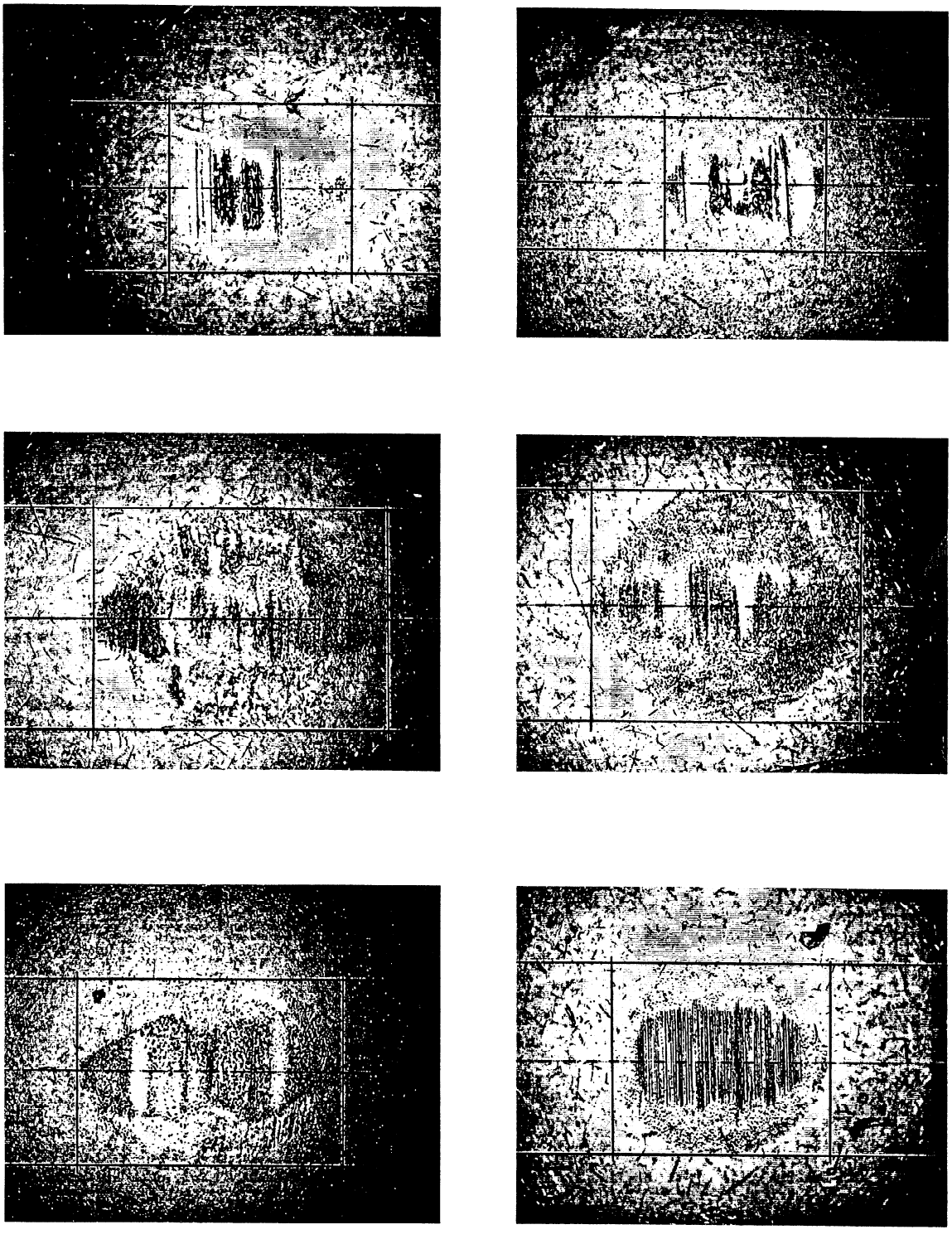


Figure A.2 — Examples of wear scars

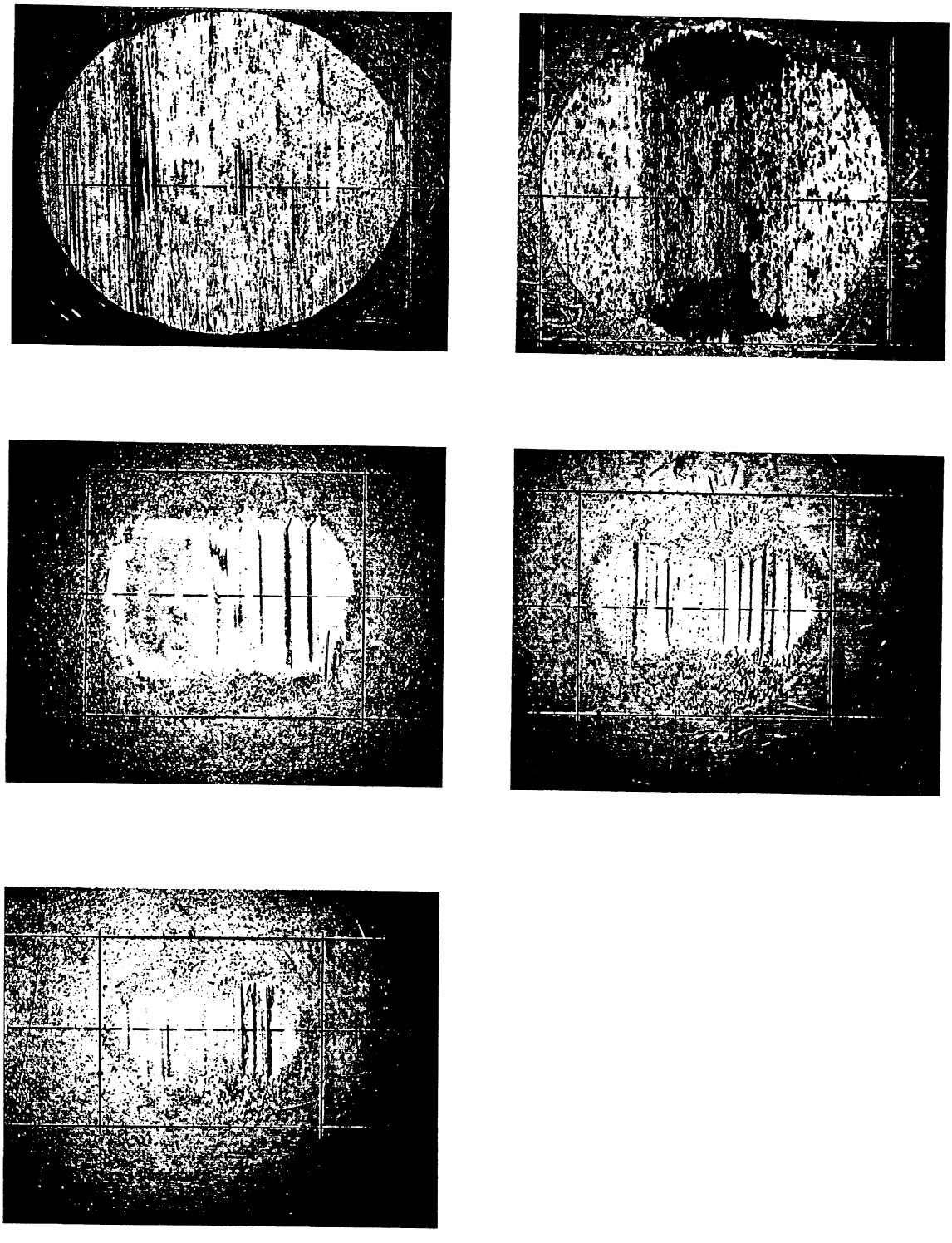


Figure A.2 — **Examples of wear scars** (concluded)

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