

Standard Test Method for Oil Separation from Lubricating Grease During Storage¹

This standard is issued under the fixed designation D 1742; 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 Department of Defense.

^{e1} NOTE—Warning notes were placed in text editorially in June 2000.

1. Scope

1.1 This test method covers the determination of the tendency of a lubricating grease to separate oil during storage in both normally filled and partially filled containers.

1.2 This test method is not suitable for greases softer than NLGI No. 1 grade.

1.3 Values stated in acceptable SI units are to be regarded as the standard except for the dimensions in Fig. 2 where inch-pound units are standard.

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 and health practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see 7.1.

2. Referenced Documents

2.1 ASTM Standards:

D 235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Drycleaning Solvent)²

E 11 Specification for Wire-Cloth Sieves for Testing Purposes³

2.2 Other Standard:

NLGI Grease Consistency Classification⁴

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.



FIG. 1 Pressure Bleeding Test Cell A

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

3.1.2.1 *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.

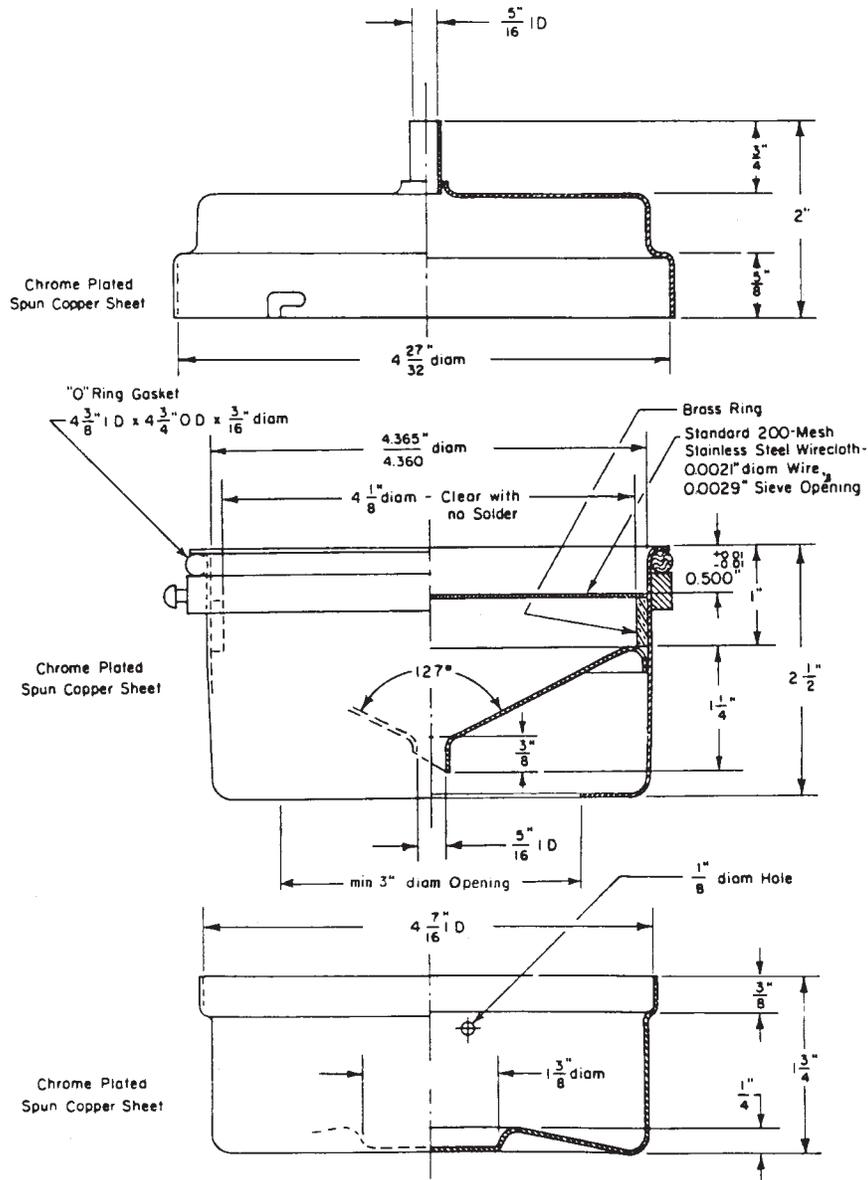
¹ This test method is under the jurisdiction of the ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.G on Lubricating Grease.

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² *Annual Book of ASTM Standards*, Vol 06.04.

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ Available from NLGI, 4635 Wyandotte St., Kansas City, MO 64112.



in.	mm	in.	mm	in.	mm	in.	mm
0.0021	0.053	1 5/8	41.3	0.500	12.70	47/16	112.7
0.0029	0.074	1 3/4	44.4	0.510	12.95	4 1/2	114.3
0.010	0.25	2	50.8	0.520	13.20	4 11/16	119.1
1/16	1.59	2 1/2	63.5	5/8	15.87	4 3/4	120.6
1/8	3.17	3 11/16	93.7	3/4	19.05	4 29/32	123.03
3/16	4.76	4 1/8	104.8	1	25.4	5 1/4	133.3
1/4	6.35	4.360	110.74	1 1/4	31.8	6	152.4
5/16	7.94	4.365	110.87	1 3/8	34.9	6 1/2	165.1
3/8	9.52	4 3/8	111.1	3	76.2		

FIG. 2 Detailed Drawing of Pressure Bleeding Test Cell A

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

4. Summary of Test Method

4.1 The sample of grease, supported on a 75- μ m (No. 200) sieve, is subjected to 1.72 kPa (0.25 psi) air pressure for 24 h at 25°C (77°F). Any oil seepage that occurs drains into a beaker and is weighed.

5. Significance and Use

5.1 When a lubricating grease separates oil, the remaining composition increases in consistency. This can affect the ability of the product to function as designed.

5.2 It has been found that the results of this test correlate directly with the oil separation that occurs in 35-lb pails of grease during storage.



FIG. 3 Pressure Bleeding Test Cell B

5.3 This test method is not intended to predict oil separation tendencies of the grease under dynamic conditions.

6. Apparatus

6.1 *Test Apparatus*⁵—The assembled apparatus consists of a tightly fitting cup and cover which contains a 75- μ m (No. 200) sieve strainer for supporting the grease, a funnel for collecting separated oil, and a 20-mL beaker for retaining the separated oil. A fitting is provided in the cover for inlet air (1.72 kPa (0.25 psi)) and a hole is provided in the side of the cup to prevent back pressure. Fig. 1 is a photograph of a disassembled apparatus. The details of construction are given in Fig. 2. The 75- μ m (No. 200) stainless steel sieve shall conform to the requirements of Specification E 11. The diameter of the 75 μ m (No. 200) sieve shall be 104.8 mm (4 $\frac{1}{8}$ in.), completely clear with no solder showing.

6.2 *Air Pressure Supply and Regulation*— An air pressure supply, controlled by reducing valves or regulators, capable of

maintaining air pressure at 1.72 ± 0.07 kPa, should be used. A manometer, or other suitable pressure indicating device, and a pressure relief valve to protect against pressure surge should be included in the pressure system.

7. Reagents

7.1 *Mineral Spirits*, conforming to Specification D 235. (**Warning**—Combustible. Vapor harmful.)

8. Sampling

8.1 The sample presented for analysis should be large enough to make possible the selection of a representative portion for testing.

8.2 Examine 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.

9. Preparation of Apparatus

9.1 Thoroughly clean the strainer, cup, and oil-collecting beaker with mineral spirits and follow by air drying.

9.2 Carefully inspect the sieve to make certain that there is no build-up which would affect the passage of oil. The sieve must also be free of any surface irregularities such as creases, dents, or punctures. Replace, if necessary.

9.3 The funnel must be clean and free of any residues which can retard the flow of oil.

9.4 Inspect the cover and cup for any dents or distortions to the mating surfaces which can interfere with a tight seal between the units.

9.5 When the O-ring seal shows wear it must be replaced.

10. Procedure

10.1 Place the strainer, screen side up, in the funnel and tare the assembly to the nearest 0.05 g. By means of a spatula, completely fill the space between the screen and the top of the funnel with grease. The volume occupied by the grease shall have a depth of 12.7 ± 0.3 mm (0.50 ± 0.01 in.). Avoid unnecessary working and entrainment of air. Use a straight edge to remove excess grease and provide a level surface. Avoid forcing any of the grease through the screen. Determine the mass of the sample to the nearest 0.05 g.

10.2 Place the tared beaker in the positioning retainer at the center of the bottom cup, and assemble the entire apparatus as indicated in Fig. 1. By means of a suitable length of rubber tubing, connect the source of air to the air inlet fitting in the cover.

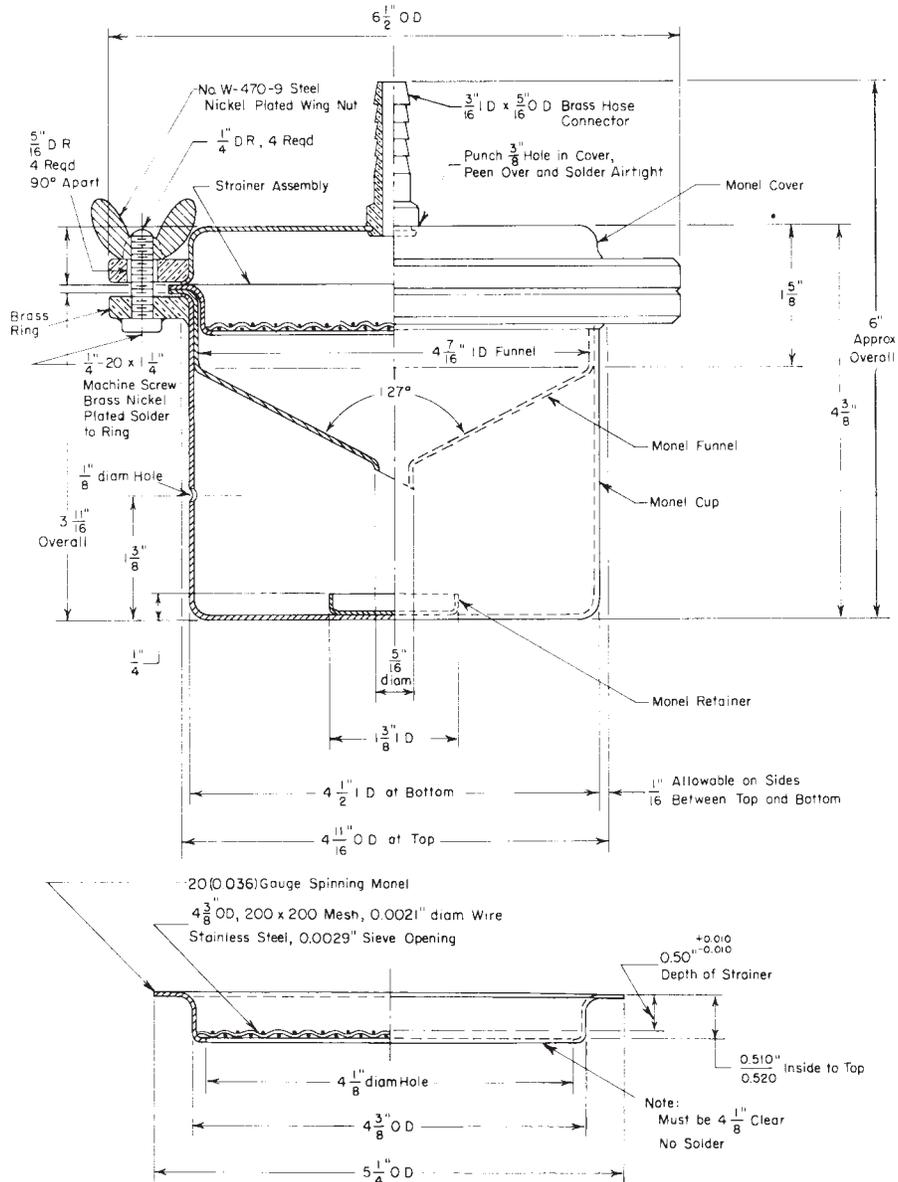
10.3 Bring the apparatus and the sample to $25 \pm 1^\circ\text{C}$ ($77^\circ \pm 2^\circ\text{F}$). Before starting the test, regulate the air pressure to 1.72 ± 0.07 kPa (0.25 ± 0.01 psi) and continue the test for 24 h at this pressure and temperature.

10.4 At the end of the test, weigh the beaker containing the collected oil to the nearest 0.05 g and record.

11. Report

11.1 Report the mass of the oil separated as a percentage of the total mass of the grease sample. Record the value to the nearest 0.1 %.

⁵ The apparatus shown in Fig. 1 and Fig. 2 is available from Koehler Instrument Co., Inc., 1595 Sycamore Ave., Bohemia, LI., NY. 11716 as Catalog No. K189; and Stanhope—Seta Ltd, Park Close, Englefield Green, Egham, Surrey TW20 OXD England. Other appropriately designed equipment can be used such as that shown in Fig. 3, Fig. 4, and Fig. 5 but which is no longer available.



in.	mm	in.	mm	in.	mm	in.	mm
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1/8	3.17	3 1/16	93.7	3/4	19.05	429/32	123.03
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FIG. 4 Detailed Drawing of Pressure Bleeding Test Cell C—Strainer Assembly

12. Precision and Bias

12.1 The precision of the test method is not known to have been obtained in accordance with currently accepted guidelines in Committee D02 research report RR: D02-1007.

12.1.1 *Repeatability* (Note 1)—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 0.1 M in only one case in twenty where:

$$M = \text{mean of two determinations} \quad (1)$$



FIG. 5 Pressure Bleeding Test Cell C

12.1.2 *Reproducibility* (Note 1)—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 0.17 M in only one case in twenty:

$$M = \text{mean of two determinations} \quad (2)$$

NOTE 1—These values of precision are based on results by 10 labora-

tories using four greases that varied in consistency from NGLI Grade No. 1 to Grade No. 3.

12.2 *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 a test method.

13. Keywords

13.1 lubricating grease; oil bleed; oil separation

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