



Designation: D6184 – 22

# Standard Test Method for Oil Separation from Lubricating Grease (Conical Sieve Method)<sup>1</sup>

This standard is issued under the fixed designation D6184; 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.

*This standard has been approved for use by agencies of the U.S. Department of Defense.*

## INTRODUCTION

The conical sieve method for measuring leakage from lubricating grease (commonly known as the cone bleed test) uses the same equipment as the obsolete, withdrawn FED-STD-791C (Federal Test Method (FTM) 791C) Method 321.3, Oil Separation from Lubricating Grease (Static Technique). Despite the lack of a precision statement, Method 321.3 was used for many years in specifications to evaluate the tendencies of base fluid separation from military and industrial greases. Test Method D6184 supersedes the FTM method by overcoming deficiencies of Method 321.3. It includes the procedures and precision determinations resultant from a cooperative study conducted by participating members of Subcommittee D02.G0.

## 1. Scope\*

1.1 This test method covers the determination of the tendency of lubricating grease to separate oil at an elevated temperature. This test method shall be conducted at 100 °C for 30 h unless other conditions are required by the grease specification.

1.2 This test method can be used on any grease with a worked penetration (Test Methods **D217**) greater than 220 mm/10 (NLGI Consistency Number 3 or softer). However, no precision data exists for greases with a penetration greater than 340 mm/10 (softer than NLGI Consistency Number 1).

1.3 The values stated in SI units are to be regarded as standard. The term 60 mesh is used to describe the wire mesh used. This unit was described in the withdrawn **E437** standard and there is no exact metric equivalent.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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:*<sup>2</sup>

**D217** Test Methods for Cone Penetration of Lubricating Grease

**D4175** Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

**E437** Specification for Industrial Wire Cloth and Screens (Square Opening Series) (Discontinued 2000) Replaced by **E 2016** (Withdrawn 2000)<sup>3</sup>

2.2 *Government Standard (Withdrawn):*

**FED-STD-791C** Lubricants, Liquid Fuels, and Related Products; Methods of Testing, Method 321.3 “Oil Separation from Lubricating Greases (Static Technique),” Withdrawn November 2007<sup>4</sup>

## 3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology **D4175**.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee **D02** on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee **D02.G0.03** on Physical Tests

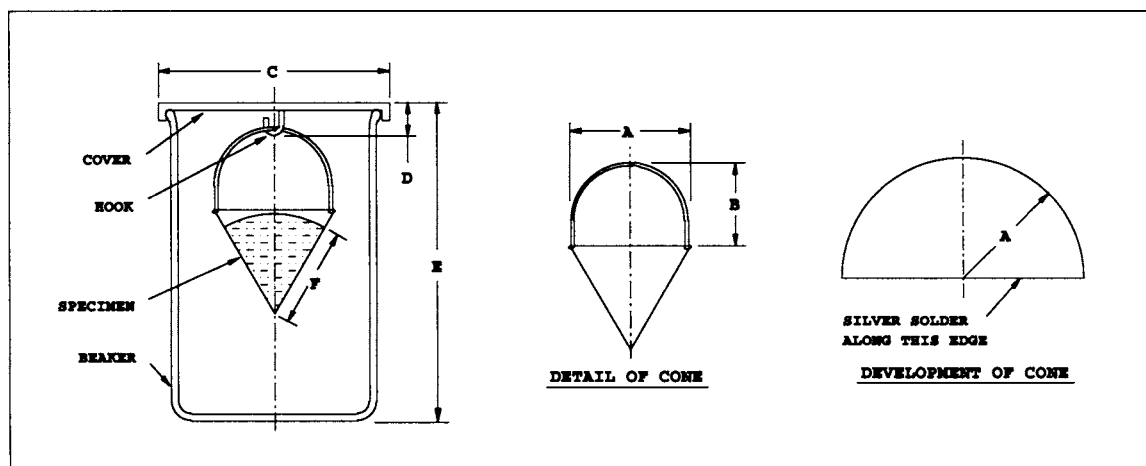
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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard’s Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

<sup>4</sup> Available online at ASSIST Quick Search (<http://quicksearch.dla.mil>).

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



NOTE 1—Dimensions are provided in millimetres (with in./lb. units in parentheses) as follows:

A	$38.1 \pm 0.3$	(1.50 ± 0.01)	radius
B	$27.0 \pm 0.3$	(1.06 ± 0.01)	height
C	70	(2.8)	approximately (to fit beaker)
D	$9.7 \pm 3.3$	(0.38 ± 0.13)	length
E	100	(4)	approximately
F	36	(1.4)	approximately

FIG. 1 Cone Fabrication and Test Setup

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

3.1.2.1 *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.

3.1.3 *oil separation, n*—the appearance of a liquid fraction from an otherwise homogeneous lubricating composition.

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

3.1.4.1 *Discussion*—The thickener 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

#### 4. Summary of Test Method

4.1 The weighed sample is placed in a cone-shaped, wire-cloth sieve, suspended in a beaker, then heated under static conditions for the specified time and temperature. Unless otherwise required by the grease specification, the sample is tested at standard conditions of  $100 \text{ }^\circ\text{C} \pm 0.5 \text{ }^\circ\text{C}$  for  $30 \text{ h} \pm 0.25 \text{ h}$ . The separated oil is weighed and reported as a percentage of the mass of the starting test sample.

#### 5. Significance and Use

5.1 When lubricating grease separates oil, the residual material may change in consistency, which can affect the ability of the product to function as designed. Test results

obtained with this procedure are not intended to predict oil separation tendencies of grease under dynamic service conditions.

5.2 This test method is used for specifications and quality control.

#### 6. Apparatus

6.1 *Test Apparatus*—The assembled apparatus consists of a cone-shaped sieve constructed of 60 mesh, corrosion-resistant wire cloth, a 200 mL tall-form beaker without spout, and a closely fitting cover with a hook in the center. Construction details and dimensions are shown in Fig. 1.

6.1.1 The conical sieve shall be fabricated of stainless steel, monel, or nickel wire cloth, of No. 60 medium-heavy mesh as defined in Table 2 of Specification E437 and shall be fabricated to the dimensions and requirements shown in Fig. 1.

NOTE 1—Sieves of stapled or wire-bound construction, instead of silver-soldered construction, have been used in the past. Such sieves do not conform to this test method.

6.2 *Oven*, capable of maintaining the specified temperature,  $100 \text{ }^\circ\text{C} \pm 0.5 \text{ }^\circ\text{C}$ .

6.3 *Balance*, having a minimum capacity of 250 g and a sensitivity of at least 0.01 g.

#### 7. Sampling

7.1 Examine the sample for any indication of non-homogeneity such as oil separation, phase changes, or gross contamination. If any abnormal conditions are found, obtain a new sample.

7.2 The sample submitted for analysis should be minimally sufficient for duplicate testing.

7.3 Although the mass of the test grease is determined, the sieve is filled by volume rather than mass. Each test will require a quantity sufficient to fill the sieve to approximately the same level as indicated in Fig. 1 (about 10 mL). Approximately the same volume shall be used for each test regardless of the grease density; the mass will range from about 8 g to 12 g.

## 8. Preparation of Apparatus

8.1 Carefully clean the sieve, beaker, and cover, by wiping with a suitable solvent. Allow the sieve to air dry. See Note 2.

NOTE 2—Use of an ultrasonic solvent bath has been found helpful in cleaning the sieve.

8.2 Inspect the sieve to make certain that it is clean and free of deposits that could affect the passage of oil. It shall be free of any screen irregularities such as tears, dents, creases, or enlarged or diminished openings.

## 9. Procedure

9.1 Preheat the oven to test temperature. Unless otherwise specified, the test shall be conducted at standard conditions of  $100\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$  for  $30\text{ h} \pm 0.25\text{ h}$ .

9.2 Weigh the beaker to within 0.01 g,  $W_i$ .

9.3 Tare the assembled apparatus consisting of the sieve, cover, and beaker as shown in Fig. 1 to the nearest 0.01 g.

9.4 With a suitable spatula, fill the cone sieve with sufficient grease to approximate the level shown in Fig. 1, being careful to avoid inclusion of air. Exercise care not to force grease through the screen. Smooth and shape the top of the grease into a slightly convex surface to prevent trapping free oil.

9.5 Assemble the entire apparatus as shown in Fig. 1, and weigh to the nearest 0.01 g. Calculate the mass of the grease by difference,  $G$ .

9.6 Place the assembled apparatus in the oven for the specified time and temperature.

9.7 Remove the apparatus from the oven and cool to room temperature. Remove the cone from the beaker, tapping it gently against the inside of the beaker to take off any oil adhering to its tip. Weigh the beaker containing the collected oil to the nearest 0.01 g,  $W_f$ .

9.8 Soon after completion of the test, clean the apparatus (Section 8) in preparation for subsequent testing.

## 10. Calculation

10.1 Calculate the oil separation as the percentage separated from the original sample as follows:

$$\text{oil separation, \%} = \frac{W_f - W_i}{G} \times 100 \quad (1)$$

where:

$W_i$  = mass of the beaker before heating,  
 $W_f$  = mass of the beaker after heating, and  
 $G$  = mass of the grease tested.

**TABLE 1 Precision Values for Several Oil Separation Percentages**

Oil Separation, %	Repeatability	Reproducibility
1	1.15	1.51
5	2.57	3.39
10	3.64	4.79
20	5.15	6.78

## 11. Report

11.1 Report the following information:

- 11.1.1 Grease identity,
- 11.1.2 Test date,
- 11.1.3 Test temperature and duration, and
- 11.1.4 Oil separation, nearest 0.1 %.

## 12. Precision and Bias

12.1 *Precision*—Eight cooperators tested eight greases for leakage in a cooperative program conducted in accordance with the design-of-experiments described in RR:D02-1007.<sup>5</sup> All tests were run in duplicate. The total range of results were from 0.1 % to 23.7 % leakage (see Table 1).

12.2 The precision of the percent oil separation as determined by this test method was obtained in accordance with ADJ6300 (D2PP software).<sup>6</sup>

12.3 The repeatability and reproducibility of the test method as determined by statistical examination of the interlaboratory results are as follows:

12.3.1 *Repeatability*—The difference between two test results, obtained by the same operator with the same apparatus under constant specified operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in 1 case in 20:

$$\text{repeatability, \% oil separation} = 1.151 \times (M)^{0.5} \quad (2)$$

where:

$M$  = mean of two determinations.

12.3.2 *Reproducibility*—The difference between two single and independent results by two 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 values only in 1 case in 20:

$$\text{reproducibility, \% oil separation} = 1.517 \times (M)^{0.5} \quad (3)$$

where

$M$  = mean of two determinations.

12.4 *Bias*—The procedure for measuring the oil separation from lubricating grease has no bias because the value of the oil loss is defined only in terms of this test method.

<sup>5</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1007. Contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org).

<sup>6</sup> Available from ASTM International Headquarters. Order Adjunct No. ADJ6300.