Designation: D8120 – 17

Standard Test Method for Ferrous Debris Quantification[1]

This standard is issued under the fixed designation D8120; 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 describes a means for quantitative determination of the concentration of ferrous debris in lubricants and greases.

1.2 This test method provides a determination of the concentration of ferrous debris in the lubricant or grease from a nominal 0 mg/kg to 10 000 mg/kg (1 % by mass) or greater.

1.3 This test method is applicable to all types of lubricating fluids (API Group I-V) and greases sampled from machinery and other mechanical equipment, including reciprocating engine oils, turbine oils, hydraulic oils, gear oils, and bearing greases.

1.4 This test method describes a means by which a sample of lubricant or grease is placed in a magnetometer apparatus, which determines the concentration of ferrous debris and provides these readings directly to the operator without further calculation.

1.5 This test method is applicable to in-service lubricants and greases at any stage of degradation.

1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7 *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.*

1.8 *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]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D5185 Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

D5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products

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

D7669 Guide for Practical Lubricant Condition Data Trend Analysis

D7690 Practice for Microscopic Characterization of Particles from In-Service Lubricants by Analytical Ferrography

D7718 Practice for Obtaining In-Service Samples of Lubricating Grease

D7720 Guide for Statistically Evaluating Measurand Alarm Limits when Using Oil Analysis to Monitor Equipment and Oil for Fitness and Contamination

D7874 Guide for Applying Failure Mode and Effect Analysis (FMEA) to In-Service Lubricant Testing

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *ferrous debris concentration, n*—the concentration, expressed as milligrams per kilogram or percentage (percent by mass) of ferrous debris in a sample of lubricant across all particle size ranges of the debris, from dissolved to large ferrous debris particles in the millimeter size range. Note that such ferrous debris may exist in a matrix in the lubricant such as when organically combined or as part of a steel alloy.

[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 D02.96.06 on Practices and Techniques for Prediction and Determination of Microscopic Wear and Wear-related Properties.

Current edition approved June 1, 2017. Published July 2017. DOI: 10.1520/D8120-17.

[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.

3.1.2 *sample, n*—a portion of in-service oil or grease drawn directly from the machinery without further processing.

4. Summary of Test Method

4.1 A sample of in-service lubricant or grease is taken from the machinery or equipment and transferred into a suitable measurement receptacle. The manufacturer's recommendations should be followed for preferred holders or receptacles but typical container formats include sample bottles, syringes, and grease samplers. The sample holder is then manually placed inside an indicated empty measurement chamber of a magnetometer apparatus. Some instruments may require the sample holder to be removed before the measurement is complete and will prompt accordingly. By measuring the difference in signal between the filled chamber and the empty chamber, the concentration of ferrous debris in milligrams per kilogram or percent by mass is determined.

5. Significance and Use

5.1 By quantifying the concentration of total ferrous debris, this test method provides a direct indication of wear in the machinery by enabling the user to pinpoint when there is a deviation from the normal buildup of ferrous debris shed by the machinery or when the concentration of ferrous debris has exceeded safe operating limits. Specific guidance regarding such procedures may be found in Guides D7669, D7720, and D7874.

5.2 This test method can be performed on-site and can be utilized as a particle-size insensitive, minimum sample preparation alternative to laboratory-based screening for abnormal machinery conditions due to the presence of wear debris by means of ferrography as described in Practice D7690, or elemental analysis methods such as atomic emission spectrometry, described in Test Methods D5185 and D6595.

6. Interferences

6.1 This test method provides a measure of the concentration of ferrous debris by measuring the magnetic susceptibility of the sample under test. Non-ferrous debris, which is ferromagnetic (such as nickel or cobalt), may be reported as ferrous debris using this test method.

7. Apparatus

7.1 The core apparatus consists of a solenoid-style magnetometer with an inner coil diameter of sufficient size to accommodate the sample holder. Standard in-service oil analysis sample bottles (2 oz capacity), syringes (3 mL capacity), and grease pots (5 mL capacity) serve as sample holders. Other sample holders can be accommodated by sleeve adaptors made of non-magnetic materials. The user should consult the manufacturer's literature for guidance in the selection and use of suitable containers for the proposed sampling volumes. An exciting AC magnetic field, with typical frequencies in the range of a few kHz to several tens of kHz and with a peak amplitude in the 0.001 T region, is applied to the lubricant under test. The induced current in either a separate sensing coil, or the excitation coil itself, due to the presence of the ferrous debris is measured. From this induced current, the magnetic susceptibility of the lubricant is determined. This magnetic susceptibility is then correlated to the concentration of ferrous debris in the lubricant by means of the known magnetic susceptibility of such debris.

8. Reagents and Materials

8.1 A disposable, plastic sample container of sufficient capacity to perform the measurement. Consult the instrument manufacturer's instructions for the recommended containers and method of sample presentation.

8.2 At least two calibration standards of uniformly suspended ferrous material that provide equivalent magnetic susceptibilities within the lower and upper quartiles of the selected measurement range. These standards may consistent of, for example, suspended M50 bearing steel in oil or a resin matrix and are to be used as check standards for the purposes of confidence testing. An optional third standard for the mid-range point is useful but not essential. Again, consult the manufacturer's instructions for recommended practices regarding (re)calibration and check intervals.

8.3 A lint-free, oil-absorbent material should be used to clean the apparatus. Some suitable examples would be polypropylene industrial wipes or clean cotton shop rag.

9. Hazards

9.1 All precautions in the sampling and handling of in-service lubricant samples should be followed as appropriate. Please see Practices D4057, D4177, or D7718 for guidance on this point.

10. Sampling, Test Specimens, and Test Units

10.1 A sample of the liquid or grease should be obtained following the guidelines described in Practices D4057, D4177, or D7718. A representative portion of the lubricant and of sufficient volume to fill the measurement bottle, syringe, and so forth should be collected in a clean, dry container. Care should be taken to ensure that the portion collected is as representative of the in-service fluid or grease as possible. Avoid sampling from the bottom of sumps or other similar dead spots, where deposits may have built up over time. Consult the relevant ASTM practices noted above for more guidance on suitable sampling points. Also, check that the sample is at a temperature compatible with the specifications of the sample container before being drawn for analysis.

10.2 Inspect the collected portion for homogeneity and if in doubt, have it re-homogenized to ensure that a representative sample can be measured in the magnetometer. Follow Practice D5854 and ensure that the collected portion is gently inverted back and forth for at least 30 s before drawing or decanting the sample into the measurement syringe or vial before proceeding.

11. Preparation of Apparatus

11.1 Turn on the apparatus and ensure that no alerts, which may indicate faults with the measurement hardware or software, are generated.

11.2 Follow the manufacturer's instructions for the operation of the instrument.

12. Calibration and Standardization

12.1 Ferrous debris magnetometers are factory-calibrated and standardized to report concentrations in milligrams per kilogram or percent by mass in oil or grease. The calibration is linear across the specified calibration range for most typical applications. Deviations from linear behavior can occur if the sample contains large debris particles. The point at which this becomes significant depends on the magnetic field strength, the excitation frequency and the susceptibility of the debris particles. However for typical iron and steels this is of the order of a millimeter or so in diameter, at which point the debris is probably visible to the naked eye.

12.2 Note that the induced current caused by the debris is actually related to the mass of ferrous material present within the magnetic field. Wear debris magnetometers are designed so that the sensing volume is essentially defined by the coil construction for a given sample vial format. This sensing volume is independent of the overall volume of sample within the vial, and will be constant for any particular sample vial format. Therefore, providing that the sample volume within the vial exceeds that of the sensing volume, the induced current becomes directly related to the concentration of ferrous debris within the sample. During factory calibration, the response determined can be related to the known concentration of ferrous debris within the sample for a particular vial format. This procedure will be repeated for each style of sample vial that the instrument can accommodate. Before performing a measurement, it is important to choose the correct operational mode, if the magnetometer accepts different sample vial formats, in order to achieve consistent results.

12.3 If it is felt necessary to check the calibration of the apparatus, the manufacturer's supplied check standards should be used to perform confidence tests. The operator should follow the procedure detailed in the instructions and compare the results relative to the specified values indicated on the respective calibration standards. If these comparisons are out of specification, the measurements should be repeated. If any are still out of range, report the results to the manufacturer for further instructions.

13. Conditioning

13.1 The apparatus should be sited away from any significant sources of magnetic interference (for example, transformers, power supplies, and so forth). Also, it is advisable to avoid standing the instrument on steel workbenches or similar.

13.2 The apparatus should be switched on and allowed to warm up for at least 15 min.

13.3 Consult the manufacturer's instructions for any further conditioning steps.

14. Procedure

14.1 Ensure that both the sample chamber and the sample vial are visibly clean of oil or grease. Remove any excess using the supplied cleaning materials.

14.2 If appropriate, verify that the apparatus is set to the expected measurement range of the lubricant under test. If the expected range is unknown, it may be prudent to start at a low range and repeat at successively higher ranges until a stable and creditable result is obtained.

14.3 Transfer the lubricant from the sampling bottle, using clean, disposable syringes or similar, to the preferred measurement container. Consult the manufacturer's instructions for the minimum quantity recommended for the type of measurement container to be used. Wipe off any spillage from the outside of the measurement container using the supplied cleaning material.

14.4 Initiate the measurement cycle by the appropriate means for the particular apparatus as described in the manufacturer's instructions.

14.5 Holding the sample vertically, insert the container in the measurement aperture when prompted.

14.6 After a short delay, remove the sample container when indicated by the apparatus.

14.7 After a further short period, the measured concentration will be displayed on the apparatus.

15. Calculation or Interpretation of Results

15.1 Upon completion of the procedure, the concentration of ferrous debris is automatically calculated by the apparatus.

15.2 If upon completion of the procedure, the concentration of ferrous debris falls outside the measurement range set on the apparatus, the operator may be alerted of that fact and may be prompted to perform the procedure again using an appropriate range.

15.3 If an error code is displayed by the apparatus upon completion of the procedure, follow the manufacturer's instructions on next steps. For example, if the sample was not inserted correctly, a warning will be displayed, no ferrous reading will be provided, and the operator will be prompted to repeat the procedure.

15.4 *Trending and Alarm Limits*—According to the particular maintenance practices employed by the user, this test method may be performed on a periodic basis. Based on knowledge of the lubricant and machinery, monitoring of the trending behavior of the reported ferrous debris concentration will facilitate the setting of appropriate alarm limits. Specific guidance regarding such procedures may be found in Guides D7669, D7720, and D7874.

16. Report

16.1 Total ferrous debris in either units of milligrams per kilogram to the nearest 1 mg/kg or in units of percent by mass to the nearest 0.01 % by mass.

16.2 Results are reported on the display screen of the apparatus once the procedure is completed and (optionally) written to a log file, which may be exported and utilized for further analysis according to the manufacturer's instructions.

17. Precision and Bias[3]

17.1 An interim precision statement of this test method has been determined by statistical examination of results on twelve in-service lubricant samples ranging in total ferrous debris from 4 mg⁄kg to 2745 mg/kg. These samples comprised a range of in-service hydraulic, gear, transmission, turbine, and diesel engine lubricants. They were run in duplicates by six different operators at six different locations. The determined temporary precision is as listed below. A full precision statement based on interlaboratory round-robin testing will be determined within the time parameters established by ASTM for such statements.

17.1.1 *Preliminary Repeatability*—The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

$$r = 0.6381(X+0.0001)^{0.6303} \quad (1)$$

where:

X = the mean of two results, in milligrams per kilogram of total ferrous debris.

18. Keywords

18.1 magnetometer; particle size; total ferrous debris

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1864. Contact ASTM Customer Service at service@astm.org.