Designation: D 566 – 02

An American National Standard
British Standard 2877

Designation: 132/96

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

This standard is issued under the fixed designation D 566; 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 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.

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 D 2265 should be used.

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

2. Referenced Documents

2.1 *ASTM Standards:*

D 217 Test Methods for Cone Penetration of Lubricating Grease[2]

D 235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvents)[3]

D 2265 Test Method for Dropping Point of Lubricating Grease Over Wide Temperature Range[3]

E 1 Specification for ASTM Thermometers[4]

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 thickner 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. **D 217**

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. **D 217**

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.

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.

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.G on Lubricating Grease.

In the IP, this test method is under the jurisdiction of the Standardization Committee.

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[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 06.04.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

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 D 566[5] and Test Method D 2265 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,[6] with rim, 100 to 103 mm in length and 11.1 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.

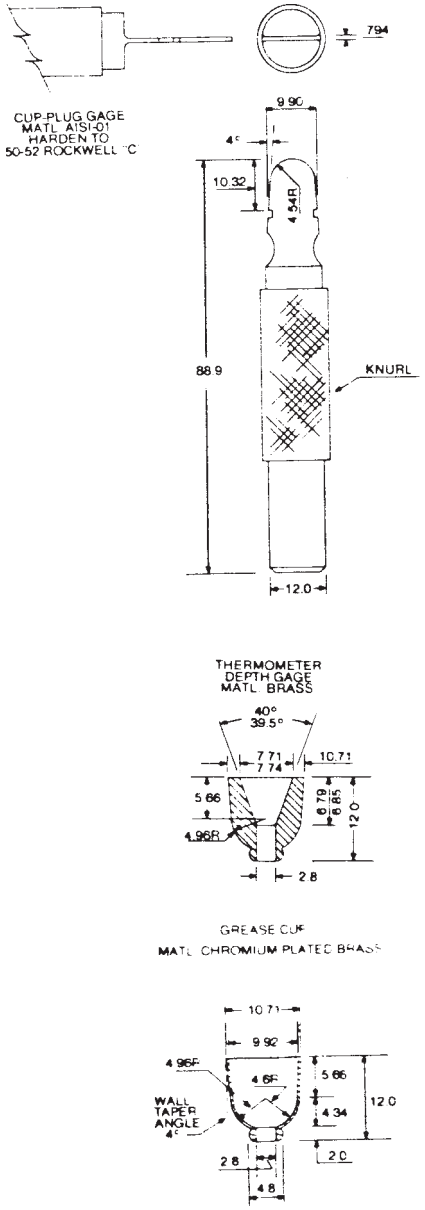
6.3 *Thermometers*, two, having ranges as shown below and conforming to the requirements prescribed in Specification E 1:

	Thermometer Number	
Temperature Range	ASTM	IP
−5 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 to 1.6 mm in diameter and 150 to 152 mm in length (Fig. 3), a cup plug gage and thermometer depth gage, both shown in Fig. 1. (**Warning**—The fluid for the oil bath must have a flash point in excess of the maximum temperature at which the bath is to be used[7] 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, or gross contamination. When any abnormal conditions are noted, obtain a new sample.



NOTE 1—Dimensions in millimetres.

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

8. Preparation of Apparatus

8.1 Thoroughly clean the cup and test tube with mineral spirits conforming to Specification D 235. (**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.

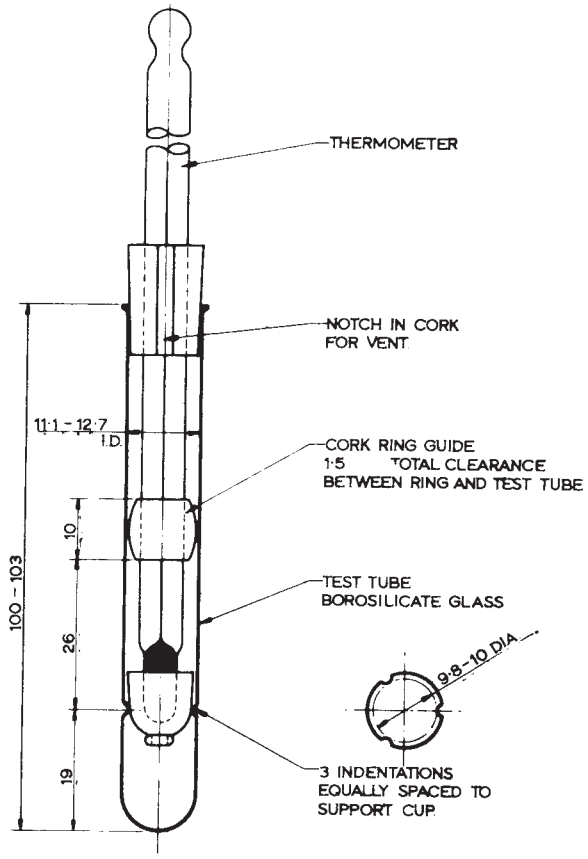
8.3 When new cups are to be used, check their dimensions by using the cup plug gage (Fig. 1). To check the bottom opening a 2.72-mm rod should fit easily while a 2.82-mm rod should not.[8] If the hole is undersized, ream to correct size. If too large, discard.

[5] Cooperative data are available from ASTM International by requesting RR:D02-1164.

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

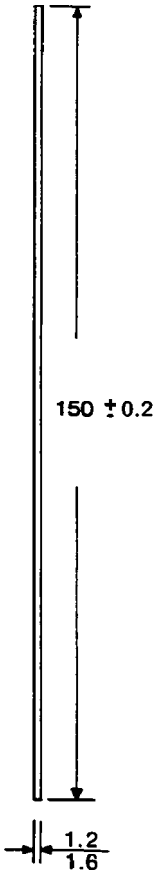
[7] Dow Corning 710 Fluid has been found satisfactory when bath temperatures as high as 288°C are required. Dow Corning is a registered trademark of the Dow Corning Corp., Midland, MI 48686.

[8] These are commonly available as a 7⁄64-in. drill and a No. 34 drill, respectively.



NOTE 1—Dimensions in millimetres.

FIG. 2 Assembled Apparatus



NOTE 1—Dimensions in millimetres.

FIG. 3 Polished Metal Rod

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

9.3 Replace the depth gage with the grease cup as shown in Fig. 2 so that the thermometer is inserted to the previously gaged 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 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 and 2°C. This condition is established when the oil bath is heated at a rate of about 1 to 1.5°C/min. As the temperature increases, material will gradually protrude through the orifice of the

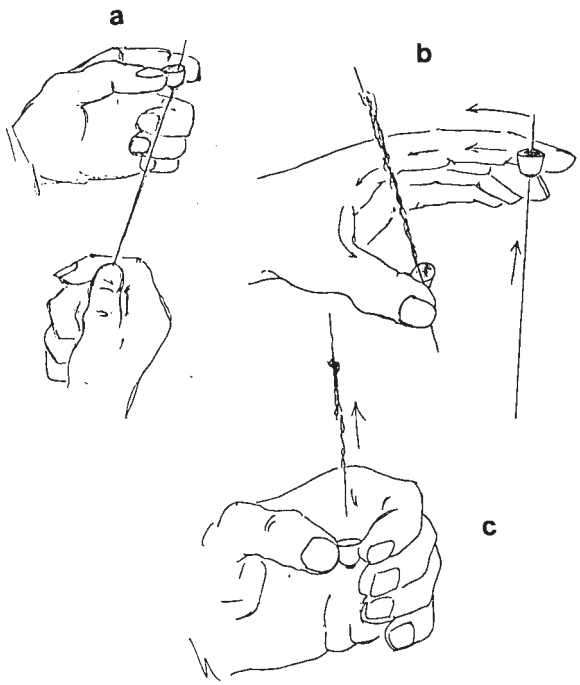


FIG. 4 Procedure to Fill Cup

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 2—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 3—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 [9]

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:

$$\text{Repeatability} = 7°C \tag{1}$$

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:

$$\text{Reproducibility} = 13°C \tag{2}$$

NOTE 4—Although the cooperative data does show that precision varied considerably with dropping point range, a statistical analysis of the 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

[9] 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.