Designation: D 4049 - 99 (Reapproved 2004)

# Standard Test Method for Determining the Resistance of Lubricating Grease to Water Spray<sup>1</sup>

This standard is issued under the fixed designation D 4049; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

# 1. Scope

- 1.1 This test method<sup>2</sup> covers the ability of a grease to adhere to a metal surface when subjected to a water spray under prescribed laboratory conditions.
- 1.2 The values stated in SI units are to be regarded as the standard. The values given 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 ASTM Standards: <sup>3</sup>
- D 217 Test Methods for Cone Penetration of Lubricating Grease
- D 235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvent)
- D 1742 Test Method for Oil Separation from Lubricating Grease During Storage
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

#### 3. Terminology

- 3.1 Definitions:
- 3.1.1 *lubricating grease*, *n*—a semi-fluid to solid product of a thickener in a liquid lubricant.
- 3.1.1.1 *Discussion*—The dispersion of the thickener forms a two-phase system and immobilizes the liquid by surface tension and other physical forces. Other ingredients are commonly included to impart special properties.

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- 3.1.2 *oil separation*, *n*—the appearance of a liquid fraction from an otherwise homogeneous lubricating composition.
  - D 1742
- 3.1.3 *thickener*, *n*—*in lubricating grease*, a substance composed of finely divided particles dispersed in a liquid lubricant to form the product's structure.
- 3.1.3.1 *Discussion*—The thickeners can be fibers (such as various metallic soaps) or plates or spheres (such as certain non-soap thickeners), which are insoluble or, at most, only very slightly soluble in the liquid lubricant. The general requirements are that the solid particles be extremely small, uniformly dispersed, and capable of forming a relatively stable, gel-like structure with the liquid lubricant.

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# 4. Summary of Test Method

4.1 The grease to be tested is coated on a stainless steel panel and sprayed with water at the specified test temperature and pressure. The amount of grease remaining on the panel after  $5 \min \pm 15 \text{ s}$  is a measure of the resistance of the grease to water spray.

# 5. Significance and Use

5.1 This test method is used to evaluate the ability of a grease to adhere to a metal surface when subjected to direct water spray. The results obtained from the use of this test method suggest correlation in operations involving direct water spray impingement such as steel mill roll neck bearing service. This test method is used for quality control and purchase specifications.

# 6. Apparatus <sup>5</sup>

- 6.1 Stainless Steel Test Panel, 18-8 (chromium-nickel) stainless steel, as shown in Fig. 1.
- 6.1.1 *Grease Application Jig*, for applying grease to panel; Fig. 2.

 $<sup>^{\</sup>rm I}$  This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.G0 on Lubricating Grease.

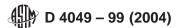
Current edition approved May 1, 2004. Published May 2004. Originally approved in 1981. Last previous edition approved in 1999 as D 4049 – 99.

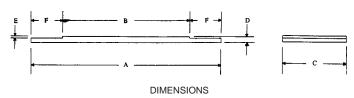
<sup>&</sup>lt;sup>2</sup> The development of this test method is described in Musilli, T. G.," Water Spray-Off Characteristics of Lubricating Grease," *NLGI Spokesman*, December 1982, pp. 323–326.

<sup>&</sup>lt;sup>3</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>&</sup>lt;sup>4</sup> A. E. Cichelli, "Evaluation of Greases for Steel Mill Service," *NLGI Spokesman*, August 1973, and A. E. Cichelli, "Grease Lubrication in Steel Mills with Emphasis on Roll Neck Bearings," *NLGI Spokesman*, April 1980.

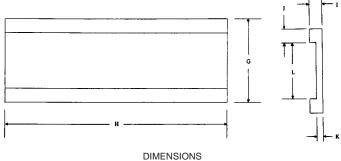
<sup>&</sup>lt;sup>5</sup> The sole source of supply of the apparatus known to the committee at this time is Koehler Instrument Co., Inc., 1595 Sycamore Ave., Bohemia, NY 11716. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee <sup>1</sup>, which you may attend.





	in.	mm
A- Overall panel length	6.0	152.4
B- Raised section length	4.0	101.6
C- Width	2.000 - 0.002	50.8 - 0.05
D- Thickness	$03/_{16} \pm 0.003$	$4.76 \pm 0.08$
E- Reduced section thickness	1/16	1.59
F- Reduced section length	1.0	25.4

Note 1—All tolerances  $\pm 0.03$  mm unless otherwise specified. FIG. 1 Stainless Steel Test Panel



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	in.	mm		
G- Width	3.0	76.2		
H- Length	8.0	203.2		
I- Thickness	7/16	11.11		
J- Raised section width	1/2	12.7		
K- Reduced section thickness	7/32	5.54		
L- Reduced section width	2.001 + 0.003	50.83 + 0.5		

Note 1—All tolerances ±0.03 mm unless otherwise specified. FIG. 2 Grease Application Jig

- 6.2 Water Spray Apparatus, as shown in Fig. 3.
- 6.3 Spray Nozzle, as shown in Fig. 4.
- 6.4 Thermometer or Thermocouple, capable of measuring  $38 \pm 0.5$ °C, to determine the temperature of the water spray, positioned to prevent breaking the spray pattern.

## 7. Reagents and Materials

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. Other grades may be used, provided 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 *Stoddard Solvent*, meeting requirements of Specification D 235. (Warning—Combustible. Vapor harmful.)
- 7.3 *n-Heptane*, reagent grade, minimum purity. (Warning—Flammable. Harmful if inhaled.)

### 8. Sampling

- 8.1 Each test will require a quantity sufficient to cover the steel panel (approximately 4-12 g).
  - 8.2 Supply a 25-g (minimum) representative sample.
- 8.3 Examine for any indication of nonhomogeneity, such as oil separation, phase changes, or gross contamination. If any abnormal conditions are found, obtain a new sample.

# 9. Preparation of Apparatus

- 9.1 Clean the test panel by brushing with Stoddard solvent. Then rinse with *n*-heptane and air dry (**Warning**—See 7.2 and 7.3)
- 9.2 Clean reservoir by flushing with water and wipe off any residual oil film from the surfaces of the reservoir and from the spray chamber area. Disassemble and clean the spray nozzle, taking care to properly position the vane in the cone body during assembly.
  - 9.2.1 Clean apparatus as in 9.2 after each test.

#### 10. Procedure

- 10.1 Weigh a clean test panel to the nearest 0.1 g, and record as  $W_1$ , and then, using the metal jig (as shown in Fig. 2), use a spatula to spread approximately a 0.8  $\pm$  0.005-mm ( $\frac{1}{32}$ -in.) thick film of grease. Clean off any grease beyond the raised section of the panel. Reweigh and record as  $W_2$ .
- 10.2 Add an adequate amount of tap water to the reservoir and to cover the heater and adjust the water temperature to 38  $\pm$  0.5°C (100  $\pm$  1°F). When the water temperature in the reservoir reaches 38  $\pm$  0.5°C (100 $\pm$  1°F), circulate 2 to 3 min to attain temperature equilibrium before spraying the panel. Adjust pump pressure to 276  $\pm$  7 kPa (40  $\pm$  1 psi) using the bypass valve. The bypass valve must be ahead of the gage, not between the gage and nozzle. Shut off the motor.
- 10.3 Insert the panel, making sure the panel is level and centered below the spray nozzle. Start the motor and spray the water on the panel for 5 min  $\pm$  15 s.
- 10.4 Shut off the motor to stop the spray, and remove the panel. Remove the excess grease outside the raised section of panel (area BC, Fig. 1) and along the sides and bottom of the panel. Place the panel in a horizontal position in an oven for 1 h  $\pm$  5 min at 66 $\pm$  1°C (150  $\pm$  2°F).
- 10.5 Remove the panel from the oven, and allow to cool. Reweigh the panel, and record as  $W_3$ .

# 11. Calculation

11.1 Calculate the percent spray-off as follows:

% spray-off = 
$$\left[\frac{(W_2 - W_3)}{(W_2 - W_1)}\right]$$
 (100)

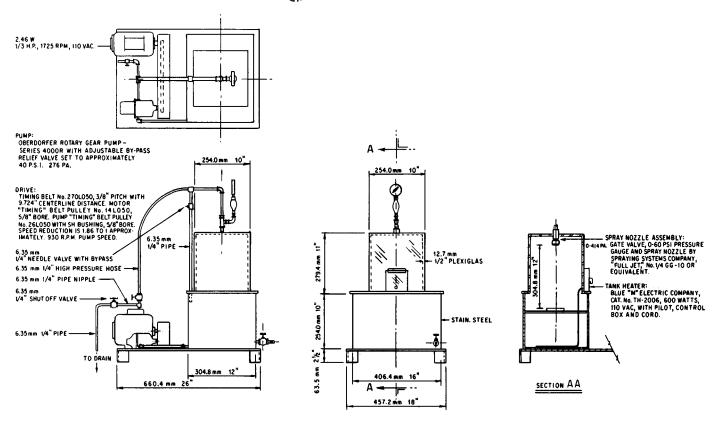
where:

 $W_1$  = initial mass of clean panel,

 $W_2$  = panel plus grease, before spraying, and

 $W_3$  = panel plus grease, after spraying.

<sup>&</sup>lt;sup>6</sup> 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 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



 ${\it Note}\,$  1—The bypass valve shall be ahead of the gage, not between the gage and nozzle.

# FIG. 3 Water Spray Apparatus

# 12. Report

12.1 Report the following: date, grease identity, and percent spray-off to the nearest  $1.0\,\%$ .

# 13. Precision and Bias <sup>7</sup>

- 13.1 The precision of this test method as determined by statistical examination of interlaboratory results in accordance with Practice E 691 is as follows:
- 13.1.1 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 value in only one case in twenty:

# 6.0% spray off

13.1.2 *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 value in only one case in twenty:

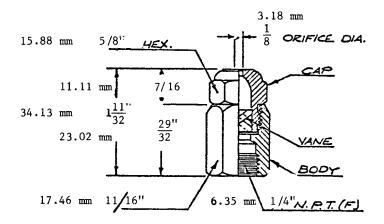
13.2 *Bias*—The procedure for measuring a grease's resistance to water spray has no bias because the value of spray-off can be defined only in terms of a test method.

#### 14. Keywords

14.1 grease; spray; water resistance; waterspray

<sup>&</sup>lt;sup>7</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1188.

# FULL CONE SPRAY PATTERN



TYPE OF LIQUID	CAPACITY G.P.M. AND SPRAY ANGLE AT PRESSURE psi					
	40	60	80	100	150	250
Water	1.9 65°	2.4 63°	2.7 61°	3.0 60°	3.6 58°	5.0 55°

kPa	psi	
276	40	
414	60	
552	80	
689	100	
1034	150	
1724	250	

FIG. 4 Full Cone Spray Tester

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