Designation: D566 – 17

British Standard 2877

Standard Test Method for Dropping Point of Lubricating Grease[1]

This standard is issued under the fixed designation D566; 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 dropping point of lubricating grease.

1.2 This test method is not recommended for use at bath temperatures above 288 °C. For higher temperatures Test Method D2265 should be used.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 **WARNING**—This test method uses mercury-filled thermometers. Mercury has been designated by many regulatory agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Safety Data Sheet (SDS) for details and EPA's website— http://www.epa.gov/mercury/faq.htm—for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law. The responsible subcommittee, D02.G.3, continues to explore alternatives to eventually replace the mercury thermometers.

1.5 *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.* For specific hazard statements, see 6.4 and 8.1.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D217 Test Methods for Cone Penetration of Lubricating Grease

D235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvent)

D2265 Test Method for Dropping Point of Lubricating Grease Over Wide Temperature Range

E1 Specification for ASTM Liquid-in-Glass Thermometers

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 lubricant by surface tension and other physical forces. Other ingredients are commonly included to impart special properties. **D217**

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

3.1.2.1 *Discussion*—Thickeners can be fibers (such as various metallic soaps) or plates or spheres (such as certain non-soaps thickeners), which are insoluble or, at most, only very slightly soluble in the liquid lubricant. The general requirements are that the solid particles are extremely small, uniformly dispersed and capable of forming a relatively stable, gel-like structure with the liquid lubricant. **D217**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *dropping point, n*—a numerical value assigned to a grease composition representing the temperature at which the first drop of material falls from the test cup; that temperature being the average of the thermometer readings of the sample and bath.

[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.G0.03 on Physical Tests.

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In the IP, this test method is under the jurisdiction of the Standardization Committee. This test method was adopted as a joint ASTM-IP standard in 1964.

This test method has been adopted for use by government agencies to replace Method 1421 of Federal Test Method Standard No. 791b.

[2] 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.

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

3.2.1.1 *Discussion*—In the normal and proper operation of this test method the temperature of the interior of the grease test cup and the temperature of the oil bath are monitored simultaneously as the bath is heated. When the first drop of material falls from the cup, the temperature of the grease test cup and the bath temperature are averaged and recorded as the result of the test.

4. Summary of Test Method

4.1 A sample of lubricating grease contained in a cup suspended in a test tube is heated in an oil bath at a prescribed rate. The temperature at which material falls from the hole in the bottom of the cup is averaged with the temperature of the oil bath and recorded as the dropping point of the grease.

5. Significance and Use

5.1 In general, the dropping point is the temperature at which the grease passes from a semisolid to a liquid state under the conditions of test. This change in state is typical of greases containing as thickeners soaps of conventional types. Greases containing as thickeners materials other than conventional soaps can, without change in state, separate oil. This test method is useful to assist in identifying the grease as to type and for establishing and maintaining bench marks for quality control. The results are considered to have only limited significance with respect to service performance as dropping point is a static test.

NOTE 1—Cooperative testing indicates that in general, dropping points by Test Method D566[3] and Test Method D2265 are in agreement. In cases where results differ, there is no known significance. However, agreement between producer and consumer as to the test method used is advisable.

6. Apparatus

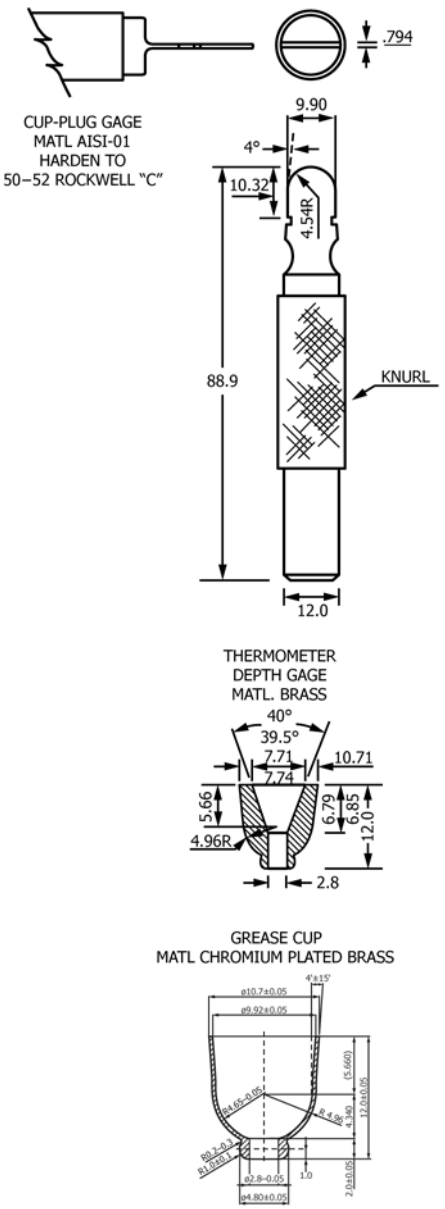
6.1 *Grease Cup*—A chromium-plated brass cup conforming to the dimensions shown in Fig. 1.

6.2 *Test Tube*—A test tube of heat-resistant glass,[4] with rim, 100 mm to 103 mm in length and 11.1 mm to 12.7 mm in inside diameter provided with three indentations about 19 mm from the bottom, equally spaced on the circumference. The depth of these indentations shall be such as to support the grease cup at about the point shown in Fig. 2.

6.3 *Thermometers,* two, having ranges as shown below and conforming to the requirements prescribed in Specification E1:

	Thermometer Number	
Temperature Range	ASTM	IP
−5 °C to + 300 °C	2C	62C

6.4 *Accessories*—A stirred oil bath consisting of a 400 mL beaker, a ring stand and ring for support of the oil bath, clamps for thermometers, two corks as illustrated in Fig. 2, a polished metal rod 1.2 mm to 1.6 mm in diameter and 150 mm to 152 mm in length (Fig. 3), a cup plug gauge and thermometer depth gauge, both shown in Fig. 1. (**Warning—**The fluid for the oil bath must have a flash point in excess of the maximum



NOTE 1—Dimensions in millimetres.

FIG. 1 Plug Gauge, Depth Gauge, and Grease Cup

temperature at which the bath is to be used[5] and allowance must be made for thermal expansion to prevent overflow. Heating is preferably done by an immersed electrical-resistance heater regulated by voltage control. An open flame must not be used as the heating source.) (**Warning—**When a hot plate is used, care must be taken to avoid spilling oil on the hot surface.)

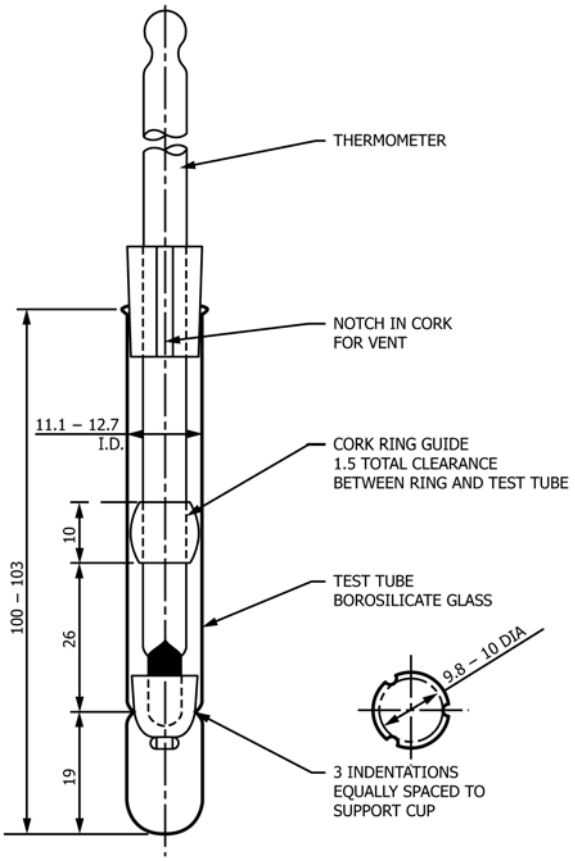
7. Sampling

7.1 When ready to test, examine the sample for any indication of non-homogeneity such as oil separation, phase changes,

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report: RR:D02-1164. Contact ASTM Customer Service at service@astm.org.

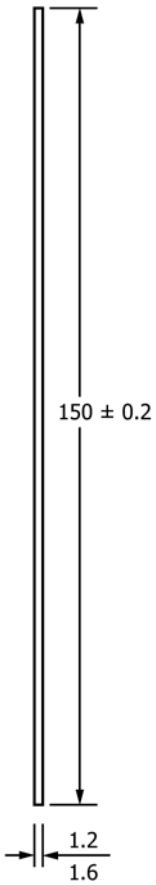
[4] Borosilicate glass has been found satisfactory for this purpose.

[5] Dow Corning 710 Fluid has been found satisfactory when bath temperatures as high as 288 °C are required. Dow Corning is a registered trademark of the Dow Corning Corp., Midland, MI 48686.



NOTE 1—Dimensions in millimetres.

FIG. 2 Assembled Apparatus



NOTE 1—Dimensions in millimetres.

FIG. 3 Polished Metal Rod

or gross contamination. When any abnormal conditions are noted, obtain a new sample.

8. Preparation of Apparatus

8.1 Thoroughly clean the cup and test tube with mineral spirits conforming to Specification D235. (**Warning—** Flammable. Vapor harmful.)

8.2 Use only cups that are clean and free from any residue from previous runs. When the interior plating of the cup shows indications of wear, discard.

8.3 When new cups are to be used, check their dimensions by using the cup plug gauge (Fig. 1). To check the bottom opening a 2.72 mm rod should fit easily while a 2.82 mm rod should not. See Note 2. If the hole is undersized, ream to correct size. If too large, discard.

NOTE 2—These are commonly available as a 7⁄64-in. drill and a No. 34 drill, respectively.

8.4 Test tube shall be clean and free of residues. Inspect the tube for evidence of chipping or cracking, particularly the points of indentation. Replace when necessary.

8.5 Inspect both cork ring guide and upper cork for charring or distortions in shape. Total clearance between the cork ring guide and the inside wall of the test tube is 1.5 mm. When either cork is abnormal, replace.

8.6 Inspect the bulb end of the thermometer to be used in the test tube. Clean if there is any residue build-up.

9. Procedure

9.1 Fill a test cup with sample by pressing the larger opening into the grease to be tested until the cup is filled. Remove excess grease with a spatula. Gently press the cup, held in a vertical position with the smaller opening at the bottom, down over the metal rod until the latter protrudes about 25 mm. Press the rod against the cup in such a manner that the rod makes contact at both upper and lower peripheries of the cup. Maintain this contact, rotating the cup on the rod along the index finger to give a spiral-like motion down the rod to remove a conical section of the grease which adheres along the rod. As the cup approaches the end of the rod, carefully slip the rod out of the cup so that a smooth film, free of air bubbles and of reproducible thickness, remains inside the cup. See Fig. 4.

9.2 Place the corks on the thermometer to be used in the test tube as shown in Fig. 2. With the thermometer depth gauge in position in the test tube, adjust the position of the upper cork on the thermometer so that the thermometer bulb bottoms snugly in the depth gauge. Observe the relative position of the top edge of the upper cork to the thermometer stem as well as the relative position of the top edge of the test tube to the cork. Care must be taken to be certain that the thermometer is

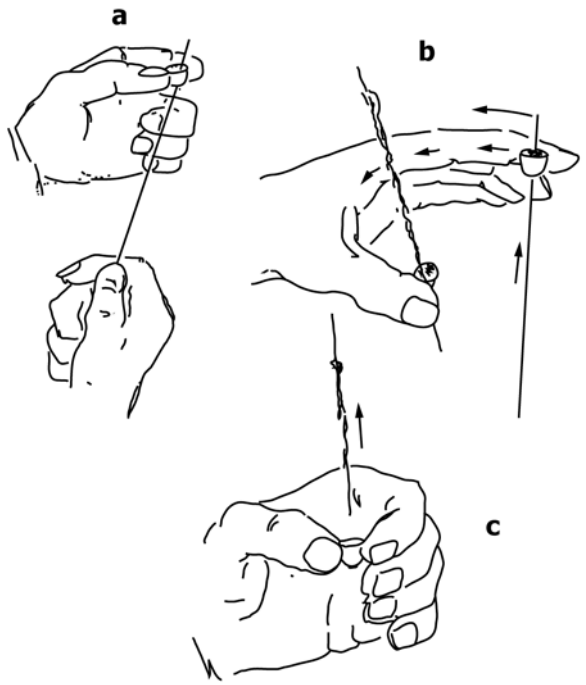


FIG. 4 Procedure to Fill Cup

inserted to the same depth when the apparatus is reassembled with the grease cup in position.

9.3 Replace the depth gauge with the grease cup as shown in Fig. 2 so that the thermometer is inserted to the previously gauged depth. When properly inserted, the bulb of the thermometer does not touch either the grease sample or the cup.

9.4 Suspend the test tube in the oil bath to a depth corresponding to the 76 mm immersion mark on the thermometer. This should leave the test tube rim at least 6 mm above the oil level.

9.5 Suspend the second thermometer in the oil bath so that its bulb is at approximately the same level as the bulb of the test tube thermometer.

9.6 Stir the oil bath and heat at a rate of 4 °C⁄min to 7 °C⁄min until the bath reaches a temperature of approximately 17 °C below the expected dropping point of the grease. At this point reduce the rate of heating so that the temperature difference between the test tube and the oil bath is maintained between 1 °C and 2 °C. This condition is established when the oil bath is heated at a rate of about 1 °C⁄min to 1.5 °C⁄min. As the temperature increases, material will gradually protrude through the orifice of the grease cup. When a drop of material falls, note the temperatures on the two thermometers and record their average to the nearest degree as the dropping point of the grease.

NOTE 3—Certain greases form a drop with a tailing thread upon melting, which can break off or which can hold until the drop reaches the bottom of the test tube; in any case, the observed dropping point is the temperature when the drop reaches the bottom of the test tube.

NOTE 4—The dropping points of some greases, particularly those containing simple aluminum soaps, are known to decrease upon aging, the change being much greater than the deviation permitted in results obtained by different laboratories. Therefore, comparative tests between laboratories should be made within a period of six days.

9.7 Two determinations can be made simultaneously in the same bath, provided both samples have approximately the same dropping points.

10. Report

10.1 Report the result to the nearest degree as the ASTM-IP dropping point, ASTM D566.

11. Precision and Bias[6]

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

11.1.1 *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 only in one case in twenty:

$$\text{Repeatability} = 7\ ^\circ\text{C} \quad (1)$$

11.1.2 *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:

$$\text{Reproducibility} = 13\ ^\circ\text{C} \quad (2)$$

NOTE 5—Although the cooperative data does show that precision varied considerably with dropping point range, a statistical analysis of the

[6] There is no research report on file because this test method was developed prior to the development of Research Report guidelines. The statistical method used to determine precision is unknown.

composite of the cooperative data was used to generate the precision statement. This test data is given in the article "Report of ASTM Technical Committee G on Dropping Point Methods for Lubricating Grease" by P. R. McCarthy appearing in the NLGI Spokesman, Vol 31, 1967, page 76.

11.1.3 *Bias*—There is no bias in this test method because the value of the dropping point can be defined only in terms of the test method.

12. Keywords

12.1 dropping point; grease; lubricating grease

SUMMARY OF CHANGES

Subcommittee D02.G0 has identified the location of selected changes to this standard since the last issue (D566 – 16) that may impact the use of this standard. (Approved May 1, 2017.)

(1) Revised Fig. 1.

Subcommittee D02.G0 has identified the location of selected changes to this standard since the last issue (D566 – 02 (2009)) that may impact the use of this standard. (Approved April 1, 2016.)

(1) Subsection 1.4 was added.