



Designation: D 5969 – 9602

An American National Standard

Standard Test Method for Corrosion-Preventive Properties of Lubricating Greases in Presence of Dilute Synthetic Sea Water Environments¹

This standard is issued under the fixed designation D 5969; 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.

1. Scope*

1.1 This test method covers the determination of the corrosion-preventive properties of greases using grease-lubricated tapered roller bearings exposed to various concentrations of dilute synthetic sea water stored under wet conditions. It is based on Test Method D 1743, which is practiced using a similar procedure and distilled water. The reported result is a pass or fail rating as determined by at least two of three bearings.

1.2 The values stated in acceptable inch-pound units for the apparatus dimensions are to be regarded as standard, and the SI conversions are provided for information only. All other values stated in acceptable SI units are standard.

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 ASTM Standards:

~~D-235 Specification 665 Test Method for Rust-Preventing Characteristics of Inhibited Mineral Spirits (Petroleum Spirits)~~

¹ This test method is under the jurisdiction of ASTM Committee ~~D-2~~ D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.G0 on Lubricating Grease.

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*A Summary of Changes section appears at the end of this standard.

~~(Hydrocarbon Dry-Cleaning Solvents) Oil in the Presence of Water²~~

~~D-665 Test Method 1193 Specification for Rust-Preventing Characteristics of Inhibited Mineral Oil in the Presence of Reagent Water³~~

~~D-1193 Specification for Reagent Water⁴~~

~~D-1743 Test 1743 Test Method for Determining Corrosion Preventive Properties of Lubricating Greases²~~

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *corrosion*—the chemical or electrochemical reaction between a material, usually a metal, and its environment that produces a deterioration of the material and its properties.

3.1.1.1 *Discussion*—In this test method, corrosion is manifested by red rust or black-spots stains on the bearing race. Stains, through which the underlying metal surface is still visible, are not considered corrosion in Test Method D 5969 and shall be ignored. **D 1743**

4. Summary of Test Method

4.1 New, cleaned, and lubricated bearings are run under a light thrust load for 60 ± 3 s to distribute the lubricant in a pattern that might be found in service. The bearings are exposed to a solution of synthetic sea water at some percentage in distilled water, then stored for $24 \pm \frac{1}{2}$ h at $52 \pm 1^\circ\text{C}$ and 100 % relative humidity. After cleaning, the bearing cups are examined for evidence of corrosion.

5. Significance and Use

5.1 This test method differentiates the relative corrosion-preventive capabilities of lubricating greases under the test conditions.

6. Apparatus

6.1 *Bearings*, Timken bearing cone and roller assembly LM11949 and cup LM11910.⁴

² ~~Annual Book of ASTM Standards, Vol 06.04, 05.01.~~

³ ~~Annual Book of ASTM Standards, Vol 05.01, 11.01.~~

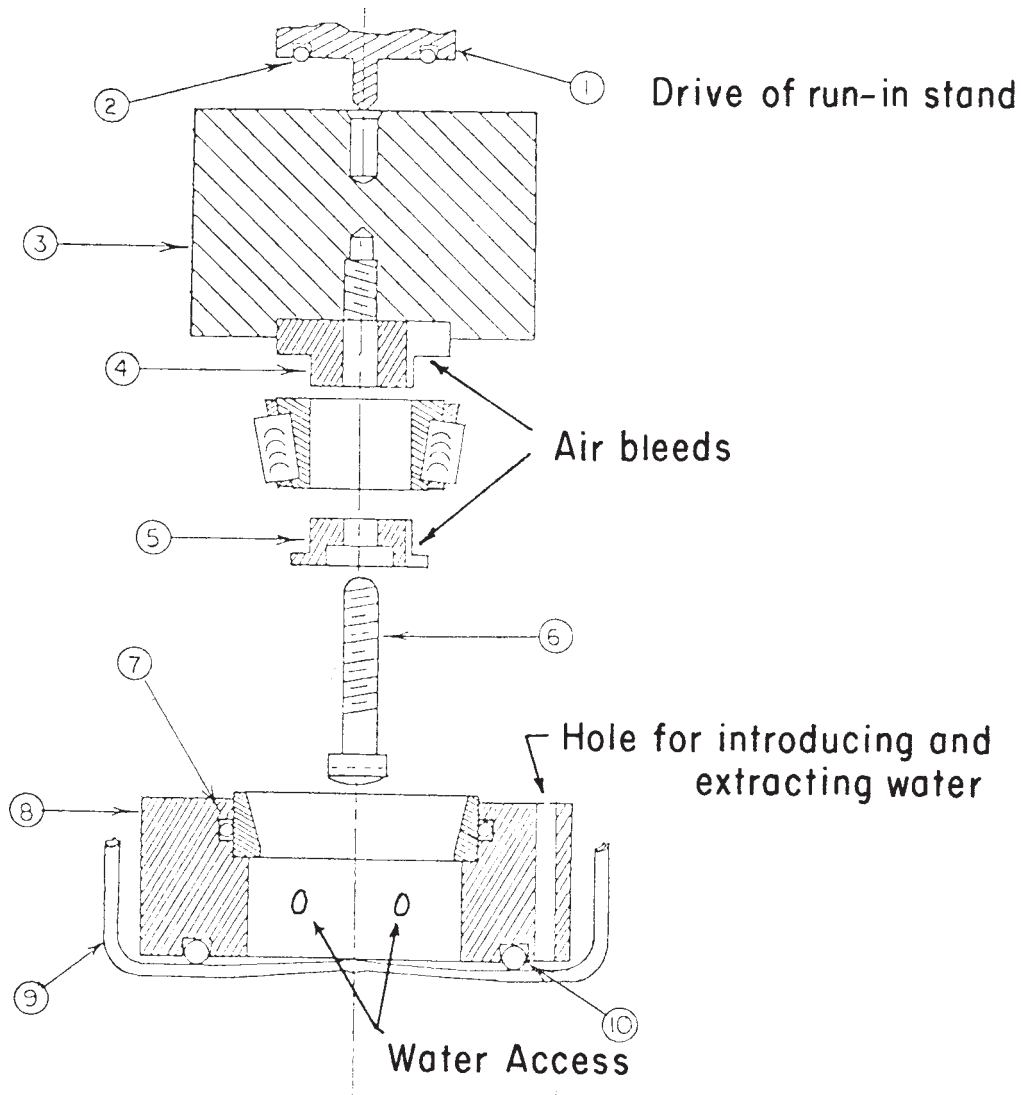
~~Annual Book of ASTM Standards, Vol 11.01.~~

⁴ Manufactured by the Timken Co., Canton, OH 44706.

- 6.2 Motor, 1750-rpm (approximate) speed, 1/15 hp (min).
- 6.3 Bearing Holder,⁵ (see Fig. 1) consists of a 1 ± 0.01 -kg mass, upper and lower plastic collars for the bearing cone (Parts A and B), a metal screw, a plastic collar for the cup (Part C), and a plastic jar with a screw cap.
- 6.4 Run-in Stand,⁵ as shown in Fig. 2.
- 6.5 Spindle/Thrust Loading Device,⁴ as shown in Fig. 3.

⁴ Manufactured by

⁵ The sole source of supply of the Timken Co., Canton, OH 44706; apparatus known to the committee at this time is Falex Corp., 1020 Airpark Drive, Sugar Grove, IL, 60554. 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 committee¹, which you may attend.



KEY	DESCRIPTION	QUANTITY
1	PISTON	1
2	O RING	1
3	WEIGHT	1
4	UPPER FLANGE	1
5	LOWER FLANGE	1
6	1/4 - 20 x 1-1/4 FILLISTER HD. MACH. SCREW S.S.	1
7	O RING	1
8	BEARING HOLDER	1
9	PLASTIC JAR	1
10	O RING	1

FIG. 1 Test Method D 1743 Bearing Holder Assembly

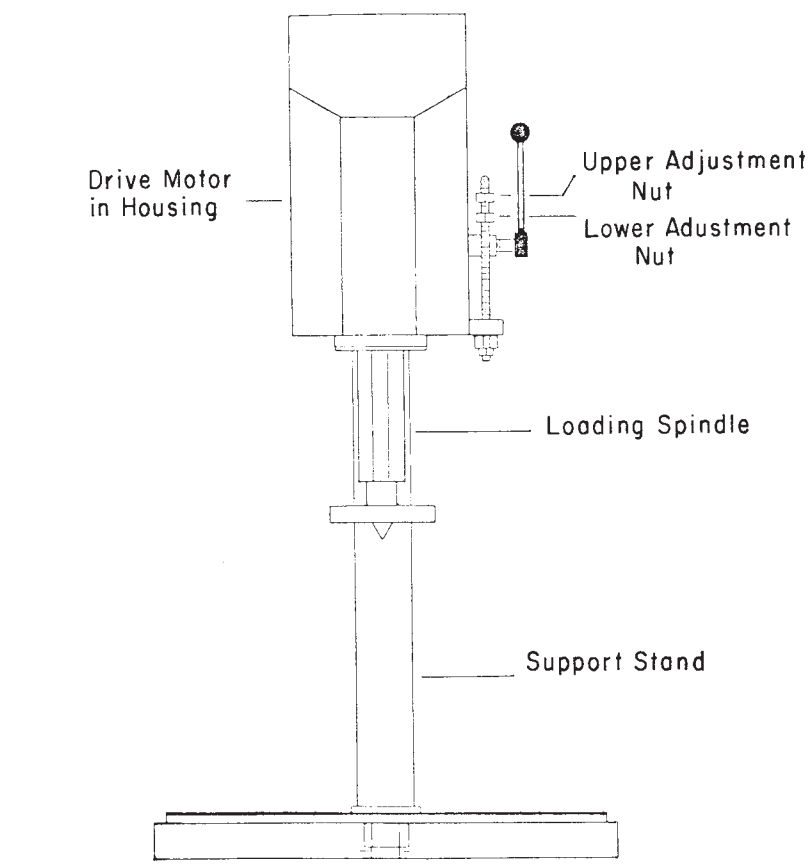


FIG. 2 Run-in Stand Drawing

- 6.6 *Mechanical Grease Packer*, as shown in Fig. 4 and Fig. 5.
- 6.7 *Pliers*,⁶ as shown in Fig. 5.
- 6.8 *Syringe*, of at least 100 ± 5 mL volume and with a needle of at least 16 gage and a minimum length of 100 mm (4 in.).
- 6.9 *Timer*, capable of measuring a 60 ± 3 -s interval.
- 6.10 *Oven*, a laboratory oven, essentially free of vibration, capable of maintaining $52 \pm 1^\circ\text{C}$.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁷ Other grades may be used, provided that it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type III.

7.3 *Isopropyl Alcohol*:

NOTE 1—**Warning: Flammable.** (**Warning—Flammable.**)

7.4 *Solvent Rinse Solution*, of the following composition by volume:

7.4.1 *Isopropyl Alcohol*, 90 %,

7.4.2 *Distilled Water*, 9 %, and

7.4.3 *Ammonium Hydroxide*, 1 %.

NOTE 2—**Warning: Poison: 1 %.** (**Warning—Poison.** Causes burns. Vapor extremely irritating. May be fatal if swallowed. Harmful if inhaled.)

⁶ This equipment is

⁶ A Waldes Truarc Plier No. 4, available from Falex Corp., 2055 Comprehensive Dr., Aurora, IL 60505; Truarc Co., or similar, modified as in Fig. 5.

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⁷ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

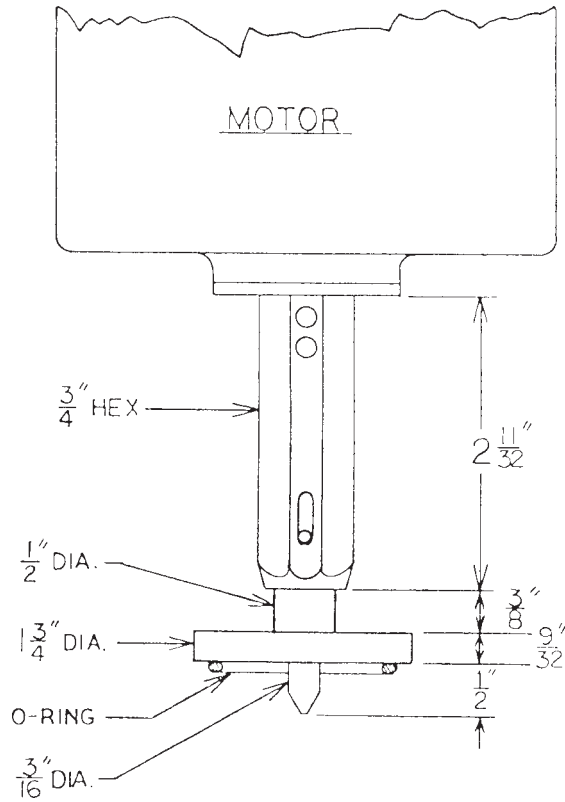


FIG. 3 Spindle/Thrust Loading Device

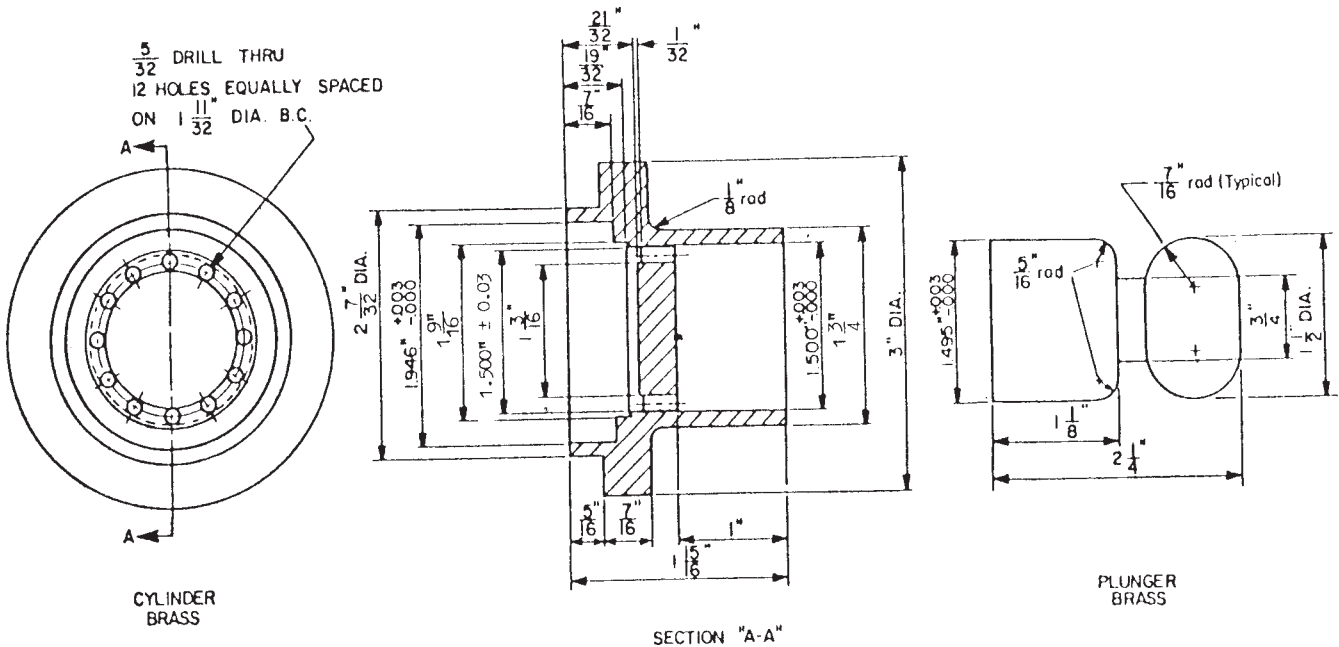


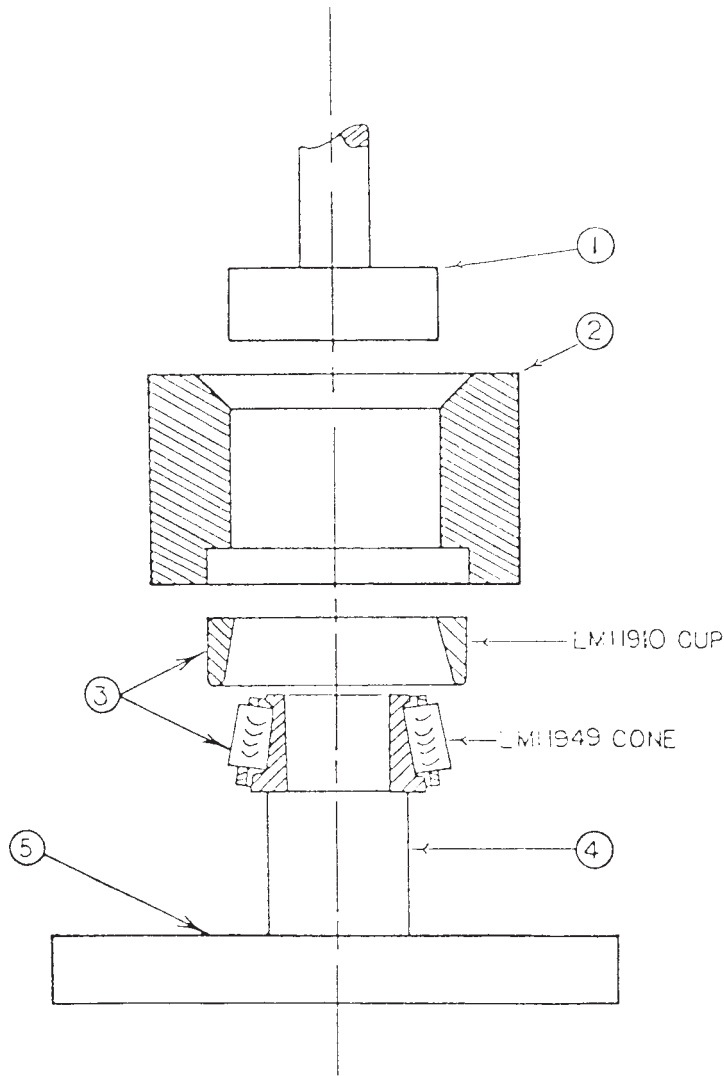
FIG. 4 Bearing Packer Brass
(Tolerances are 0.003 inches unless otherwise specified.)

7.5 Stoddard Solvent, as described in Specification D-235.

NOTE 3—**Warning:** Combustible. Reagent Grade. (**Warning**—Combustible. Vapor harmful.)

7.6 Synthetic Sea Water, prepared as described in Test Method D-665.

NOTE 4—**Warning:** Poison. D 665. (**Warning**—Poison. May be fatal if swallowed.)



KEY	DESCRIPTION	QUANTITY
1	GREASE PACK PLUNGER	1
2	CYLINDER	1
3	LM11900 BEARING ASSEMBLY	1
4	STUD	1
5	BASE	1

FIG. 4 Bearing Packer—Alternative (continued)

8. Standardization of Thrust Loading Device

8.1 Pack a bearing, and install it into the holder. Place the assembly into a plastic jar as described in 10.1-10.4. Place the jar onto the base of the motor drive spindle and center it under the indexing pin of the drive. Lower the drive until the O-ring just contacts the 1-kg weight. Run the bottom nut of the depth gage (see Fig. 2) down to the stop. Place a 3-mm spacer on top of this nut. Bring the top nut down to the spacer. While holding the top nut in position, remove the spacer and run the bottom nut up and tighten it against the top nut. When the O-ring is compressed against the 1-kg weight until the adjustment nut hits the stop, there will be a 29-N load added, giving a total load of 39 N on the bearing. (The loads described are provided by the forces of the spring in the thrust loading spindle and the sum of the 1-kg weight and spring, respectively. These loads are approximate. The 1-kg weights should be within 0.010 kg of their stated values. The thrust loading spindle should be calibrated by some suitable method when it is first put into service, recalibrated periodically, and replaced if its spring does not provide sufficient force to spin the test bearings without slippage during the 60-s run to distribute the grease.) Examine the O-ring periodically, and replace it if it shows any cracks or other signs of deterioration.

8.2 The thrust loading device should be standardized before use, once per day if used daily, and again if there is reason to believe that the standardization has changed. The thrust loading device may be standardized using one of the greases to be tested.

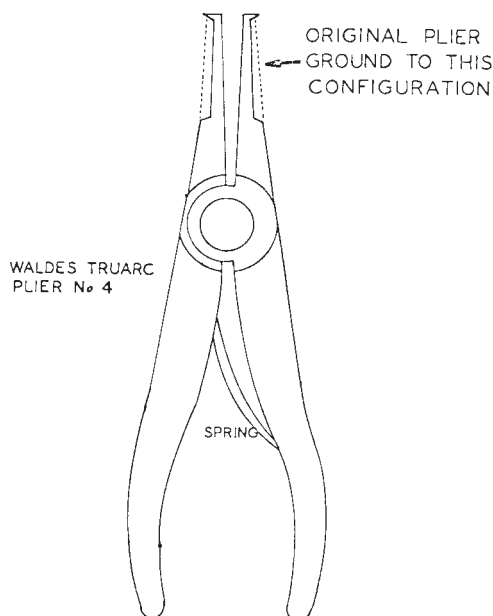


FIG. 5 Plier to Remove Bearing from Grease Packer

9. Preparation of Bearings

9.1 Examine the test bearings carefully and select only bearings that have outer races (cups) and rollers entirely free of corrosion. During the bearing preparation, handle the bearings with tongs or rubber or plastic gloves. Do not touch bearings with the fingers at any time.

9.2 Wash the selected bearings thoroughly in hot (52 to 66°C) Stoddard solvent (**Warning**—see [Note 3](#)) [7.5](#)) to remove the rust preventive. To ensure complete removal of the rust preventive, subject the bearings to a second wash in fresh hot Stoddard solvent.

NOTE 51—The washing temperatures specified are considerably above the flash point of the solvent. Accordingly, the washing operation should be carried out in a well-ventilated hood where no flames or other ignition sources are present.

9.3 Transfer the bearing from the Stoddard solvent to the solvent rinse solution (**Warning**—see [Note 2](#)) [7.4.3](#)) to remove the Stoddard solvent and any fingerprints that may be present. Then rinse the bearing and slowly rotate it in fresh hot ($66 \pm 5^{\circ}\text{C}$) solvent rinse solution.

NOTE 62—Fresh solvent rinse solution must be used to avoid selective evaporation of the components at the rinse temperature.

9.4 Remove the bearing from the solvent rinse solution and place it on filter paper to drain. After draining, dry the bearing in an oven at $70 \pm 5^{\circ}\text{C}$ for 15 to 30 min.

9.5 Permit the bearing to cool to room temperature and reexamine its surfaces to ensure that corrosion-free and free-turning specimens have been selected. (Take care not to spin the bearings after cleaning and drying.)

9.6 Wash and dry the bearing packer using the same technique as for the preparation of the bearings.

10. Procedure

10.1 With the reservoir of the grease packer resting on a clean bench top, and while wearing gloves, place the bearing cup with the small diameter face down into the recess of the packer. Place the bearing cone over the cut, and while holding the bearing assembly against the packer, lift and invert the whole unit and return it to the bench.

10.2 Fill the reservoir with the grease sample, and use the plunger to force grease through the bearing. Carefully remove the plunger from the reservoir to avoid sucking air into the bearing, and slide the packer unit over the edge of the bench. While holding the bearing assembly in the packer, invert the unit to its original position on the bench.

10.3 Using a small square-ended spatula, remove excess grease from the bearing bore and the annulus between the grease packer and outer perimeter of the bearing cup. The bearing is removed from the packer by either the use of the pliers⁵ or by placing a gloved index finger in the bore and lifting it out.

10.3.1 While holding the bearing, use the spatula to remove excess grease above the cage on both sides of the bearing. This procedure is done to ensure that approximately the same volume of grease is used each time.

10.4 Using Fig. 1 as a guide, hold the packed bearing between gloved fingers with the large inside diameter of the cup downward and insert the small diameter plastic flange on top of the bore, and the larger flange into the bottom of the bore. Slide the bearing assembly onto the 1-kg weight so that the large diameter flange fits into the recess on top of the weight. Insert the bolt through the assembly and screw the bolt tightly into the weight. Lower the plastic bearing holder (Part 8) over the bearing. (The large O-ring faces upward.) Press down the holder so that the bearing fits squarely into the holder.

10.5 Invert a plastic jar over the bearing assembly. Slide the two components over the edge of the bench, and with fingers pressing the weight against the inner bottom of the jar, invert the entire assembly.

10.6 Place the jar onto the base of the motor-driven spindle and center under the indexing pin of the drive. After the pin is engaged, start the motor and bring the drive into the center of the 1-kg weight. Load until the nut hits the depth stop. Run for 60 ± 3 s and allow the bearing to coast to a stop. Hold it with two fingers and then raise the drive. Take extreme care not to break the contact between the races and rollers at this point and in the following steps.

NOTE 73—At no time during or after the 60-s run shall the grease be redistributed or forced back into the bearing.

10.7 Using synthetic sea water (**Warning**—see Note 4) 7.6) prepared as in Test Method D 665, make a dilution with distilled water (by volume, at the desired concentration) to obtain the proper test solution. The synthetic sea water should be no more than one week old.

10.8 Fill a clean syringe with 100 ± 5 mL of the freshly prepared synthetic sea water test solution. With the run-in bearing in the jar, simultaneously start a timer and begin adding the synthetic sea water solution into the hole provided for this purpose in the bearing holder. Add the 100 mL of the solution within 20 ± 3 s. When the timer shows 60 s, start withdrawing the solution. By 80 ± 3 s, complete the withdrawal of 70 ± 5 mL of the solution. Leave the remaining 25 to 30 mL of the solution in the jar. Make sure that water does not touch the bearing after 70 ± 5 mL is withdrawn. It may be difficult to withdraw 70 ± 5 mL of water in 10 seconds using a 16 gage needle. A larger needle may be required.

10.9 Screw the cap on the jar and transfer to a dark oven essentially free from vibration for 24 h at $52 \pm 1^\circ\text{C}$.

NOTE 84—Test Method D 1743 calls for 48 h at $52 \pm 1^\circ\text{C}$.

10.10 Prepare three bearings for each grease sample to be tested at the percentage of dilute synthetic sea water of interest. Each group of three bearings is one test. See Table 1 for conversion.

11. Rating Procedure

11.1 Remove the bearing from the test jar and place the bearing cup in a 50/50 mixture by volume of isopropyl alcohol (**Warning**—see Note 1 7.3 and Note 3) 7.5) and Stoddard solvent. The solvent mixture can be heated to facilitate the removal of the grease. Agitate vigorously to remove the grease. Repeat the rinsing using fresh solvent mixture or gently wipe the bearing with a clean cloth or tissue to ensure that traces of grease are removed.

11.2 Transfer the bearing cup from the solvent mixture and allow to dry on clean filter paper.

11.3 Examine the cup raceway for evidence of corrosion without the use of magnification (Section 5). Use only a pass or fail rating. Criteria for failure shall be the presence of any corrosion spot 1.0 mm or larger in the longest dimension. Ignore the number of spots.

NOTE 95—Spots that are easily removed by rubbing lightly with soft tissue (alone or wetted with any solvent nonreactive to rust or steel at room temperature) shall not be considered as corrosion in the rating.

TABLE 1 Conversions

in.	mm
$\frac{1}{32}$	0.794
$\frac{1}{8}$	3.175
$\frac{5}{32}$	3.969
$\frac{3}{16}$	4.763
$\frac{9}{32}$	7.144
$\frac{5}{16}$	7.938
$\frac{3}{8}$	9.525
$\frac{7}{16}$	11.113
$\frac{1}{2}$	12.700
$\frac{19}{32}$	15.081
$\frac{21}{32}$	16.669
$\frac{3}{4}$	19.050
1	25.400
$1\frac{1}{8}$	28.575
$1\frac{3}{16}$	31.262
$1\frac{1}{4}$	31.750
$1\frac{11}{32}$	34.131
1.495	37.973
1.500	38.100
$1\frac{9}{16}$	39.688
$1\frac{3}{4}$	44.450
1.785	45.339
$1\frac{15}{16}$	49.213
1.946	49.428
$2\frac{7}{32}$	56.356
$2\frac{1}{4}$	57.150
$2\frac{11}{32}$	59.531
3	76.200

12. Report

12.1 The reported result shall be the pass or fail rating as determined by at least two of the three bearings at the percentage of dilute synthetic sea water of interest.

13. Precision and Bias ⁸

13.1 Due to the nature of the results, the precision of this test method was not obtained in accordance with RR:D02-1007, “Manual on Determining Precision Data for ASTM Methods on Petroleum Products and Lubricants.”⁹

~~13.1 Due to the nature of the results, the precision of this test method was not obtained in accordance with RR:D02-007, “Manual on Determining Precision Data for ASTM Methods on Petroleum Products and Lubricants.”~~

13.2 *Precision*—The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

13.2.1 Repeatability may be judged by the fact that 92 % of duplicate results obtained by six laboratories at four concentrations of synthetic sea water (0 %, 5 %, 10 %, and 100 %) with eight samples were in agreement.

13.2.2 Reproducibility may be judged by the fact that six laboratories matched consensus 77 % of the time at four concentrations of synthetic sea water (0 %, 5 %, 10 %, and 100 %) with eight samples showing good or poor protection against corrosion.

13.3 *Bias*—No statement is made about the bias of this test method since the result merely states whether there is conformance to the criteria for success specified in the procedure.

14. Keywords

14.1 corrosion; lubricating grease; synthetic sea water

~~Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions~~

~~⁸ A research report on the testing development of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD. this test method is in preparation.~~

~~⁹ A research report on the development of this test method is in preparation.~~

~~⁹ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1007.~~

SUMMARY OF CHANGES

Committee D02.G0 has identified the location of selected changes to this standard since the last issue (D 5969–96) that may impact the use of this standard.

(1) Corrected the definition in 3.1.1.

(2) Added tolerances to Fig. 4.

(3) Clarified text in 10.8.

(4) Moved warnings from notes to section text throughout.

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