Designation: D7918 – 17

Standard Test Method for Measurement of Flow Properties and Evaluation of Wear, Contaminants, and Oxidative Properties of Lubricating Grease by Die Extrusion Method and Preparation[1]

This standard is issued under the fixed designation D7918; 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 the determination and evaluation of flow properties, wear levels, contaminants, and oxidative condition of new and in-service lubricating grease.

1.2 This test method provides guidance on evaluating in-service grease samples, NLGI grades 00 to 3, for wear, consistency, contamination, and oxidation.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3.1 *Exception*—The exception to this will be where units of references were developed by the developers of the test equipment and necessary to report the results of the test.

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.*

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]

D217 Test Methods for Cone Penetration of Lubricating Grease

D6595 Test Method for Determination of Wear Metals and Contaminants in Used Lubricating Oils or Used Hydraulic Fluids by Rotating Disc Electrode Atomic Emission Spectrometry

D7527 Test Method for Measurement of Antioxidant Content in Lubricating Greases by Linear Sweep Voltammetry

D7546 Test Method for Determination of Moisture in New and In-Service Lubricating Oils and Additives by Relative Humidity Sensor

D7718 Practice for Obtaining In-Service Samples of Lubricating Grease

D7843 Test Method for Measurement of Lubricant Generated Insoluble Color Bodies in In-Service Turbine Oils using Membrane Patch Colorimetry

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 *active grease sampling device, n*—device designed to take an active sample of a lubricating grease from a bearing, gear, or drive shaft located in a grease lubricated component. **D7718**

3.1.1.1 *Discussion*—The Grease Thief (trademarked) Type 2[3] is a tool which meets this description and can be used to obtain an active grease sample from a lubricated component. A full description and dimensions of this device can be found in Annex A2.

[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 D02.96.07 on Integrated Testers, Instrumentation Techniques for In-Service Lubricants.

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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.

[3] The Grease Thief Type I and Type II is described in US Patent No. 7984661. Interested parties are invited to submit information regarding the identification of an alternative(s) to this patented item to the ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. The sole source of the Grease Thief Type I and Type II are known to the committee is York Laboratories, LLC located at 410 Kings Mill Rd., York, PA 17401. 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.

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

3.1.2 *active sampling, v*—to use a sampling device to actively gather an in-service lubricating grease sample from a grease-lubricated component. **D7718**

3.1.3 *calibration, n*—the determination of the values of the significant parameters by comparison with values indicated by a set of reference standards. **D6595**

3.1.4 *consistency, n—of lubricating grease*, the degree of resistance to movement under stress. **D217**

3.1.4.1 *Discussion*—The term "consistency" is used somewhat synonymously with "penetration." Generally, consistency refers to the worked penetration of a grease.

3.1.5 *linear sweep voltammetry, n*—a test method designed to monitor the anti-oxidant additive content in lubricating greases. **D7527**

3.1.6 *lubricating grease, n*—a semi-fluid to solid product of dispersion of a thickener in a liquid lubricant. **D217**

3.1.6.1 *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.

3.1.7 *in-service lubricating grease, n*—lubricating grease that has been applied as a lubricant to a gear, bearing, or drive screw for any period of time. **D7718**

3.1.8 *passive grease sampling device, n*—a device designed to gather a sample from the equipment by being attached to the grease reservoir at the purge point. **D7718**

3.1.8.1 *Discussion*—The Grease Thief (trademarked) Type 1[3] is a tool which meets this description and can be used to obtain a representative grease sample from a purge path. A full description and dimensions of this device can be found in Annex A1.

3.1.9 *passive sampling, v*—to use a passive grease sampling device to collect a purged sample of in-service lubricating grease from a purge path. **D7718**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *die extrusion index, n*—an average of three unique test conditions of the in-service lubricating grease sample expressed as a percent when compared to the average of three unique test conditions of the unused lubricating grease baseline.

3.2.2 *die extrusion tester, n*—a device that measures the consistency of in-service lubricating greases and compares the measurement to the values obtained from a baseline sample of lubricating grease.

3.2.3 *ferrous debris level, n*—the total amount of ferrous metal as measured by a Hall-effect sensor in the grease sample independent of particle size.

3.2.4 *full attenuation, n*—the loss of intensity in signal strength.

3.2.5 *grease colorimetry, n*—the science of color measurement by the evaluation of the CIE LAB values of grease in the visible light region of the electromagnetic spectrum.

3.2.6 *grease ribbon, n*—grease that is extruded onto a substrate.

3.2.7 *indexing die, n*—a single-use orifice die to measure the consistency as compared to the baseline.

3.2.8 *spring cell calibration cylinder, n*—a cylinder that houses a spring and a push rod to compress the spring.

3.2.9 *substrate, n*—the single-use strip onto which a ribbon of grease is extruded.

3.2.10 *substrate segment, n*—a peel-away section of the substrate containing a portion of the extruded grease; the segments are pre-cut to hold 0.25 g of extruded grease ribbon.

3.2.11 *speed 1, n*—the slowest speed in which the grease is extruded from the indexing die.

3.2.12 *speed 2, n*—the fastest speed in which the grease is extruded from the indexing die.

3.2.13 *speed 3, n*—the intermediate speed at which the grease is extruded from the indexing die.

4. Summary of Test Method

4.1 Testing of a grease sample includes steps that characterize the ferrous wear within the sample, the consistency of the sample, and properties related to its chemistry.

4.2 A grease sample of known volume is measured to determine the density of ferrous material in the sample.

4.3 A grease sample held with a defined geometry sample holder is placed into a temperature-controlled instrument and extruded onto a substrate as a thin ribbon or strip of grease. The extrusion process provides a measurement of the consistency of the grease.

4.4 The test method requires the grease to be tested at three different rates to reflect the non-Newtonian nature of greases. Testing at several different rates creates a series of step changes that are then compared to an unused baseline grease that has also been tested under the same conditions.

4.5 While the flow properties are being measured, the grease is simultaneously deposited onto a thin-film substrate containing substrate segments. Each substrate segment contains approximately 0.25 g of grease. The individual substrate segments are used for further testing of wear, contamination, and oxidative properties.

4.6 The substrate is removed from the instrument and processed further to obtain information related to the chemistry and content of the grease sample including linear sweep voltammetry and grease colorimetry.

5. Significance and Use

5.1 Trending the wear, contamination, consistency, and oxidative properties of a lubricating grease is a crucial part of condition-monitoring programs. Changes in these properties or deviations from the new grease can be indicative of problems within the lubricated component, such as the mixing of incompatible thickener types, excessive wear or contaminant levels, or significant depletion of antioxidant levels. These test methods also makes it possible to develop trends that can be used to predict failures before they occur and allow for corrective action to be taken.

6. Interferences

6.1 *Particulate Matter*—When the in-service grease sample is heavily contaminated with particulate matter, it may be necessary to perform a dilution of the sample in order to properly extrude the sample onto the substrate. Larger pieces of particulate matter may also cause spikes in the load profile graph. The data from these spikes should be discounted from the average force calculations because they are not indicative of changes in the flow properties of the sample.

6.1.1 Heavily contaminated grease samples are considered any grease that contains solid particulate of size or quantity that results in clogging of the die, which inhibits the completion of the test.

NOTE 1—The specific steps to perform a dilution of grease samples is not addressed in this test method.

6.2 *Sample Size*—The passive sampling device shall be at least 70 % filled to perform the testing indicated within this test method. If the sampling device is insufficiently filled, the instrument may not record a force reading over the entirety of the program.

6.3 *Color*—The grease colorimetry test may become insignificant if the color of the grease causes full attenuation and minimal transmittance of the signal making it incomparable to the new grease.

7. Apparatus

7.1 *Passive Grease Sampling Device*—The device that holds the grease sample. The device is typically capable of holding approximately 1.5 g to 2 g of grease when full. A full description and dimensions of this device can be found in Annex A1.

7.2 *Active Grease Sampling Device*—The device that holds an active grease sample from a grease-lubricated component. A full description and dimensions of this device can be found in Annex A2.

7.3 *Die Extrusion Tester*[4]—Designed for the passive grease sampling device, the die extrusion tester is a temperature-controlled chamber consisting of a table that is controlled by a linear actuator that holds the substrate, a bracket that holds the passive grease sampling device, and a second linear actuator to push the handle of the passive grease sampling device. Both the passive grease sampling device and the second linear actuator are mounted perpendicular to the substrate table. A load cell is mounted between the second actuator and the handle of the passive grease sampling device to measure the force during the extrusion. The die extrusion tester is shown in Fig. 1.

7.4 *Constant Temperature Chamber*—The test specimens in the passive grease sampling device should be heated in the temperature-controlled chamber of the die extrusion tester prior to performing any test. The passive grease sampling device shall have the indexing die firmly engaged on the threaded open end of the device.

7.5 *Ferrous Debris Analyzer*—An analyzer that allows the passive grease sampling device to be inserted completely in the coil geometry, designed to measure the total amount of ferrous debris in the grease sample. By use of a Hall-effect sensor, the total amount of ferrous debris is designed to measure quantitatively in parts per million (ppm). Upon receipt of the sample, it is the first test performed on the grease.

7.6 *Linear Sweep Voltammetry*—Linear sweep voltammetry measures the amount of hindered phenols, amines, and zinc dithio dialkyl phosphate (ZDDP) in a lubricating grease. Using 0.25 g of grease from the substrate, one substrate segment is inserted into a vial and the antioxidant concentration remaining in the grease is measured and compared to an unused sample of the same grease.

7.7 *Grease Colorimetry*—The grease colorimetry test provides a spectrum in the 400 nm to 700 nm region of the visible light spectrum. An optical spectroscopy cell is used and the substrate segment is placed into the same holder. The delta-E values within the CIE LAB scale are recorded and a spectrum can be generated in a method similar to Test Method D7843.

7.8 *Direct Imaging System*—A particle sizing and identification component consisting of a CCD (charge-coupled device) video chip, pulsed diode backlight, magnifying lens, fine focus dial, software, and readout system.

[4] Wurzbach, R. and Williams, L., "Die Extrusion Method For Comparing Changes in Grease Consistency and Flow Characteristics," *Viscosity and Rheology of In-Service Fluids as They Pertain to Condition Monitoring, ASTM STP 1564*, 2013, ASTM International.

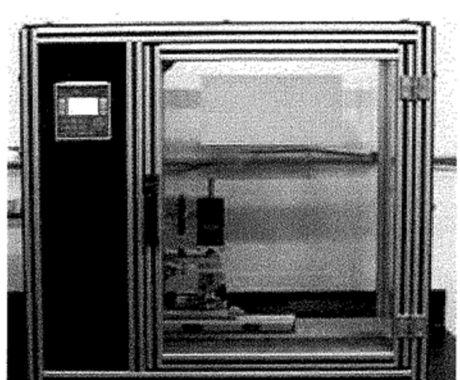

FIG. 1 Die Extrusion Tester[4]

7.9 *Relative Humidity Sensor Analyzer*—The test specimens in the passive grease sampling device are extruded on the substrate of the die extrusion tester, to create an approximate 0.25 g thin-film deposition on a plastic substrate. One plastic substrate is weighed and placed in a clean, dry glass vial with a septum and sealing cap. The vial is placed into the heating chamber of the relative humidity sensor analyzer, and a dry carrier gas is introduced to transfer the moisture driven from the sample by the heating, and passed over the humidity sensor to calculate moisture content based on calculated total flow and original sample mass.

8. Reagents and Materials

8.1 *Spring Cell Calibration Cylinder*—This device is used to perform a calibration check on the die extrusion tester prior to use. See Annex A3 for the full calibration procedure.

8.2 *Indexing Die*—Threads onto the end of the passive grease sampling device prior to performing the die extrusion test.

8.3 *Substrate*—The three-layered plastic substrate shall be long enough to hold the grease ribbon once it is extruded from the indexing die. The substrate's three layers consist of a rigid bottom layer of high-density polyethylene, a double-sided releasable adhesive, and a top layer of low-density polyethylene. The top layer of the substrate is divided into peel-away segments that, when fully covered with the grease ribbon, each contains approximately 0.25 g of grease.

9. Sampling

9.1 Good grease-sampling procedures are critical to good analyses, and samples should be taken in accordance with Practice D7718.

10. Preparation of Test Specimen

10.1 *Ferrous Debris Analyzer*—Prior to analysis, the passive grease sampling tool shall be removed from the storage container. The passive grease sampling tool shall be placed into the ferrous debris analyzer to determine the total amount of ferrous wear present (in ppm).

10.2 *Passive Grease Sampling Tool*—Upon completion of the ferrous debris analyzer test, the protective cap shall be removed and an indexing die shall be threaded onto the open end of the passive grease sampling device.

NOTE 2—If the in-service grease sample is received in an active grease sampling device, the stinger probe must be removed and replaced with the handle of a passive grease sampling device prior to use.

NOTE 3—If the in-service grease sample is received in any sampling container other than the passive or active grease sampling device, it must be transferred into a passive grease sampling device by hand, with a clean, single-use glove and/or a clean, single-use 10 mL slip tip syringe.

11. Preparation of Apparatus

11.1 *Preparation of the Die Extrusion Tester:*

11.1.1 *Heating the Chamber*—Prior to the performance of any tests, a thermocouple is used to regulate the temperature of the heating tray to achieve a temperature sufficient to pre-heat samples to meet the test temperature requirements for the trial specified in 12.3.3. The samples shall be on the heating tray for at least 20 min to come to thermal equilibrium, which is verified with an embedded thermocouple.

11.1.2 *Validation Check*—A check shall be performed with a spring cell calibration cylinder to confirm calibration prior to the analysis of routine samples. The procedure and accuracy guidelines for the validation check are described in Annex A3. This procedure shall be performed at the beginning of each shift or if the instrument has not been used for an extended period of time.

11.2 *Preparation of the Optical Spectroscopy Cell*—Check the mirror, optical path, and the tray for cleanliness.

11.3 This test method requires no additional preparation steps for the other instruments.

12. Testing Procedure

12.1 *Procedure for Evaluation of Ferrous Debris Level:*

12.1.1 *Factory Calibration*—The parameters for the analytical instrumentation are pre-set by the manufacturer of the equipment. Daily validation checks must be performed and analyses of test specimens must be within the linear range of response.

12.1.2 *Routine Standardization*—A minimum two-point routine standardization shall be performed if the instrument fails the validation check.

12.1.3 Set the instrument to grease mode and drop the sample into the coil.

12.1.4 Record the results in the nearest whole number in ppm ferrous content.

12.2 *Analysis of Grease Samples by Die Extrusion:*[4]

12.2.1 *Factory Calibration*—The parameters for the analytical instrumentation are pre-set by the manufacturer of the equipment. A calibration curve must be determined prior to use to calculate a K constant and an offset for the reference spring cell. Daily validation checks must be performed and results must be within the linear range of response.

12.2.2 *Routine Standardization*—A minimum two-point routine standardization shall be performed if the instrument fails the validation check. The procedure for the routine standardization is described in Annex A4.

12.3 *Steps for Standard and In-service Sample Preparation Procedure:*

12.3.1 Load a substrate, with the segments facing up.

12.3.2 Upon preparation of the sample, move the heated passive grease sampling device from the heating tray and immediately place into the sample-holding bracket with the opening of the indexing die facing the same direction that the table moves. A new, clean indexing die and substrate shall be used for each analysis.

12.3.3 The die extrusion tester shall be programmed to only perform the test if the sample is in the appropriate temperature range. Therefore, if the sample loses too much heat in the loading process, it will need to be reheated. The appropriate temperature is verified using a non-contact thermometer that has been modeled and calibrated by the manufacturers of the instrument to ensure internal grease temperature of 30 °C ± 2 °C.

12.3.4 The die extrusion tester shall be programmed to run the following steps:

12.3.4.1 The prime must be set so that it allows sufficient time for the die to be filled. Grease shall be exiting the die before continuing to Rate 1.

12.3.4.2 Rate 1—0.15 mm/s ± 0.01 mm/s for 19 s ± 1 s.

12.3.4.3 Rate 2—0.73 mm/s ± 0.04 mm/s for 13 s ± 1 s.

12.3.4.4 Rate 3—0.44 mm/s ± 0.02 mm/s for 22 s ± 1 s.

12.3.5 Start the program, which will extrude the lubricating grease onto the substrate and collect the flow response data. This methodology is good for grades 1, 2, and 3 greases. Consult the vendor manual for how to run grades 00 and 0.

NOTE 4—Die extrusion testing only uses the speeds that show a force response in the final calculation of the die extrusion index. The passive grease sampling device must be at least 70 % full to run the die extrusion test.

12.3.6 Verify during the prime and prior to Rate 1 step that the load data shows an increase from the start load. Inspect the piston for movement. Its movement should correspond to an increase in the load cell reading. If no change of the load cell reading is observed during the prime while the piston is moving, immediately terminate the test by stopping the instrument. If the load cell displays a zero reading during extrusion, it could be an indication of misalignment or instrument malfunction. Return to 12.3.1 and repeat the test until a satisfactory validation step is achieved.

12.3.7 Once the test is complete, the data shall be transferred to a computer to be analyzed. For calculations see Section 13.

12.3.8 Remove the empty passive grease sampling device from the die extrusion tester and dispose in the trash.

12.3.9 Remove the substrate containing the grease ribbon for additional testing.

12.4 *Procedure for Grease Colorimetry:*

12.4.1 *General Test Procedure*—The test procedure will consist of an unused grease reference reading and the sample (in-service grease) reading.

12.4.2 Prepare the grease according to 12.3.

12.4.3 *Calibration of the Spectrophotometer:*

12.4.3.1 Place the blank substrate segment into the drawer of the optical spectroscopy cell to collect the background spectrum.

12.4.3.2 Attach the surface head to the cell so that it is perpendicular to the substrate (or the separator is parallel to the side of the cell containing the drawer).

12.4.3.3 Turn the i-Lab on using the power button in the center of the unit.

12.4.3.4 Using the arrow keys, highlight GreaseBk and press the power button to select.

12.4.3.5 Remove the protective cap from the i-Lab and place it onto surface head, which should already be attached to the cell.

12.4.3.6 Press the Power button again to obtain the background spectrum.

12.4.3.7 Remove the unit from the cell.

12.4.4 *Procedure for Sample Data Collection:*

12.4.4.1 Using the arrow keys, select the GreaseCIE method and press power.

12.4.4.2 Invert the unit back onto the surface head and press power again to obtain the spectrum of the sample.

12.4.4.3 Repeat test as needed for multiple samples.

12.4.5 *Procedure for Linear Sweep Voltammetry:*

12.4.5.1 Prepare the grease sample according to 12.3.

12.4.5.2 Add one substrate segment with 0.25 g of grease to the vial and follow the procedure outlined in section 8.5.1 in Test Method D7527 to perform a linear sweep voltammetry test.

NOTE 5—Step 8.5.1.3 in Test Method D7527 shall be replaced with a single grease strip from the substrate; instead of smearing the grease evenly on the inside of the 7 mL vial, the user shall take one full strip of the grease substrate and curl it to fit inside the 7 mL vial. User may proceed following the remainder of the steps in section 8.5.1 in Test Method D7527.

12.5 *Analysis of Particle Concentration by Direct Imaging:*

12.5.1 For each sample, the grease is extruded in a thin film and collected on a translucent polyethylene substrate as described in 12.3.

12.5.2 The linear actuator that controls the moving table the plastic substrate sits upon is used to parade the grease ribbon transversely across the fixed view of the high-resolution camera.

12.5.3 The grease ribbon width shall be equal to or greater than the frame width of the camera, and the length large enough to capture a sufficient number of unique non-overlapping frames during video analysis.

12.5.4 Prior to analysis, the backlighting is adjusted to provide a consistent average background intensity between samples even as grease color and opacity may vary.

12.5.5 The focus is also adjusted as necessary for each sample, with a fine vertical position dial.

12.5.6 The camera is used to capture a video recording of the grease ribbon as it passes.

12.5.7 The travel speed of the table is set to maximize the number of frames to be collected during video recording, while minimizing overall test duration.

12.5.8 The video recording is then processed with particle sizing and counting software to determine the average particle area per frame in the sample.

12.5.9 Out-of-focus frames, or frames that are otherwise compromised by grease distribution inconsistency, are excluded from the count.

12.5.10 The results in μm^2 are correlated to mg/g values in three different size ranges (>10 µm, >25 µm, >80 µm) with the calibration curve.

12.6 *Procedure for Moisture Analysis Using Relative Humidity Sensor:*

12.6.1 *Instrument Calibration*—The calibration of this device is performed by the manufacturer according to the device operating procedures and adjustments to the mass flow sensor and relative humidity sensor outputs. Verification of the adequacy of instrument calibration is performed using a precision capillary pipette with distilled water, as is described in Test Method D7546.

12.6.2 *Calibration Check*—The standardization check of this device is achieved by filling a precision capillary pipette with distilled water, and verifying the tared mass of the water.

The procedure for the routine standardization is described in Section 11 of Test Method D7546.

12.7 *Steps for Standard and In-service Sample Preparation Procedure:*

12.7.1 Prepare the grease according to 12.3.

12.7.2 Remove the substrate with the grease ribbon from the die extrusion tester.

12.7.3 Remove one full center section of the substrate by grasping the edges with a gloved hand and separating from the adhesive backing, being sure not to contact the grease.

12.7.4 Determine the mass of the substrate section and grease, and subtract the average tare value of an empty substrate section.

12.7.5 Place the substrate with the extruded grease face up in a clean, dry glass vial, of the type designed for use in the Vapor Pro instrument.

12.7.6 Fasten a clean septum and bottle cap securely, and place vial on the bottle holder with the substrate at the bottom side of the vial.

12.7.7 Prepare the instrument with the following settings:

12.7.7.1 The "grease" method profile shall have a set temperature of 175 °C.

12.7.7.2 The ending criteria shall be a minimum of 2 min run time and the moisture rate shall be less than 0.45 µg/s, decreasing.

12.7.8 When setup is complete, begin the test profile.

NOTE 6—If the unit is not at the correct temperature, it will give you an error and start acclimating to the correct temperature. Once the correct temperature is reached, repeat the prior step again.

12.7.9 Watch the rate curve and verify that the moisture loss rate increases to a singular maximum height and then decreases until the conclusion of the test. If the moisture rate does not conform to this profile, the test should be re-run. A grease sample that fails to follow this profile may need an alternate heating profile, and a new strip must be used to restart test once new parameters are determined.

12.7.10 As suggested by the manufacturer, a lower test temperature may be needed if the graph doesn't follow the described profile.

12.7.11 Repeat test as needed for multiple samples.

13. Calculations

13.1 *Die Extrusion Test:*

13.1.1 The flow response data obtained from the die extrusion tester shall be transferred onto a computer for calculation of the die extrusion index for the used or in-service grease sample.

13.1.2 To calculate the die extrusion index, take the average stable die extrusion load obtained for each speed, divide by the value obtained for the baseline, average over the three speeds, and then multiply by 100. The calculation can be seen below in Eq 1.

$$\text{Die Extrusion Index} = \frac{\left[\left(\frac{\text{Load } 1_s}{\text{Load } 1_b} \times 100\right) + \left(\frac{\text{Load } 2_s}{\text{Load } 2_b} \times 100\right) + \left(\frac{\text{Load } 3_s}{\text{Load } 3_b} \times 100\right)\right]}{3} \quad (1)$$

NOTE 7—Load x_s represents the average load for speed x of the sample and Load x_b represents the average load for speed x of the baseline.

NOTE 8—In instances where the force readings may have spiked or dropped due to large particles or air bubbles, the data should be modified to exclude these spikes or drops in the final calculations for the die extrusion index.

NOTE 9—There is software available to perform these calculations, which is provided with the purchase of the die extrusion tester.

13.2 *Ferrous Debris Level:*

13.2.1 Number is calculated and reported as a whole number ppm value.

NOTE 10—For cases where the passive grease sampling device is not completely filled, the passive grease sampling device will be tared and weighed to compensate for the low volume sample. A mass compensation calculation will be done to normalize the results for fdM+.

13.3 *Linear Sweep Voltammetry:*

13.3.1 Calculations shall be performed following Test Method D7527.

13.4 *Grease Colorimetry:*

13.4.1 A series of transmittance values are gathered from 400 nm to 700 nm on a handheld spectrophotometer. The data shall be graphed using *x-y* smooth curve plot. On the *x*-axis shall be wavelength and on the *y*-axis shall be transmittance.

14. Report

14.1 The report shall include the die extrusion load 1_s, load 2_s, and load 3_s. The load values shall be rounded to the nearest whole number value. The die extrusion index shall be reported using rounded whole number values only. The die extrusion index for the baseline grease shall be assigned a value of 100.

NOTE 11—The results of the test depend on the choice of an appropriate baseline sample or specimen for reference. Typical acceptable references include unused grease of the same type that the in-service sample is suspected to be. It is the responsibility of the analyst to decide which baseline to choose so the comparison data provides the most meaningful results.

14.2 The load data obtained from the die extrusion test shall be graphed as a function of time as the ribbon is extruded from the die. The baseline graph and sample graph shall overlay each other to demonstrate the changes in flow properties. On the *x*-axis shall be time and the *y*-axis shall be in grams. The integer values in grams for speed 1, speed 2, speed 3, and the calculated die extrusion index shall also be reported for the sample and baseline on the report.

14.3 Ferrous debris level, linear sweep voltammetry, and grease colorimetry data are gathered and reported as graphs and numbers on a grease analysis report that evaluates overall condition of the grease in four areas: wear, consistency, contamination, and oxidation. The data gathered is reported as part of a comprehensive condition based maintenance program.

15. Precision and Bias

15.1 The precision of this test method is based on an interlaboratory study of Test Method D7918, conducted in 2014. Two operators tested six different types of grease at three loads and calculated GTI based on obtained load values. Every "test result" represents an individual determination, and all participants were instructed to report ten replicate test results per material. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. RR:D02-1802.[5]

15.1.1 *Repeatability (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

15.1.1.1 Repeatability can be interpreted as maximum difference between two results, obtained under repeatability conditions, that are accepted as plausible due to random causes under normal and correct operation of the test method.

15.1.1.2 Repeatability limits are listed in Tables 1-4.

15.1.2 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus 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 one case in 20.

15.1.2.1 Reproducibility can be interpreted as maximum difference between two results, obtained under reproducibility conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

15.1.2.2 Reproducibility could not be determined from the limited amount of data collected.

15.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

15.1.4 Any judgment in accordance with statements 15.1.1 and 15.1.2 would have an approximate 95 % probability of being correct.

15.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

15.3 The precision statement was determined through statistical examination of all 420 reported results, from two operators, on six different greases. These six materials were described as the following:

(1) Sample Type 1: NLGI 1
(2) Sample Type 2: NLGI 2
(3) Sample Type 3: NLGI 3
(4) Sample Type 4: 50/50 mixture lithium complex thickener: polyurea thickener
(5) Sample Type 5: copper particulate concentration
(6) Sample Type 6: worked sample

15.4 To judge the equivalency of two test results, it is recommended to choose the material closest in characteristics to the test material.

16. Keywords

16.1 consistency; die extrusion index; die extrusion tester; ferrous debris level; grease colorimetry; in-service lubricating grease sampling; linear sweep voltammetry; lubricating grease; lubricating grease sampling device

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1802. Contact ASTM Customer Service at service@astm.org.

TABLE 1 Load 1 (grams)

Material	Average[A]	Repeatability Standard Deviation	Repeatability Limit
	$\bar{x}$	s_r	r
Sample Type 1	300.55	23.73	66.43
Sample Type 2	537.65	44.32	124.08
Sample Type 3	822.25	57.29	160.42
Sample Type 4	497.55	20.23	56.63
Sample Type 5	514.55	66.95	187.47
Sample Type 6	485.60	33.75	94.50

[A] The average of the laboratories' calculated averages.

TABLE 2 Load 2 (grams)

Material	Average[A]	Repeatability Standard Deviation	Repeatability Limit
	$\bar{x}$	s_r	r
Sample Type 1	365.05	27.19	76.13
Sample Type 2	687.30	50.40	141.12
Sample Type 3	1069.90	58.69	164.33
Sample Type 4	651.70	19.85	55.58
Sample Type 5	702.45	35.09	98.25
Sample Type 6	618.80	24.92	69.77

[A] The average of the laboratories' calculated averages.

TABLE 3 Load 3 (grams)

Material	Average[A]	Repeatability Standard Deviation	Repeatability Limit
	$\bar{x}$	s_r	r
Sample Type 1	300.6	30.6	85.6
Sample Type 2	582.3	50.1	140.3
Sample Type 3	927.2	71.1	199.2
Sample Type 4	562.9	21.5	60.3
Sample Type 5	586.4	37.1	103.9
Sample Type 6	526.4	34.6	96.7

[A] The average of the laboratories' calculated averages.

TABLE 4 GTI (dimensionless)

Material	Average[A]	Repeatability Standard Deviation	Repeatability Limit
	$\bar{x}$	s_r	r
Sample Type 4	97.5	3.3	9.2
Sample Type 5	102.7	6.2	17.4
Sample Type 6	92.9	5.1	14.4

[A] The average of the laboratories' calculated averages.

ANNEXES

(Mandatory Information)

A1. GREASE THIEF TYPE 1

A1.1 This is a cylinder with a ⅛ in. national pipe thread at either end of a precision bore cylinder. Inside the cylinder is a precision-molded piston with a handle extending out the back of the piston that allows for movement of the piston. There is a pair of relief holes on the cylinder for excess lubricating grease to purge when the cylinder becomes full of sample. There are molded barbs in the handle that prevent the piston and handle from sliding forward out of the cylinder. The Grease Thief Type 1 is depicted in Fig. A1.1.

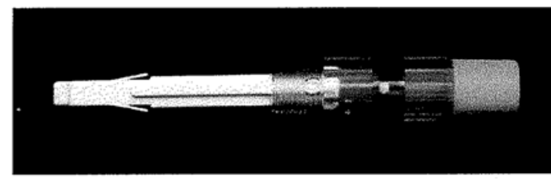

FIG. A1.1 Grease Thief Type 1

A2. GREASE THIEF TYPE 2

A2.1 This is a cylinder with a ⅛ in. national pipe thread at either end of a precision bore cylinder. There is a pair of relief holes at one end of the cylinder to allow for purging of excess lubricating grease. Inside of the cylinder is a precision-molded piston with a stinger probe the length of the cylinder minus the length of the piston. Also, the piston should have a handle that extends out the opposite end of the cylinder that allows the piston and probe assembly to be attached to an active grease-sampling device extension rod. The Grease Thief Type 2 is depicted in Fig. A2.1.

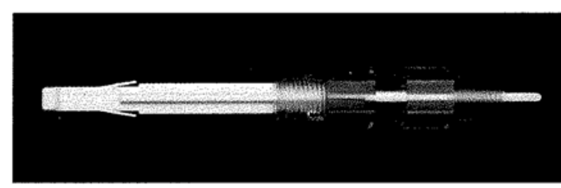

FIG. A2.1 Grease Thief Type 2

A3. VALIDATION CHECK

A3.1 Remove any objects from the substrate table, including the substrates.

A3.2 Place the spring cell calibration cylinder into the sample bracket.

A3.3 Run the instrument in calibration mode.

A3.3.1 Calibration mode consists of a test run at 0.25 mm/s for 70 s.

A3.4 Locate two data points sufficiently far apart and within the linear response of the spring cell calibration cylinder, datapoints X_1 and X_2. Their corresponding force values should lie within ±5 % deviation of the spring cell calibration curve provided with the spring cell following the form: $f_1(x) = A*x + B$, where A and B are constants, x is the datapoint the function is evaluated.

A4. ROUTINE STANDARDIZATION TO CALCULATE NEW LOAD CELL SCALE AND OFFSET

A4.1 Set the load cell scale (K) to 1 and the load cell offset to 0.

A4.2 Secure the spring cell calibration cylinder in the sample holding bracket.

A4.3 Run the instrument in calibration mode.

A4.4 Locate two data points sufficiently far apart and within the linear response of the spring cell calibration cylinder at datapoints X_1 and X_2.

A4.5 Create a load cell calibration curve following the form $f_2(x) = C*x + D$, by calculating the best fit linear plot between the selected points. If the R^2 value of the best fit to the entire dataset is less than 0.95, the spring cell may be compromised or the trial may need to be run again.

A4.6 The new load cell scale is found by the equation: K = A/C. The new offset is found by the equation: offset = $f_1(X_1)–f_2(X_1)*K$. Enter the K and offset value determined into the instrument.

A4.7 Following steps A4.1 – A4.6, determine if the instrument is within calibration.

A4.8 If the calibration procedure fails twice, the load cell may be damaged or the instrument may require factory calibration.

SUMMARY OF CHANGES

Subcommittee D02.96 has identified the location of selected changes to this standard since the last issue (D7918 – 15) that may impact the use of this standard. (Approved May 1, 2017.)

(1) Added new subsections 7.8 and 7.9.

(2) Added new subsections 12.5, 12.6, and 12.7.