



## Standard Practice for Evaluating Compatibility of Binary Mixtures of Lubricating Greases<sup>1</sup>

This standard is issued under the fixed designation D 6185; 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 practice covers a protocol for evaluating the compatibility of one or three binary mixtures of lubricating greases by comparing their properties or performance relative to those of the neat greases comprising the mixture.

1.2 Three properties are evaluated in a primary testing protocol using standard test methods: (1) dropping point by Test Method D 566 (or Test Method D 2265); (2) shear stability by Test Methods D 217, 100 000-stroke worked penetration; and (3) storage stability at elevated-temperature by change in 60-stroke penetration (Test Method D 217). For compatible mixtures (those passing all primary testing), a secondary (nonmandatory) testing scheme is suggested when circumstances indicate the need for additional testing.

1.3 Sequential or concurrent testing is continued until the first failure. If any mixture fails any of the primary tests, the greases are incompatible. If all mixtures pass the three primary tests, the greases are considered compatible.

1.4 This practice applies only to lubricating greases having characteristics suitable for evaluation by the suggested test methods. If the scope of a specific test method limits testing to those greases within a specified range of properties, greases outside that range cannot be tested for compatibility by that test method. An exception to this would be when the tested property of the neat, constituent greases is within the specified range, but the tested property of a mixture is outside the range because of incompatibility.

1.5 This practice does not purport to cover all test methods that could be employed.

1.6 *This standard does not purport to address all the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- D 217 Test Methods for Cone Penetration of Lubricating Grease<sup>2</sup>
- D 566 Test Method for Dropping Point of Lubricating Grease<sup>2</sup>
- D 972 Test Method for Evaporation Loss of Lubricating Greases and Oils<sup>2</sup>
- D 1092 Test Method for Measuring Apparent Viscosity of Lubricating Greases<sup>2</sup>
- D 1263 Test Method for Leakage Tendencies of Automotive Wheel Bearing Greases<sup>2</sup>
- D 1264 Test Method for Determining Water Washout Characteristics of Lubricating Greases<sup>2</sup>
- D 1403 Test Method for Cone Penetration of Lubricating Grease Using One-Quarter and One-Half Scale Cone Equipment<sup>2</sup>
- D 1478 Test Method for Low-Temperature Torque of Ball Bearing Greases<sup>2</sup>
- D 1742 Test Method for Oil Separation from Lubricating Grease During Storage<sup>2</sup>
- D 1743 Test Method for Determining Corrosion Preventive Properties of Lubricating Greases<sup>2</sup>
- D 1831 Test Method for Roll Stability of Lubricating Grease<sup>2</sup>
- D 2265 Test Method for Dropping Point of Lubricating Grease Over Wide Temperature Range<sup>2</sup>
- D 2266 Test Method for Wear Preventive Characteristics of Lubricating Grease (Four-Ball Method)<sup>2</sup>
- D 2509 Test Method for Measurement of Load-Carrying Capacity of Lubricating Greases (Timken Method)<sup>2</sup>
- D 2595 Test Method for Evaporation Loss of Lubricating Greases over Wide Temperature Range<sup>2</sup>
- D 2596 Test Method for Measurement of Extreme-Pressure Properties of Lubricating Grease (Four-Ball Method)<sup>2</sup>
- D 3336 Test Method for Life of Lubricating Greases in Ball Bearings at Elevated Temperatures<sup>3</sup>
- D 3337 Test Method for Determining Life and Torque of Lubricating Greases in Small Ball Bearings<sup>3</sup>
- D 3527 Test Method for Life Performance of Automotive Wheel Bearing Grease<sup>3</sup>
- D 4049 Test Method for Determining the Resistance of Lubricating Grease to Water Spray<sup>3</sup>

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<sup>2</sup> Annual Book of ASTM Standards, Vol 05.01.

<sup>3</sup> Annual Book of ASTM Standards, Vol 05.02.

- D 4170 Test Method for Fretting Wear Protection by Lubricating Grease<sup>3</sup>
- D 4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants<sup>3</sup>
- D 4290 Test Method for Determining the Leakage Tendencies of Automotive Wheel Bearing Greases Under Accelerated Conditions<sup>3</sup>
- D 4425 Test Method for Oil Separation from Lubricating Grease by Centrifuging (Koppers Method)<sup>3</sup>
- D 4693 Test Method for Low-Temperature Torque of Grease-Lubricated Wheel Bearings<sup>4</sup>
- D 4950 Classification and Specification for Automotive Service Greases<sup>4</sup>
- D 5706 Test Method for Determining Extreme Pressure Properties of Lubricating Greases Using a High-Frequency Linear-Oscillation (SRV) Test Machine<sup>4</sup>
- D 5707 Test Method for Measuring Friction and Wear Properties of Lubricating Grease Using a High-Frequency, Linear-Oscillation (SRV) Test Machine<sup>4</sup>

## 2.2 Federal Standard:

Federal Test Method Standard 791C, Method 3467.1, Storage Stability of Lubricating Grease<sup>5</sup>

## 3. Terminology

### 3.1 Definitions:

3.1.1 *bleed (bleeding)*, *n*—of lubricating greases, the separation of a liquid lubricant from a lubricating grease for any cause.

3.1.2 *lubricant*, *n*—any material interposed between two surfaces that reduces the friction or wear between them.

3.1.3 *lubricating grease*, *n*—a semifluid to solid product of a dispersion of a thickener in a liquid lubricant.

3.1.3.1 *Discussion*—The dispersion of the thickener forms a two-phase system and immobilizes the liquid lubricant by surface tension and other physical forces. Other ingredients imparting special properties are often included. **D 4175**

3.1.4 *spatulate*, *v*—to mix or blend by spreading and folding with a flat thin, usually metal, tool.

3.1.5 *syneresis*, *n*—of lubricating greases, the separation of liquid lubricant from a lubricating grease due to shrinkage or rearrangement of the structure.

3.1.5.1 *Discussion*—Syneresis is a form of bleeding caused by physical or chemical changes of the thickness. Separation of free oil or the formation of cracks that occur in lubricating greases during storage in containers is most often due to syneresis.

3.1.6 *thickener*, *n*—in a lubricating grease, a substance composed of finely divided particles dispersed in a liquid lubricant to form the product's structure.

3.1.6.1 *Discussion*—The thickener can be fibers (such as various metallic soaps) or plates or spheres (such as certain non-stop thickeners) which are insoluble or, at most, only very slightly soluble in the liquid lubricant. The general require-

ments are that the solid particles be extremely small, uniformly dispersed, and capable of forming a relatively stable, gel-like structure with the liquid lubricant. **D 217**

### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *compatibility*, *n*—of lubricating greases, the characteristic of lubricating greases to be mixed together without significant degradation of properties or performance.

3.2.1.1 *Discussion*—When a mixture of two greases has properties or performance significantly inferior to both of the neat, constituent greases, then the two greases are incompatible. If the properties are inferior to those of one neat grease but not inferior to those of the other, then such is not necessarily considered an indication of incompatibility. To be considered significantly inferior, the property of the mixture would be worse than the poorer of the two neat greases by an amount exceeding the repeatability of the test method used to evaluate the property (see *pass* and *fail*). Incompatibility most often is manifested by a degradation in physical properties rather than in chemical properties, although, occurrence of the latter is not unknown.

3.2.2 *borderline compatibility*, *n*—of lubricating greases, the characteristic of lubricating greases to be mixed together with only slight degradation of properties or performance.

3.2.2.1 *Discussion*—*Slight degradation* means that the properties or performance of the mixture is poorer than those of the two neat greases but by an amount less than the repeatability of the test method used to evaluate the property. (See *borderline pass*).

3.2.3 *primary compatibility tests*, *n*—of lubricating greases, those test methods employed first to evaluate compatibility

3.2.3.1 *Discussion*—The test methods considered the most significant in the evaluation of grease compatibility, insofar as they provide the most information with the least expenditure of testing resources, include tests for dropping point, consistency (usually softening) after shearing conditions, and consistency change after storage at elevated temperatures.

3.2.4 *secondary compatibility tests*, *n*—of lubricating greases, those test methods used to evaluate compatibility when the primary compatibility tests are insufficient or inconclusive.

3.2.4.1 *Discussion*—Such tests are driven by the critical features of a given application. For example, if the application subjects the grease to water contamination, water washout or water spray-off tests and, perhaps, corrosion tests would be used for additional evaluation. Secondary compatibility tests are suggested, but not required, by this practice.

3.2.5 *pass*, *n*—in compatibility testing of grease mixtures, a test result that is equal to or better than that of the poorer of the two constituent greases.

3.2.6 *borderline pass*, *n*—in compatibility testing of grease mixtures, a test result that is inferior to that of the poorer of the two constituent greases by an amount not exceeding the repeatability of the test method used for the evaluation.

3.2.6.1 *Discussion*—*Borderline pass*, *borderline fail*, *borderline compatible*, and *borderline incompatible* are synonymous terms.

3.2.7 *fail*, *n*—in compatibility testing of grease mixtures, a test result that is inferior to that of the poorer of the two

<sup>4</sup> Annual Book of ASTM Standards, Vol 05.03.

<sup>5</sup> Available from Standardization Documents Order Desk, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

constituent greases by an amount exceeding the repeatability of the test method used for the evaluation.

3.2.8 *50:50 mixture, n*—a uniform blend of 50 mass % of each of two component greases.

3.2.9 *10:90 mixture, n*—a uniform blend of 10 mass % of one grease with 90 mass % of a second grease.

3.2.10 *90:10 mixture, n*—a uniform blend of 90 mass % of one grease with 10 mass % of a second grease.

#### 4. Summary of Practice

4.1 *Option 1*—A 50:50 mixture of two greases to be evaluated for compatibility is prepared by spatulating. This mixture and the two neat, constituent greases are tested using the primary compatibility tests (dropping point, 100 000-stroke worked penetration, and change in 60-stroke penetration due to high-temperature storage). Depending on the performance of the mixture, relative to those of the constituent greases, 10:90 and 90:10 mixtures may need to be tested in addition. Alternatively, Option 2 can be used. Instead of testing mixtures in sequential order, 10:90 and 90:10 mixtures are tested at the same time the 50:50 mixture is evaluated. If all mixtures pass the primary compatibility tests, or if the application requires the evaluation of specific properties, secondary compatibility tests can be employed for further evaluation. Such tests can be run concurrently, if desired.

#### 5. Significance and Use

5.1 The compatibility of greases can be important for users of grease-lubricated equipment. It is well known that the mixing of two greases can produce a substance markedly inferior to either of its constituent materials. One or more of the following can occur. A mixture of incompatible greases most often softens, sometimes excessively. Occasionally, it can harden. In extreme cases, the thickener and liquid lubricant will completely separate. Bleeding can be so severe that the mixed grease will run out of an operating bearing. Excessive syneresis can occur, forming pools of liquid lubricant separated from the grease. Dropping points can be reduced to the extent that grease or separated oil runs out of bearings at elevated operating temperatures. Such events can lead to catastrophic lubrication failures.

5.1.1 Because of such occurrences, equipment manufacturers recommend completely cleaning the grease from equipment before installing a different grease. Service recommendations for grease-lubricated equipment frequently specify the caveat—*do not mix greases under any circumstances*. Despite this admonition, grease mixing will occur and, at times, cannot be avoided. In such instances, it would be useful to know whether the mixing of two greases could lead to inadequate lubrication with disastrous consequences. Equipment users most often do not have the resources to evaluate grease compatibility and must rely on their suppliers. Mixing of greases is a highly imprudent practice. Grease and equipment manufacturers alike recognize such practices will occur despite all warnings to the contrary. Thus, both users and suppliers have a need to know the compatibility characteristics of the greases in question.

5.2 There are two approaches to evaluating the compatibility of grease mixtures. One is to determine whether such mixtures meet the same specification requirements as the

constituent components. This approach is not addressed by this practice. Instead, this practice takes a specification-independent approach; it describes the evaluation of compatibility on a relative basis using specific test methods.

5.2.1 Three test methods are used because fewer are not sufficiently definitive. For example, in one study, using 100 000-stroke worked penetration for evaluation, 62 % of the mixtures were judged to be compatible.<sup>6</sup> In a high-temperature storage stability study, covering a broader spectrum of grease types, only one-third of the mixtures were compatible.<sup>6</sup> These studies used different criteria to judge compatibility.

5.2.2 Compatibility cannot be predicted with certainty from foreknowledge of grease composition. Generally, greases having the same or similar thickener types will be compatible. Uncommonly, even greases of the same type, although normally compatible when mixed, can be incompatible because of incompatible additive treatments. Thus, compatibility needs to be judged on a case-by-case basis.

5.3 Two constituent greases are blended in specific ratios. A 50:50 mixture simulates a ratio that might be experienced when one grease (Grease A) is installed in a bearing containing a previously installed, different grease (Grease B), and no attempt is made to flush out Grease B with Grease A. The 10:90 and 90:10 ratios are intended to simulate ratios that might occur when attempts are made to flush out Grease B with Grease A.

NOTE 1—Some companies evaluate 25:75 and 75:25 ratio mixtures instead of 10:90 and 90:10 ratio mixtures. But, the latter two ratios, which are prescribed by this practice, are considered more representative of the flushing practice described in 5.3.

5.3.1 Incompatibility is most often revealed by the evaluation of 50:50 mixtures. However, in some instances 50:50 mixtures are compatible and more dilute ratios are incompatible. (See Appendix X1 and Meade.)<sup>7</sup>

5.4 Compatibility information can be used in product information literature supplied with specific greases. It can be used also in literature describing lubrication practices and equipment maintenance.

#### 6. Apparatus

6.1 The equipment and materials required for this practice shall be those required by the test methods used to evaluate compatibility. At the least, this will include those required by the primary compatibility Test Methods D 217 and D 566 (or D 2265) and a laboratory oven.

6.1.1 *Dropping Point Equipment*, grease cup, test tube, thermometer, temperature bath, and accessories as described in Test Method D 566, or dropping point assembly, aluminum block oven, and peripheral equipment as described in Test Method D 2265.

6.1.2 *Penetration Equipment*, penetrometer with standard cone, grease worker with motorized drive, temperature bath, and peripheral equipment as described in Test Methods D 217.

6.1.3 *High-Temperature Storage Test Equipment*:

<sup>6</sup> Myers, E. H., "Incompatibility of Greases," *NLGI Spokesman*, April 1983, p 24.

<sup>7</sup> Meade, F. S., "Compatibility of Greases," Rock Island Arsenal Laboratory Report, No. 56-2405, PB 121731, Aug. 20, 1956.



6.1.3.1 *Laboratory Oven*, static-air or stirred-air type, capable of maintaining the test temperature within  $\pm 3^{\circ}\text{C}$  and equipped with one or more grill-type, wire shelves.

6.1.3.2 *Penetration Equipment*, full-scale, as described in 6.1.2; or 1/2-scale penetrometer, cone, and worker, as described in Test Method D 1403.

## 7. Procedure

7.1 Either of two similar testing options can be used—the sequential testing protocol described in Option 1 (A1.1), or the concurrent testing protocol described in Option 2 (A1.2). Using Option 1, a 50:50 mixture and the two constituent greases are tested using three primary tests. If this mixture is found compatible, 10:90 and 90:10 mixtures are tested. Using Option 2, all mixtures (10:90, 50:50, and 90:10) and the two constituent greases are tested concurrently.

7.1.1 With either option, the test procedures (that is, dropping point, shear stability, or storage stability tests) can be run concurrently or sequentially in any order. The test procedures are presented in the order of the time required to run an individual test.

7.2 *Preparation of Mixtures*—Regardless of whether one or three mixtures of differing ratios are to be tested sequentially or concurrently, they shall be prepared in similar fashion.

7.2.1 Prepare a fresh 50:50 mixture of the two greases to be evaluated for compatibility. (For convenience, the neat, constituent greases are called A and B.) The amounts to be mixed shall be determined from the amount of grease required by the tests. Prepare at least 10 % more mixture than is actually needed for the tests. Do not prepare more than can be used immediately, that is no more than about 4 h should elapse between mixture preparation and the start of any test.

7.2.2 Weigh equal amounts  $\pm 1\%$  of each neat grease and transfer to a hard, flat, impervious surface such as a glass or stainless steel plate. About a 400 mm-square surface is convenient.

7.2.3 With a suitable spatula, fold and blend the two greases into each other until a uniform blend is produced.

**NOTE 2—Caution:** Great care should be taken to minimize the occlusion of air.

**NOTE 3—Warning:** Do not use any other mixing equipment to prepare grease mixtures.

7.2.4 When 10:90 and 90:10 mixtures are to be tested, prepare fresh mixtures of these proportions in the same fashion. The weighing tolerance shall be  $\pm 1\%$  of each neat grease.

7.3 *Option 1* (see A1.1)—Use the primary test protocol to test the Constituent Greases A and B and the 50:50 mixture. If resources permit, the dropping points, shear stability, and storage stability tests can be run concurrently. Otherwise, any sequence of these tests can be used.

7.3.1 *Dropping Point*—Determine and record the dropping points as described in Test Method D 566 or D 2265.

7.3.1.1 Compare dropping point of the mixture with those of constituent greases. If the dropping point of the mixture is equal to or greater than that of either constituent grease, record as *compatible* or *pass*. If the dropping point of the mixture is less than the lower of the constituent greases by an amount equal to or less than repeatability of the test method (see 9.1.1

for repeatability values), record as *borderline compatible* or *borderline pass*. If the dropping point of the mixture is less than the lower of the constituent greases by an amount greater than repeatability of the test method, record as *incompatible* or *fail*.

7.3.1.2 If the mixture is incompatible (fail), no further testing need be done. If the mixture is compatible or borderline compatible (pass or borderline pass), further tests are required. If not yet run, test the mixture and the neat greases for shear stability (100 000-stroke worked penetration) or high temperature storage stability, or both (7.3.2 and 7.3.3, respectively).

7.3.2 *Shear Stability*—Determine the 10 000-stroke worked penetrations as described in Test Methods D 217.

7.3.2.1 Compare the 10 000-stroke worked penetration of the mixture with those of constituent greases. If the penetration of the mixture is equal to or between those of the constituent greases, record as *compatible* or *pass*. If the penetration of the mixture is less than the lower of the constituent grease or greater than the higher of the constituent grease by an amount equal to or less than repeatability of the test method (see 9.1.2), record as a *borderline compatible* or *borderline pass*. If the penetration of the mixture is less than the lower of the constituent grease or greater than the higher of the constituent grease by an amount greater than repeatability of the test method (see 9.1.2), record as *incompatible* or *fail*.

7.3.2.2 If the mixture is incompatible (fail), no further testing need be done. If the mixture is compatible or borderline compatible (pass or borderline pass), further tests are required. If not yet run, test the mixture and the neat greases for dropping or high temperature storage stability, or both (7.3.1 and 7.3.3, respectively).

7.3.3 *High-Temperature Storage Stability*—The following variation of Federal Test Method (FTM) 3467.1 shall be followed. Determine the 60-stroke penetrations of the neat, constituent greases and the blends. Test Methods D 217 is preferred, but 1/2-scale equipment (Test Method D 1403) may be substituted. One-quarter-scale equipment is considered generally unsuitable. Record the test method (Test Methods D 217 or Test Method D 1403) used.

**NOTE 4**—Variations from FTM-3467.1 include oven requirements, sample containers, allowance of 1/2-scale measurements, and specified storage conditions.

**NOTE 5**—According to Test Method D 1403, 1/2-scale measurements are converted to full-scale values; the appropriate repeatability is applied as described.

7.3.3.1 Place test samples in worker cups or suitably sized glass or steel containers; loosely cap or cover with aluminum foil. Place containers on the wire shelf located about midway to upper third of the laboratory oven preheated to  $120 \pm 3^{\circ}\text{C}$ . Maintain  $120 \pm 3^{\circ}\text{C}$  for  $70 \pm \frac{1}{4}$  h.

7.3.3.2 Greases having low dropping points (less than  $125^{\circ}\text{C}$ ); for example, hydrated calcium greases, cannot be tested under these conditions. When testing compatibility of such greases, the storage stability test shall be run at  $75 \pm 3^{\circ}\text{C}$  for  $1400 \pm 4$  h.

7.3.3.3 Remove samples from the oven and allow to cool. Determine 60-stroke worked penetration in accordance with Test Methods D 217 (or Test Method D 1403). Calculate