

Standard Test Method for Measurement of Extreme-Pressure Properties of Lubricating Grease (Four-Ball Method)¹

This standard is issued under the fixed designation D2596; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

- 1.1 This test method covers the determination of the load-carrying properties of lubricating greases. Three determinations are made:
 - 1.1.1 Load-Wear Index (formerly called Mean-Hertz Load),
- 1.1.2 Weld Point, by means of the Four-Ball Extreme-Pressure (EP) Tester, and
 - 1.1.3 Last nonseizure load (LNSL).
- 1.2 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.
- 1.3 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 applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 American National Standard:² B3.12 Metal Balls

3. Terminology

- 3.1 Definitions:
- 3.1.1 *compensation line*, *n*—a line of plot on log-log paper where the coordinates are scar diameter in millimetres and applied load in kilograms-force (or Newtons) obtained under dynamic conditions.
 - 3.1.1.1 *Discussion*—Shown in Fig. 1 as line ABE.
- 3.1.2 *compensation scar diameter*—the average diameter, in millimetres, of the wear scar on the stationary balls caused by the rotating ball under an applied load in the presence of a lubricant, but without causing either seizure or welding.
- ¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricantsand is the direct responsibility of Subcommittee D02.G0.04 on Functional Tests Tribology.
- Current edition approved July 1, 2015. Published September 2015. Originally approved in 1967. Last previous edition approved in 2014 as D2596 14. DOI: 10.1520/D2596-15.
- 2 Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

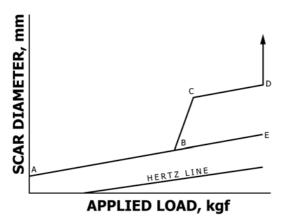
- 3.1.3 *corrected load, n*—the load in kilograms-force (or Newtons) obtained by multiplying the applied load by the ratio of the Hertz scar diameter to the measured scar diameter at that load
- 3.1.3.1 *Discussion*—In this test method, the corrected load is calculated for each run.
- 3.1.4 *hertz line*, *n*—a line of plot on log-log paper where the coordinates are scar diameter in millimetres and applied load in kilograms-force (or Newtons) obtained under static conditions.
 - 3.1.4.1 Discussion—Shown in Fig. 1 as a hertz line.
- 3.1.5 hertz scar diameter, n—the average diameter, in millimetres, of an indentation caused by the deformation of the balls under static load (prior to test). It may be calculated from the equation:

$$D_h = 8.73 \times 10^{-2} (P)^{1/3} \tag{1}$$

where:

 D_h = Hertz diameter of the contact area in millimetres, and P = static applied load in kilograms-force.

- 3.1.6 *immediate seizure region, n*—that region of the scarload curve characterized by seizure or welding at the startup or by large wear scars.
- 3.1.6.1 *Discussion*—Under conditions of this test method, the immediate seizure region is shown by line CD. Also, initial deflection of indicating pen on the optional friction-measuring device is larger than with nonseizure loads.
- 3.1.7 *incipient seizure or initial seizure region*, *n*—that region at which, with an applied load, there is a momentary breakdown of the lubricating film.
- 3.1.7.1 *Discussion*—This breakdown is noted by a sudden increase in the measured scar diameter, shown in Fig. 1 as line BC, and a momentary deflection of the indicating pen of the optional friction-measuring device.
- 3.1.8 *last nonseizure load, n*—the last load at which the measured scar diameter is not more than 5 % greater than the compenation value at that load.
 - 3.1.8.1 *Discussion*—Shown in Fig. 1 as Point B.
- 3.1.9 *load-wear index (or the load-carrying property of a lubricant), n*—an index of the ability of a lubricant to minimize wear at applied loads.



ABE—compensation line.
B—Point of last nonseizure load.
BC—Region of incipient seizure.
CD—Region of immediate seizure.
D—Weld point.

FIG. 1 Schematic Plot of Scar Diameter Versus Applied Load

- 3.1.9.1 *Discussion*—Under the conditions of this test, specific loadings in kilograms-force (or Newtons) having intervals of approximately 0.1 logarithmic units, are applied to the three stationary balls for ten runs prior to welding. The load-wear index is the average of the sum of the corrected loads determined for the ten applied loads immediately preceding the weld point.
- 3.1.10 *weld point, n*—the lowest applied load at which sliding surfaces seize and then weld.
- 3.1.10.1 *Discussion*—Under the conditions of this test, the lowest applied load in kilograms-force (or Newtons) at which the rotating ball seizes and then welds to the three stationary balls, indicating the extreme-pressure level of the lubricating grease has been exceeded. See Fig. 1, Point D.
- 3.1.10.2 *Discussion*—Some lubricating greases do not allow true welding, and extreme scoring of the three stationary balls results. In such cases, the applied load which produces a maximum scar diameter of 4 mm is reported as the weld point.

4. Summary of Test Method

4.1 The tester is operated with one steel ball under load rotating against three steel balls held stationary in the form of a cradle. The rotating speed is 1770 r/min \pm 60 r/min. Lubricating greases are brought to 27 °C \pm 8 °C (80 °F \pm 15 °F) and then subjected to a series of tests of 10 s duration at increasing loads until welding occurs.

5. Significance and Use

- 5.1 This test method, used for specification purposes, differentiates between lubricating greases having low, medium, and high level of extreme-pressure properties. The results do not necessarily correlate with results from service.³
- 5.2 It is noted that lubricating greases that have as their fluid component a silicone, halogenated silicone, or a mixture

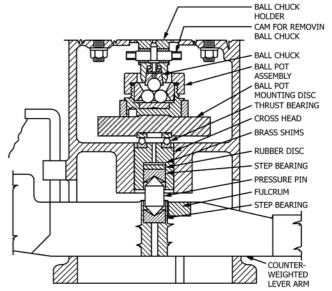


FIG. 2 Sectional View of Four-Ball EP Tester

comprising silicone fluid and petroleum oil, are not applicable to this method of test.

6. Apparatus

6.1 Four-Ball Extreme-Pressure Lubricant Tester, ⁴ illustrated in Fig. 2.

Note 1—It is important to distinguish between the Four-Ball EP Tester and the Four-Ball Wear Tester. The Four-Ball Wear Tester can be used under a variety of test conditions at loads up to 490 N (50 kgf). The Four-Ball EP Tester is designed for testing under more severe conditions and lacks the sensitivity necessary for the Four-Ball Wear Test.

- 6.2 *Microscope*, ⁴equipped with calibrated measuring scale and readable to an accuracy of 0.01 mm.
 - 6.3 Timer, graduated in tenths of a second.

Note 2—Optional equipment with Four-Ball apparatus consists of a friction-measuring device electrically driven and conveniently graduated in $10 \, \mathrm{s}$ markings.

7. Materials

7.1 Cleaning Fluids—For preparing balls and apparatus for the test should be those capable of removing metal preservative coating from the balls, eliminating carryover effects from one test to the next. The cleaning fluid selected should be non-film-forming and not contribute to the wear or antiwear properties of the test lubricant (for example, chlorinated solvents should *not* be used).

Note 3—Previously, to remove the corrosion inhibitor coating on the test balls, first washing with Stoddard Solvent (Warning—See 7.1.1), followed by rinsing with n-heptane (Warning—See 7.1.2), and allowing to air dry has proven to satisfactory remove the corrosion inhibitor coating. Depending on the lubricating grease chemistry, other solvents

³ Further details on this test method may be found in: Sayles, F. S., et al, *National Lubricating Grease Institute Spokesman*, Vol 32, No. 5, August 1968, pp. 162 –167.

⁴ The sole source of supply of the apparatus (microscopes 103.10 A and 103.10 B) known to the committee at this time is Falex Corporation, 1020 Airpark Dr., Sugar Grove, IL, 60554-9585. 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, ¹ which you may attend.

may be required to completely remove the residual lubricating grease from the test cup assembly parts. It is up to the user of this test method to determine the solvents required to thoroughly clean the ball cup assembly parts and other parts of the test machine that have come in contact with the lubricating grease being tested.

- 7.1.1 Stoddard Solvent or Mineral Spirits, Reagent Grade—(Warning—Combustible. Health hazard.)
- 7.1.2 *ASTM n-heptane*⁵—(**Warning**—Flammable. Health hazard.)
- 7.2 Test Balls⁶—Test balls shall be chrome alloy steel, made from AISI standard steel No. E-52100, with diameter of 12.7 mm (0.5 in.), Grade 25 EP (Extra Polish). Such balls are described in ANSI Specifications B3.12, for Metal Balls. The Extra-Polish finish is not described in that specification. The Rockwell C hardness shall be 64 to 66, a closer limit than is found in the ANSI requirement.

8. Preparation of Apparatus

8.1 Thoroughly clean four new test balls, ball pot, and chuck assemblies by first washing using the solvents selected in 7.1.

Note 4—Previously, users were directed to first wash the parts with Stoddard Solvent (**Warning**—See 7.1.1) to remove the corrosion inhibitor coating, followed by a rinse with n-heptane (**Warning**—See 7.1.2), and allowing to air dry. These are listed as guidance, but are not mandatory.

Note 5—Do not use solvents such as carbon tetrachloride or other solvents that may inherently possess extreme pressure properties which may affect the results.

8.2 Lower the crosshead by raising the lever arm. Lock the lever arm in the raised position by means of a locking arrangement for that purpose.

9. Procedure

- 9.1 Bring lubricant to be tested to 27 °C \pm 8 °C (80 °F \pm 15 °F).
- 9.2 Completely fill the ball pot with the lubricating grease to be tested, avoiding the inclusion of air pockets. Imbed the three steel test balls in the grease. Place the lock ring carefully over the three balls and screw down the lock nut securely. Scrape off the excess grease pushed onto the lock nut.

Note 6—Subsequent independent investigations reported in 1971 by several laboratories indicate that optimum test repeatability is obtained when the force on the lock-down nut is maintained within the range 50 ft·lbf \pm 5 ft·lbf (68 N·m \pm 7 N·m), applied and measured by means of a torque wrench. Significantly lower weld points were obtained when the force applied was approximately 100 ft·lbf (136 N·m).

- 9.3 Press one ball into the ball chuck and mount the chuck into chuck-holder.
- 9.4 Examine the ball chuck carefully before each run. The chuck is continually subjected to wear and seizure and should be replaced when it will not fit into the ball chuck-holder tight enough to support its own weight, or if the ball seat shows signs of seizure.

- 9.5 Install the ball pot assembly on the test apparatus in contact with the fourth ball. Place the mounting disk between ball pot and thrust bearing.
- 9.6 Place the weight tray and weights on the horizontal arm in the correct notch for a base test load of 784 N (80 kgf). Release the lever arm and gently apply (Note 7) the test load to the balls, making certain the ball pot assembly and mounting disk are centered. If the optional friction-measuring device is used, connect the calibrated arm on the ball pot to the indicator spring by means of the clip and wire, placing clip and indicator support over the numbers which correspond to the applied load.

Note 7—Shock-loading should be avoided as it may deform the balls permanently.

- 9.7 Start the motor and run for $10 \text{ s} \pm 0.2 \text{ s}$. The time for the apparatus to "coast" to a stop is not considered.
- 9.8 Remove the load from the balls by raising the lever arm and locking it in raised position. If the friction-measuring device is used, remove clip and wire. Remove the ball pot assembly; remove the chuck and discard the ball.
 - 9.9 Measure the scar diameter of test balls as follows:
- 9.9.1 Option A—Remove the lock nut and release the test balls. Clean the balls with the solvents selected in 7.1 and then *n*-heptane, and wipe dry with soft cloth. Place the individual balls on a suitable holder and by means of a microscope, measure to the nearest 0.01 mm the scar diameters both parallel (horizontal) and normal (vertical) to the striations in the scar surface of one of the three test balls.
- 9.9.2 Option B—Retain the balls in the ball pot. Wipe excess grease from the balls and ball pot. Wash the ball surfaces with the solvents selected in 7.1 and then *n*-heptane. Using a microscope, measure to the nearest 0.01 mm the scar diameters both parallel (horizontal) and normal (vertical) to the striations in the scar surface of one of the three test balls. Measurement by microscope of the scar diameters on all three balls rather than one ball as outlined in Options A or B may be made if the operator so desires.
- 9.10 Record (Table 1, Column 5) for the 784 N (80 kgf) load the average scar diameter by any one of the two techniques described in 9.9. Compare this average scar diameter (Table 1, Column 5) to the compensation line average scar diameter (Table 1, Column 6). If the average scar diameter is not more than 5 % from the compensation scar diameter (Table 1, Column 7), repeat the test at the next higher load (Table 1, Column 1), and again compare scar diameters. Continue this procedure until the last nonseizure load is determined.
- 9.11 If the measured scar diameter for the 784 N (80 kgf) load is more than 5 % from the compensation scar diameter, the next run is made at the next lower load (Table 1, Column 1). Continue this procedure until the last nonseizure load is determined.

Note 8—When the optional friction-measuring device is used, the last nonseizure load is detected by a gradual transverse movement of the indicating pen.

9.12 Make additional runs at consecutively higher test loads (Table 1, Column 1), recording the measured scar diameter(s)

⁵ Described in the 1998 *Annual Book of ASTM Standards*, Vol 05.04, Motor Fuels, Section I, Annex 2, Section A2.7, Reference Materials.

⁶ Steel balls meeting this description were used in developing the precision of the test. They are available from ball bearing or laboratory equipment manufacturers and distributors. All balls used in one test should be taken from one carton (of 500 balls) as received from the supplier.

TABLE 1 Suggested Form for Recording Test Results

Column 1 Applied Load, kgf ^A (L)	Column 2 Scar Diameter Ball 1, mm	Column 3 Scar Diameter Ball 2, mm	Column 4 Scar Diameter Ball 3, mm	Column 5 Average Scar Diameter, mm (\bar{X})	Column 6 Compensation Scar Diameter, mm	Column 7 Compensation Scar Diameter, +5 % mm	Column 8 LD _n Factor	Column 9 Corrected Load, kgf LD_{n}/\bar{X}
6							0.95	
8							1.40	
10							1.88	
13		•••		•••	0.21	0.22	2.67	
16		•••		•••	0.23	0.24	3.52	
20		•••		•••	0.25	0.26	4.74	
24		•••		•••	0.26	0.27	6.05	
32					0.29	0.30	8.87	
40					0.31	0.33	11.96	
50		•••		•••	0.34	0.36	16.10	
63		•••		•••	0.37	0.39	21.86	
80		•••		•••	0.40	0.42	30.08	
100					0.44	0.46	40.5	
126					0.47	0.49	55.2	
160					0.52	0.55	75.8	
200		•••		•••	0.58	0.61	102.2	
250							137.5	
315							187.1	
400							258	
500							347	
620				•••			462	
800							649	

^A To convert from kilograms-force to newtons, multiply by 9.806.

and discarding test balls, until welding occurs. Make a check run at this point. If welding does not occur on the check run, then repeat the test at the next higher load until welding is verified.

- 9.13 Shut off the motor immediately to prevent damage to the tester. Excessive wear or seizure of the ball and ball chuck may result if caution is not observed. Welding may be detected by any or all of the following:
- (1) If friction-measuring device is used, a sharp transverse movement of the indicating pen.
 - (2) Increased noise level of motor.
 - (3) Smoking from the ball pot.
 - (4) A sudden drop in the lever arm.

10. Calculations and Reports

10.1 *Corrected Load*—Calculate and record (Table 1, Column 9) for each applied load between the last nonseizure load and weld point using the equation:

Corrected load,
$$kgf = LD_b/X$$
 (2)

where:

applied load, kgf, that is, total weight applied (tray and weights) multiplied by lever arm ratio,

 D_h = Hertz scar diameter, mm, and

X = average scar diameter, mm.

10.2 Load-Wear Index (see Note 9)—Calculate and report the Load-Wear Index (formerly Mean-Hertz Load) in kilograms-force using the equation:

Load – Wear Index,
$$kgf = A/10$$
 (3)

where:

A = sum of the corrected loads determined for the ten applied loads immediately preceding the weld point (Note 10).

Note 9—The method of calculation was amended in 1982. For a lubricant with a weld point of $400~\mathrm{kg}$ or less the Load Wear Index is the same with the old and the present method of calculation.

Note 10—If tests applied to the lubricant indicate it follows the compensation line, then A may be defined as:

A = sum of corrected loads plus compensation line corrected loads, for the ten applied loads immediately preceding the weld point.

For convenience, Table 2 has been constructed to give the compensation line corrected load for any portion of the compensation line. This value is obtained by noting the intersection of the last nonseizure load and weld load values. For example: the last nonseizure load of a lubricant was found to be 490 N (50 kgf). Subsequent runs in the seizure portion of the curve were made at 618 N, 784 N, 981 N, 1236 N, and 1569 N (63 kgf, 80 kgf, 100 kgf, 126 kgf, and 160 kgf) loads with weld-point found to be 1961 N (200 kgf). Table 2 notes the value at intersection of 490 N and 1961 N (50 kgf and 200 kgf) to be 1456 N (148.6 kgf). This value, the compensation line corrected load, was obtained by correcting loads of 490 N, 392 N, 314 N, 235 N, and 196 N (50 kgf, 40 kgf, 32 kgf, 24 kgf, and 20 kgf) using compensation line scar diameters. This fulfills the definition of Load-Wear Index, that a total of twelve runs be made, the eleventh run causing welding of the test balls and the twelfth to verify the weld point. If the tests applied to the lubricant indicate the wear scars do not follow the compensation line, then Table 2 cannot be applied and actual determinations must be made for all ten applied loads preceding the weld point.

10.3 *Weld Point*—Report the verified weld point as found in 9.12.

10.4 Last Nonseizure Load—Report the last nonseizure load (LNSL) as determined in 9.10.

11. Precision and Bias⁷

11.1 The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

11.1.1 Load Wear Index (Formerly Mean-Hertz Load):

⁷ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1182.

TABLE 2 Total of Compensation Line Corrected Loads

Last Non- seizure — Load, kgf ^A	Weld Load kgf											
	800	620	500	400	315	250	200	160	126	100	80	
200	583	639	684	720	749	770						
160	410	466	511	547	576	597	615					
126	269.8	325.8	370.5	407	435	457	474	489				
100	159.7	215.8	260.5	296.7	325.3	346.9	364.4	378	390			
80	71.6	127.7	172.4	208.6	237.2	258.2	276.3	290	302	311.0		
63		56.1	100.8	137.0	165.6	187.1	204.7	218.8	230.4	239.3	246.7	
50			44.7	80.9	109.5	131.0	148.6	162.7	174.3	183.2	190.6	
40				36.2	64.8	86.4	103.9	118.0	129.6	138.6	145.9	
32					28.6	50.2	67.7	81.8	93.4	102.4	109.7	
24						21.6	39.1	53.2	64.8	73.8	81.1	
20							17.6	31.6	43.2	52.2	59.5	
16								14.1	25.7	34.6	42.0	
13									11.6	20.6	27.9	
10										9.0	16.3	
8											7.4	

^A To convert from kilograms-force to newtons, multiply by 9.806.

11.1.1.1 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 the following values only in one case in twenty:

$$0.14\left(\bar{X}\right) \tag{4}$$

where:

 \bar{X} = average of two test results.

11.1.1.2 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, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

$$0.44\left(\bar{X}\right) \tag{5}$$

where:

 \bar{X} = average of the two test results.

Note 11—There were eight laboratories that participated in the round robin. Statistics were derived in accordance with the ISO 4259 Precision Program.

11.1.2 *Weld Point*—Precision data were determined on samples having weld points up to a maximum of 6080 N (620 kgf).

Note 12—No precision data are currently available for higher weld point lubricants.

11.1.2.1 Repeatability—The difference between two tests 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 one increment loading only in one case in twenty.

11.1.2.2 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, in the normal and correct operation of the test method, exceed one increment loading only in one case in twenty.

11.2 Last Non-Seizure Load:

Repeatability =
$$0.35 \left(\bar{X}\right)$$
 (6)

Reproducibility =
$$0.78 \left(\bar{X} \right)$$
 (7)

where:

 \bar{X} = average of the two test results.

11.3 *Bias*—The procedure in this test method has no bias because the value of extreme-pressure can be defined only in terms of a test method.

12. Keywords

12.1 extreme pressure; four-ball EP; grease; Hertz line; load-wear index; lubricating grease; weld point

SUMMARY OF CHANGES

Subcommittee D02.G0 has identified the location of selected changes to this standard since the last issue (D2596 – 14) that may impact the use of this standard. (Approved July 1, 2015.)

(1) Revised 1.1, 7.1, 8.1, 9.2, 9.9, and 9.10, adding new Note 3, Note 4, and Note 5.

Subcommittee D02.G0 has identified the location of selected changes to this standard since the last issue $(D2596 - 10^{\epsilon 1})$ that may impact the use of this standard. (Approved Oct. 1, 2014.)

(1) Added new subsection 10.4.

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