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# 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.*

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

D217 Test Methods for Cone Penetration of Lubricating Grease
D4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants
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
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.

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**

[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.

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 are 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.

## 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

[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.

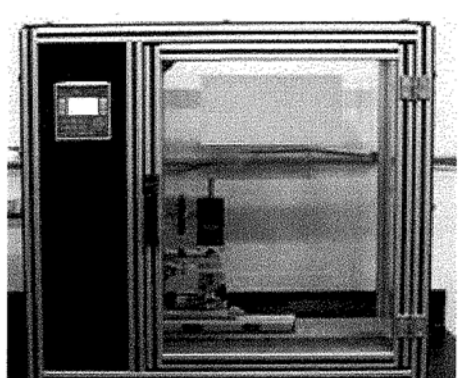

FIG. 1 Die Extrusion Tester[4]

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