Designation: D1092 − 12 (Reapproved 2017)

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 U.S. 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 °C to 38 °C (−65 °F to 100 °F). Measurements are limited to the range from 25 P to 100 000 P at 0.1 s^{-1} and 1 P 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.

1.3.1 In addition, temperature measuring devices such as liquid-in-glass thermometers, thermocouples, thermistors, or platinum resistance thermometers that provide equivalent or better accuracy and precision, that cover the temperature range for ASTM thermometer 49C, may be used.

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:*[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 test 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 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.

[1] 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.G0.02 on Consistency and Related Rheological Tests.

Current edition approved Aug. 1, 2017. Published August 2017. Originally approved in 1950. Last previous edition approved in 2012 as D1092 – 12. DOI: 10.1520/D1092-12R17.

[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.

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.

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.

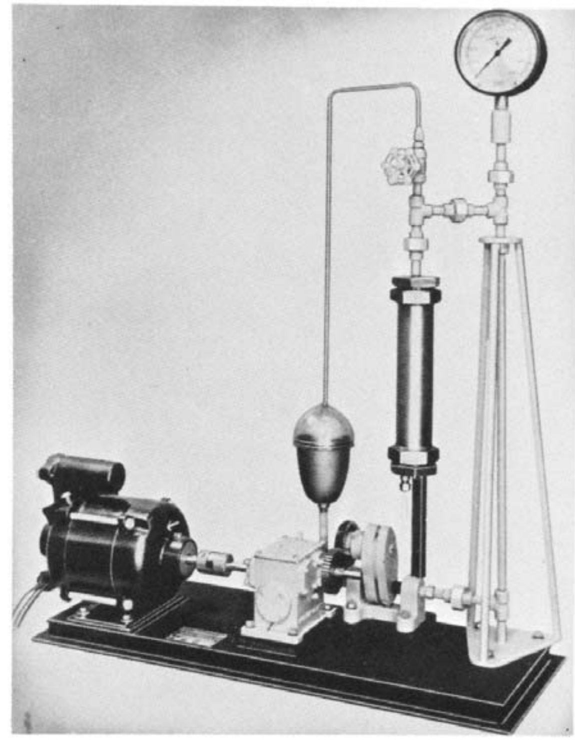

FIG. 2 Photograph of Apparatus

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.

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

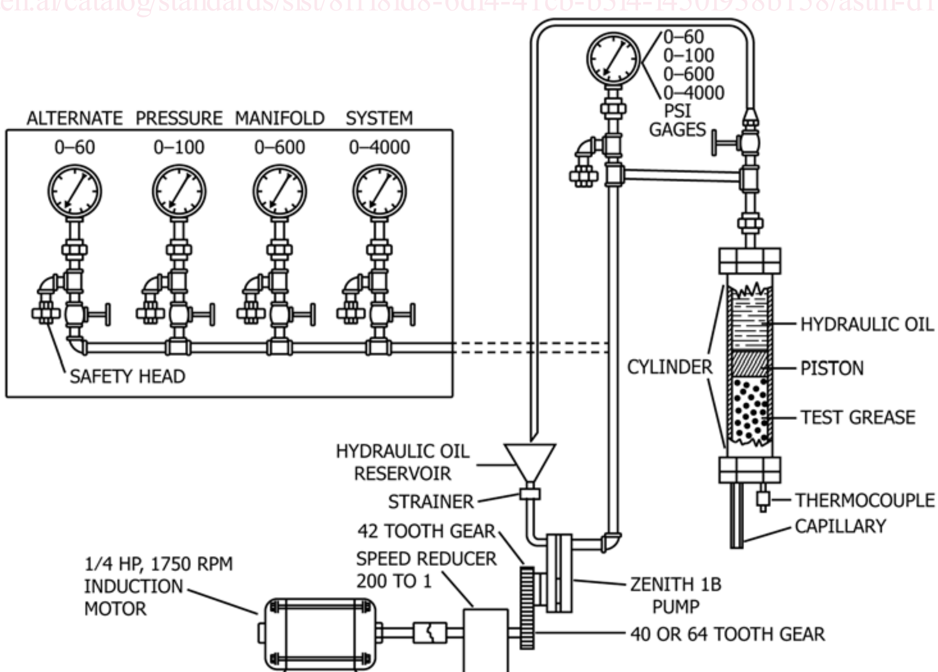

FIG. 1 Schematic Drawing of Apparatus

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 psi, 1000 psi, 1500 psi (3.45 MPa, 6.89 MPa, 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.

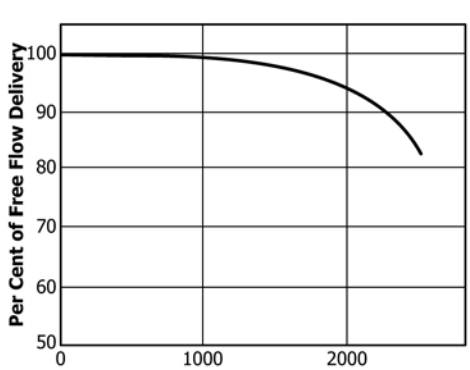

FIG. 3 Typical Pump Calibration Curve

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.

NOTE 5—The use of an equivalent non-mercury filled replacement thermometer, such as a thermistors, platinum resistance thermometer, other liquid in glass thermometer, or thermocouple is under study in Subcommittee E20.09.

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

NOTE 7—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:

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

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

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

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:

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

where:

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

10.3 Also calculate the shear rates as follows:

$$S = (4v/t)/\pi R^3 \qquad (5)$$

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.

TABLE 1 Suggested Data Sheet for Recording Test Results (With Illustrative Test Values)

Sample . . . . *No. 2 Grease* Temperature . . . . *25°C*

Date . . . . *Nov. 1, 1948* Operator . . . . *R.S.*

1	2	3	4[A]	5[B]	6[A]	7[C]
Capillary	Gear	Observed Pressure, *P*, psi	$K = 68944$ $\pi R^4/(8Lv/t)$	Apparent Viscosity, *n* poises, $= P \times K$	Shear Rate, *S*, s^{-1} = $(4v/t)/\pi R^3$	Shear Stress, dynes per sq cm = $n \times S$
1	40	25.5	28.10	716	15	10 740
2	40	38.3	6.83	267	61	16 300
3	40	48.8	3.61	176	120	21 100
4	40	63.5	1.90	120	230	27 800
5	40	96.5	0.89	86	480	41 300
6	40	125	0.58	72.6	755	54 800
7	40	286	0.139	39.8	3 140	125 000
8	40	546	0.0464	25.3	9 320	235 500
1	64	29.5	17.60	520	24	12 470
2	64	45.8	4.27	195	98	19 100
3	64	60	2.26	135.5	195	26 400
4	64	82.3	1.19	97.9	370	36 250
5	64	130	0.556	72.4	770	55 800
6	64	165	0.363	59.9	1 220	73 200
7	64	384	0.087	33.4	5 020	167 500
8	64	720	0.029	20.9	14 900	311 000

[A] Values in this column are predetermined.
[B] Column 3 times Column 4.
[C] Column 5 times Column 6.

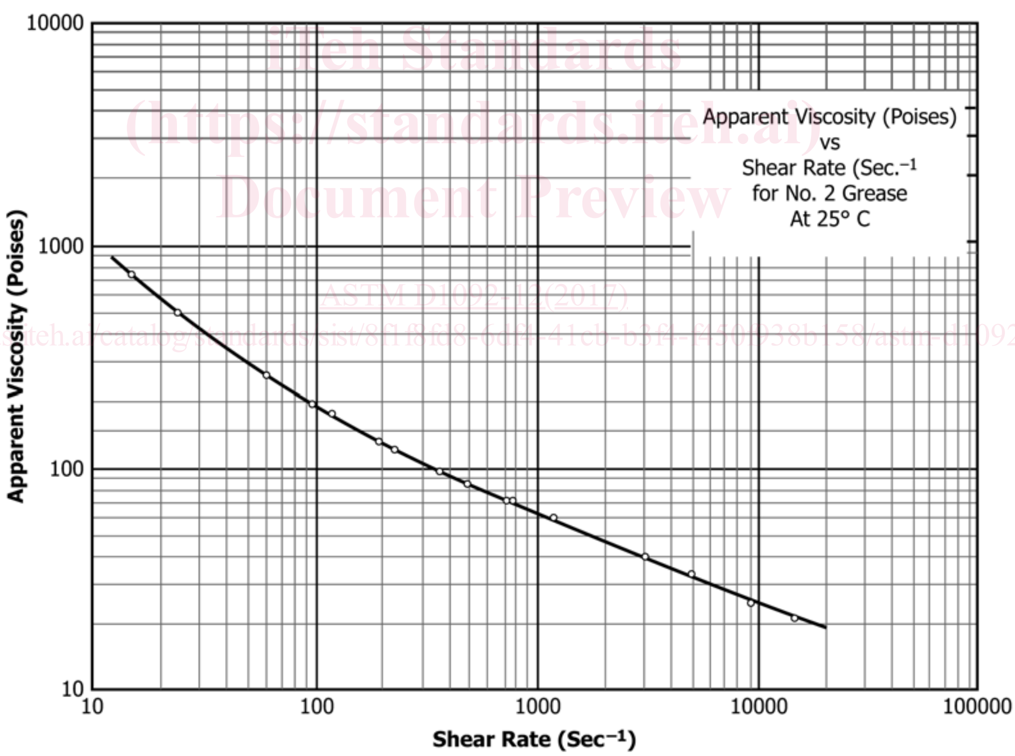

FIG. 4 Typical Chart for Apparent Viscosity versus Shear Rate

NOTE 8—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 charts are given in the article by Rein and McGahey.[3]

11. Precision and Bias

11.1 Due to the nature of the results, the precision of this test method was not obtained according to RR:D02-1007, "Manual on Determining Precision Data for ASTM Methods on Petroleum Products and Lubricants." The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

[3] Rein and McGahey, "Predicting Grease Flow in Large Pipes," *NLGI Spokesman*, April 1965.