Designation: D1404/D1404M – 99 (Reapproved 2008)

Standard Test Method for Estimation of Deleterious Particles in Lubricating Grease[1]

This standard is issued under the fixed designation D1404/D1404M; 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.

1. Scope

1.1 This test method covers a procedure for the detection and estimation of deleterious particles in lubricating grease.

1.2 This test method is applicable to all lubricating greases. It can also be used to test other semi-solid or viscous materials. Grease fillers, such as graphite and molybdenum disulfide, can be tested for abrasive contaminants by first mixing them into petrolatum or grease known to be free of deleterious particles.

1.3 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard. Within the text, the SI units are shown in brackets.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

D785 Test Method for Rockwell Hardness of Plastics and Electrical Insulating Materials

D4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants

3. Terminology

3.1 *Definitions*—See Terminology D4175.

3.2 *Definitions:*

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

3.2.2 *lubricating grease*, *n*—a semi-fluid to solid product of a dispersion of a thickener in a liquid lubricant.

3.2.2.1 *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 are commonly included to impart special properties.

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

3.2.3.1 *Discussion*—The solid thickener can be fibers (such as various metallic soaps) or plates or spheres (such as certain non-soap thickeners), which are insoluble or, at the 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.

3.3 *Definitions of Terms Specific to This Standard:*

3.3.1 *deleterious particles*, *n*—in lubricating grease, minute bits of solid material present as a contaminant and abrasive to acrylic plastic.

4. Summary of Test Method

4.1 A small portion of the lubricating grease sample is placed between two clean, highly polished acrylic-plastic plates held rigidly and parallel in metal holders. A pressure of 200 psi [1.38 MPa] is applied, and one plate is rotated 30° relative to the other. Particles harder than the plastic and exceeding in size the distance between the plates will imbed in the plates and cause characteristic, arc-shaped scratches in the plates.

4.2 The relative number of such solid particles can be estimated by counting the total number of arc-shaped scratches on the two plates.

[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.G0.01 on Chemical and General Laboratory Tests.

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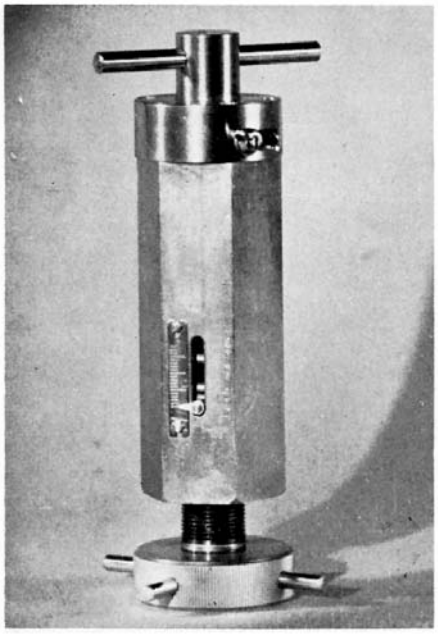
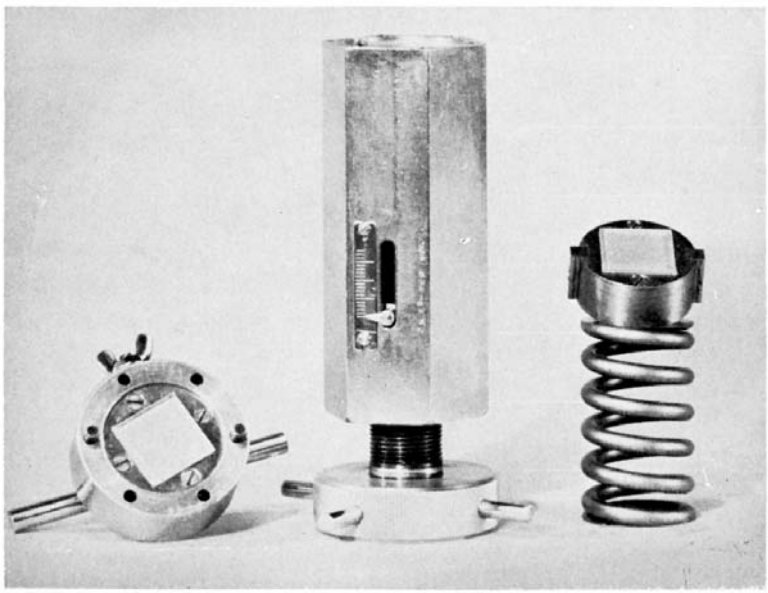
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



(*a*) Assembled View



(*b*) Unassembled View

FIG. 1 Apparatus for Determining Deleterious Particles in Lubricating Grease

5. Significance and Use

5.1 The significance of the number of scratches as far as correlation with field performance is concerned has not been established. A particle which is abrasive to plastic will not necessarily be abrasive to steel or other bearing materials. Some correlation was obtained in that the contaminant used in Sample 3 (see 10.1.1) had a greater wear rate in a laboratory ball bearing abrasive wear test than the contaminant in Sample 2.

NOTE 1—The number of scratches obtained cannot be used to draw fine differences between greases, but rather, to group them into two or three general classes. One such possible division could be:

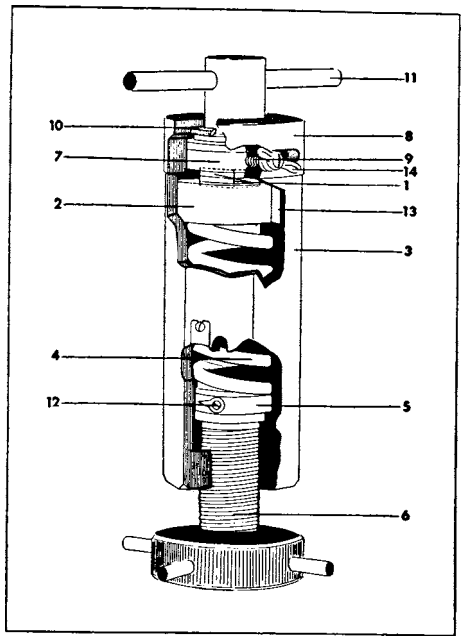
1 ... . less than 10 scratches
2 ... . 10 to 40 scratches
3 ... . more than 40 scratches

5.2 An advantage of this test method is that each test takes only a few minutes to run.

5.3 This test method is used for quality control and specification purpose.

6. Apparatus

6.1 The test apparatus is shown in Figs. 1 and 2. As illustrated in Fig. 2, the plastic test plates (commercial acrylic



1 plastic test plates
2 lower plate holder
3 body
4 spring
5 spring follower plate
6 loading screw
7 upper plate holder
8 cap
9 threaded pin
10 leather washer
11 handle
12 indicator pin
13 key
14 wing nut

FIG. 2 Cross-section View of Apparatus

plastic, produced in uniform, highly polished sheets),[3,4] *1*, are rigidly held in a parallel position in square recesses in parts *2* and *7*. The holder, *7*, is part of the cap assembly, *7* to *11*, which can be removed as a unit from the body, *3*, by removing four cap screws. The lower plate holder, *2*, can slide along the axis of the main housing, but it is restricted from turning by keys spaced 180° apart. Pressure is applied to the plastic plates, *13*, through the holder, *2*, by means of the coil spring, *4*, spring follower plate, *5*, and loading screw, *6*. The large loading screw also serves as a base for the apparatus. The indicator pin, *12*, on the spring follower plate, *5*, shows the linear amount of spring compression, which in turn, is a measure of the pressure applied to the two plastic plates. The upper holder, *7*, is kept from rotating by the threaded pin, *9*, which projects through a milled slot in cap, *8*, and ends with a locking wing nut, *14*. To rotate the upper plate and holder, the wing nut is loosened, and the handle, *11*, is turned. A leather washer, *10*, between holder, *7*, and cap, *8*, is used to facilitate turning.

NOTE 2—Although 6.1 describes an apparatus of specific design, any other device that provides the essential operating conditions can be used. Such device is permitted if it can hold the two plastic test plates parallel to each other; apply and measure a minimum pressure of 200 psi [1.38 MPa] on the plastic plates; and provide for 30° relative rotation of the two plates.

6.2 *Plastic Test Plates*[4,5]—acrylic plastic test plates (two per test), 1 ± 0.050 in. square by ⅛ ± 0.025 in. [25.4 ± 1.3 mm square by 3.2 ± 0.64 mm] uniformly thick, having a Rockwell M hardness of 94 ± 10 (Test Method D785), and having highly polished surfaces protected on both sides with protective paper.

NOTE 3—Whether dimensioned in inches or millimetres, the plastic test plates should be fabricated to match the square recesses in holders *2* and *7*.

7. Reagents and Materials

7.1 Stoddard solvent conforming to Specification D235. (**Warning**—Flammable. Vapor harmful.)

NOTE 4—Other naphthas or pure paraffinic hydrocarbons, such as *n*-heptane, can be substituted providing they are suitable volatile and do not soften or otherwise attack acrylic plastic.

8. Procedure

8.1 Use a fine, smoothing file to break sharp edges of one face of each test plate. (A sharp edge could scratch the face of the opposite plate.) Remove plastic filings and dust. Do not remove protective paper at this time.

8.2 Completely remove the tension from the spring and disassemble the apparatus from the top, Fig. 1b. Snap a test plate into each of the square recesses of holders *2* and *7* (Fig. 2); locate the plates such that the surfaces with the filed edges are opposite each other.

NOTE 5—The square recesses should be clean, that is, free of solid material, to preclude uneven loading of the plastic plates.

8.3 Remove the protective paper from the test plates. The exposed polished surfaces must be free of scratches and dust. Remove any incidental dust with a soft brush (beware of static charges attracting more dust), damp chamois skin, or compressed-gas (ionizing-type recommended) dust remover.

8.4 Place a pea-sized (about 0.25 g), representative sample of the test grease on the face of the lower test plate. Bolt on the cap assembly, making sure the threaded pin, *9*, is against one stop. Use the locking wing nut, *14*, to retain this position. Turn the loading screw to apply a pressure of 200 psi [1.38 MPa] as indicated by the pointer and scale and the apparatus maker's spring calibration.

NOTE 6—The scale indicates linear displacement and does not directly indicate pressure. The apparatus should be supplied with a calibration to convert the scale reading to pressure. (Consult the manufacturer if this calibration information is missing.) One equipment maker reported the following calibration:

$$\text{Scale reading of } 29 = 200 \text{ psi } [1.38 \text{ m}PA], \pm 5\ \%. \quad (1)$$

Equipment made at other times or by other manufacturers can vary and can have different calibrations.

NOTE 7—The pressure applied to the plates determines the thickness of the grease layer between them. A pressure of 200 psi [1.38 MPa] gave a thinner layer than 100 psi [0.69 MPa], but increasing the pressure from 200 to 500 psi [1.38 to 3.45 MPa] caused very little change.

8.5 With this pressure applied, loosen the locking wing nut, *14*, on the cap assembly and turn handle, *11*, once as far as it will go (about 30°).

8.6 Release the spring pressure and disassemble the apparatus. Carefully remove the plastic plates from their holders.

8.7 Mark the plates for identification and remove the protective paper from the back faces. Wash the test grease from the

[3] The sole source of supply of sized test plates known to the committee at this time is Koehler Instrument Company, Inc., 1595 Sycamore Ave., Bohemia, NY 11716.

[4] If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[5] The apparatus shown in Figs. 1 and 2 is available from Falex Corp., 1020 Airpark Dr., Sugar Grove, IL 60554–9585, and from Koehler Instrument Company, Inc., 1595 Sycamore Ave., Bohemia, New York 11716.

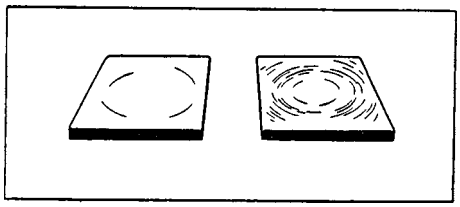


FIG. 3 Plastic Test Plates Showing Different Degrees of Scratching

plates with filtered Stoddard solvent (**Warning**—Flammable. Vapor harmful) and blow dry with filtered air. Handle the test plates very carefully to avoid accidental scratches.

8.8 Count and record the total number of arc-shaped scratches on the two plastic test plates. See Fig. 3 for examples of different degrees of scratching.

9. Report

9.1 Report the following information:

9.1.1 Date,

9.1.2 Grease identity,

9.1.3 Test pressure to the nearest 10 psi [0.07 MPa], and

9.1.4 Total number of scratches on the two test plates.

10. Precision and Bias [6]

10.1 *Precision*—The precision of this test method was not determined with any currently acceptable guidelines of either ASTM or Committee D02.

10.1.1 A series of cooperative tests were run on three grease samples, two of which were artificially contaminated. The results from six different laboratories were:

Sample	Average No. of Scratches	Standard Deviation
1	4	6
2	41	8
3	113	27

10.2 *Bias*—The procedure in Test Method D1404/D1404M has no bias because the value of the estimation of deleterious particles in lubricating grease can be defined only in terms of a test method.

11. Keywords

11.1 abrasive particles; cleanliness; contamination; deleterious particles; lubricating grease; particles; scratch test

[6] There is no research report on file because this test method was developed before research report guidelines were instituted and data are no longer available.