Designation: D1092 – 11

Standard Test Method for Measuring Apparent Viscosity of Lubricating Greases[1]

This standard is issued under the fixed designation D1092; 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.

1. Scope

1.1 This test method covers measurement, in poises, of the apparent viscosity of lubricating greases in the temperature range from −54 to 38°C (−65 to 100°F). Measurements are limited to the range from 25 to 100 000 P at 0.1 s^{-1} and 1 to 100 P at 15 000 s^{-1}.

NOTE 1—At very low temperatures the shear rate range may be reduced because of the great force required to force grease through the smaller capillaries. Precision has not been established below 10 s^{-1}.

1.2 This standard uses inch-pound units as well as SI (acceptable metric) units. The values stated first are to be regarded as standard. The values given in parentheses are for information only. The capillary dimensions in SI units in Fig. A1.1 and Fig. A1.2 are standard.

1.3 **WARNING—**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 Material Safety Data Sheet (MSDS) 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.

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]

D88 Test Method for Saybolt Viscosity

D217 Test Methods for Cone Penetration of Lubricating Grease

D3244 Practice for Utilization of Test Data to Determine Conformance with Specifications

3. Terminology

3.1 *Definitions:*

3.1.1 *apparent viscosity, n*—of a lubricating grease is the ratio of shear stress to shear rate calculated from Poiseuille's equation, and is measured in poises (see 10.1).

3.1.2 *capillary, n*—For the purpose of this method, a capillary is any right cylindrical tube having a length to diameter ratio of 40 to 1.

3.1.3 *shear rate, n*—the rate at which a series of adjacent layers of grease move with respect to each other; proportional to the linear velocity of flow divided by the capillary radius, and is thus expressed as reciprocal seconds.

4. Summary of Test Method

4.1 The sample is forced through a capillary by means of a floating piston actuated by the hydraulic system. From the predetermined flow rate and the force developed in the system, the apparent viscosity is calculated by means of Poiseuille's equation. A series of eight capillaries and two pump speeds are

[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.G0.02 on Consistency and Related Rheological 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.

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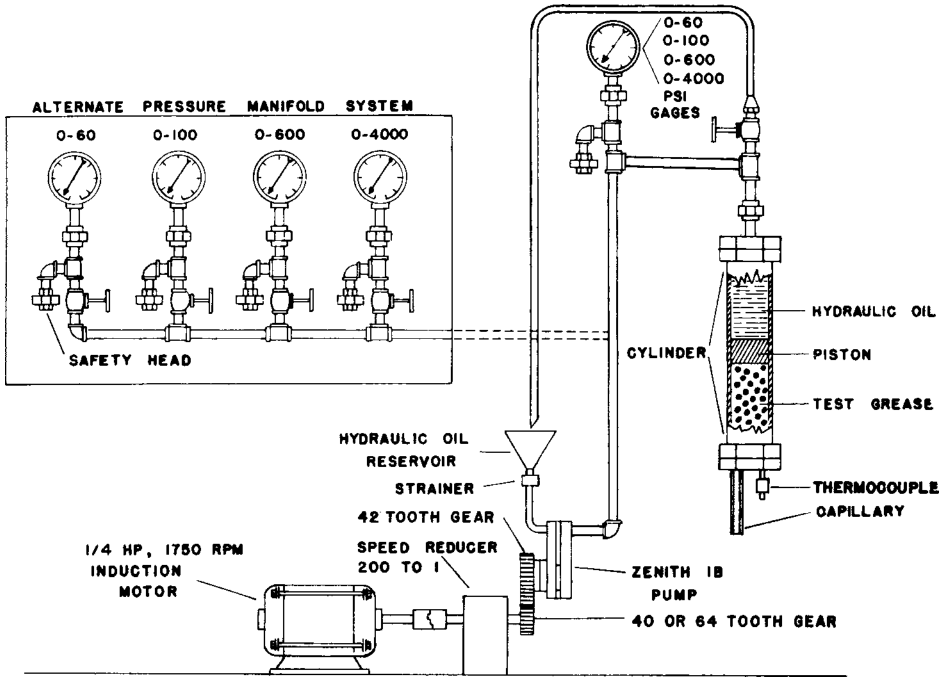

FIG. 1 Schematic Drawing of Apparatus

used to determine the apparent viscosity at sixteen shear rates. The results are expressed as a log-log plot of apparent viscosity versus shear rate.

5. Significance and Use

5.1 Apparent viscosity versus shear rate information can be useful in predicting pressure drops in grease distribution systems under steady-state flow conditions at constant temperature.

6. Apparatus

6.1 The assembled pressure viscometer consists of four major divisions, the power system, the hydraulic system, the grease system (described in the annex and shown in Fig. 1), and a bath of optional design. Fig. 2 is a photograph of the first three divisions as commonly used at room temperature. This form of the apparatus can be used with a cylindrical insulated tank 178 mm (7 in.) in diameter and 508 mm (20 in.) deep. The bath medium may be kerosene or alcohol cooled manually with dry ice. Alternatively the grease system, the grease and hydraulic system, or all three major divisions can be built into any liquid or air bath that will cover the temperature range and maintain the grease at test temperature ±0.25° C (±0.5°F).

7. Sampling

7.1 A single filling of the grease cylinder requires about 0.223 kg (½ lb) of grease which is the minimum size sample.

NOTE 2—It is possible for an experienced operator to complete the 16 single determinations with a single filling. However, some samples reach the equilibrium pressure slowly, making it advisable to have a sample of several pounds available.

7.2 Generally no special preparation of the sample is necessary.

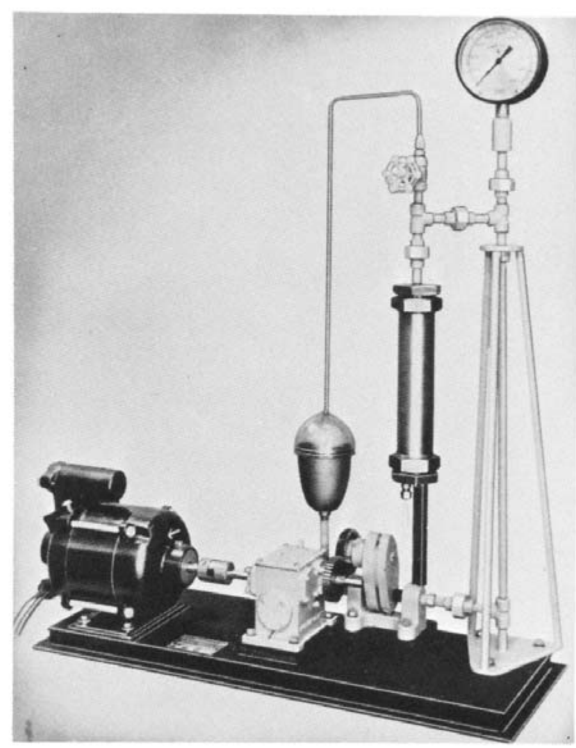

FIG. 2 Photograph of Apparatus

NOTE 3—The apparatus works the samples to some extent as they pass through the capillary. Somewhat better precision is obtained if they are previously worked as described in Test Methods D217. Working of some greases may cause aeration.

NOTE 4—It is desirable to filter some greases through a 60-mesh screen to prevent plugging the No. 8 capillary. Follow prudent laboratory practice to keep equipment cleaned and flushed before use.

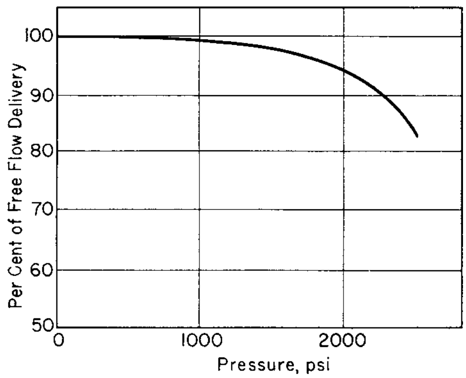

FIG. 3 Typical Pump Calibration Curve

8. Calibration and Standardization

8.1 To calibrate the hydraulic system, remove the grease cylinder and replace it with a needle valve. Select a hydraulic oil of about 2000 cSt (2000 mm^2/s) viscosity at the test temperature. Fill the system with hydraulic oil and circulate the oil until it is free of air bubbles. At atmospheric pressure, quickly place a 60-mL Saybolt receiving flask (Test Method D88), under the outlet and start a timer. Determine the delivery time for 60 mL and calculate the flow rate in cubic centimetres per second assuming 1 mL equal to 1 cm^3. Repeat this observation at 500, 1000, 1500 psi (3.45, 6.89, 10.4 MPa) and at sufficient pressures above 1500 psi to develop a calibration curve of the type as shown in Fig. 3. The developed curve of the type is used to correct flow rates when grease is dispensed. Repeat the calibration at intervals to determine if wear is changing the pump flow.

8.2 An alternative procedure for the calibration of the hydraulic system is the measurement of the rate of flow of the test grease. To cover the desired range of shear rates, flow rates over an approximate range of pressure are determined. Any suitable means of measuring the rate of grease flow may be used.

9. Procedure

9.1 Charge the sample so as to reduce inclusion of air to a minimum. Soft greases may be poured into the cylinder or drawn up by vacuum; heavy samples must be hand packed. When filling the cylinder by vacuum, remove the capillary end cap and place the piston flush with the open end and then insert into the sample. Apply vacuum to the opposite end of the cylinder until the cylinder is fully charged with grease. This must be facilitated by tapping with a wooden block. Replace the capillary end cap and fill the upper end of the cylinder above the piston with hydraulic oil.

9.2 Fill the entire hydraulic system with hydraulic oil. Disconnect, invert and fill the gage and gage connections with oil. With the entire hydraulic system connected and completely filled with oil, adjust the temperature of the sample to the test temperature ±0.25°C (±0.5°F) as determined by a thermocouple inserted in the capillary end cap. Operate the pump until oil flows from the gage connection on the viscometer before reconnecting the gage. With the entire viscometer assembled, circulate hydraulic oil with the return valve open until all trace of air is eliminated.

9.2.1 The time to attain test temperature varies with the bath. At −54°C (−65°F) the grease in an unstirred liquid bath should be ready to test in 2 h. Air baths can take as long as 8 h. An ASTM Thermometer 74F in the bath serves as a convenient secondary means of measuring the temperature at –54°C (−65°F). In an air bath the thermometer must be within 25.4 mm of the capillary.

9.3 With No. 1 capillary in place and the 40-tooth gear connected, operate the pump with the return valve closed until equilibrium pressure is obtained. Record the pressure. Change to the 64-tooth gear and again establish equilibrium. Record and relieve the pressure. Replace the No. 1 capillary with subsequent ones and repeat these operations until tests have been run with all capillaries at both flow rates. With some soft or hard greases, it cannot be practical to use all of the capillaries.

NOTE 5—It may be necessary to refill the cylinder with fresh grease when all 16 determinations are to be made.

NOTE 6—The use of an equivalent non-mercury filled replacement thermometer is under study in Subcommittee E20.09.

10. Calculation

10.1 Calculate apparent viscosity of the grease as follows:

$$\eta(\text{apparent viscosity}) = F/S \tag{1}$$

where F is the shear stress, and S is the shear rate. Therefore:

$$\eta = F/S = \frac{p\pi R^2/2\pi RL}{(4v/t)/\pi R^3} = p\pi R^4/(8Lv/t) = P68944\pi R^4/(8Lv/t) \tag{2}$$

where:

p = pressure dynes/cm^2,
L = capillary length, cm,
P = observed gage pressure, psi (multiply by 68944 to convert to dynes per square centimetre),
R = radius of capillary used, cm, and
v/t = flow rate, cm^3/s.

10.2 Calculations may be reduced to a minimum by preparing a table of 16 constants, one for each capillary and shear rate (Table 1). For example, viscosity with No. 1 capillary and the 40-tooth gear is given as follows:

$$\eta = P(\text{observed})68944\pi R^4/(8Lv/t) \text{ or } PK_{(1\text{–}40)} \tag{3}$$

where:

$$K_{(1-40)} = 68944\ \pi\ R^4/(8\text{L}v/\text{t}) \tag{4}$$

10.3 Also calculate the shear rates as follows:

$$S = (4v/t)/\pi R^3 \tag{5}$$

Correct the flow rate to correspond to the observed pressure by reference to Fig. 3. Calculate 16 shear rates for the eight capillaries and two flow rates. This calculation need not be repeated for each run since it will remain constant until recalibration of the pump indicates a revision.

10.4 Plot a curve of apparent viscosity versus shear rate on log-log paper, as shown in Fig. 4.

NOTE 7—Shear stresses also can be calculated by multiplying apparent viscosities by their corresponding shear rates. For solving various problems involving the steady flow of greases, shear stress-shear rate relationships may be plotted on appropriate charts. Instructions on the use of these