Designation: D7596 – 23

Standard Test Method for Automatic Particle Counting and Particle Shape Classification of Oils Using a Direct Imaging Integrated Tester[1]

This standard is issued under the fixed designation D7596; 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 of particle concentration, particle size distribution, particle shape, and soot content for new and in-service oils used for lubrication and hydraulic systems by a direct imaging integrated tester.

1.1.1 The test method is applicable to petroleum and synthetic based fluids. Samples from 2 mm^2/s to 150 mm^2/s at 40 °C may be processed directly. Samples of greater viscosity may be processed after solvent dilution.

1.1.2 Particles measured are in the range from 4 µm to ≥ 70 µm with the upper limit dependent upon passing through a 100 µm mesh inlet screen.

1.1.3 Particle concentration measured may be as high as 5 000 000 particles per mL without significant coincidence error.

1.1.4 Particle shape is determined for particles greater than approximately 20 µm in length. Particles are categorized into the following categories: sliding, cutting, fatigue, nonmetallic, fibers, water droplets, and air bubbles.

1.1.5 Soot is determined up to approximately 1.5 % by weight.

1.1.6 This test method uses objects of known linear dimension for calibration.

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

1.3 *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.*

1.4 *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]

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D2896 Test Method for Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

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)

D5967 Test Method for Evaluation of Diesel Engine Oils in T-8 Diesel Engine

D6304 Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration

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

D7279 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids by Automated Houillon Viscometer

E2412 Practice for Condition Monitoring of In-Service Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry

[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.

Current edition approved Jan. 1, 2023. Published January 2023. Originally approved in 2010. Last previous edition approved in 2014 as D7596 – 14. DOI: 10.1520/D7596-23.

[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.

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

G40 Terminology Relating to Wear and Erosion

2.2 *ISO Standards:*[3]

ISO 12103-1 1997 Road Vehicles—Test Dust for Filter Evaluation—Part 1: Arizona Test Dust

ISO 4406 Hydraulic Fluid Power—Fluids—Method for Coding Level of Contamination by Solid Particles

2.3 *SAE Standards:*[4]

SAE AS 4059 Aerospace Fluid Power – Cleanliness Classification for Hydraulic Fluids

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *contaminant particles, n*—particles introduced from an extraneous source into the lubricant of a machine or engine.

3.2.2 *direct imaging integrated tester, n*—an instrument for counting particles as they flow through a cell by means of imaging; instrument may also determine particle shape and fluid viscosity.

3.2.3 *ISO Codes, n*—standard method for coding the level of contamination by solid particles. This code simplifies the reporting of particle count data by converting the number of particles per mL into three classes covering ≥4 µm, ≥6 µm, and ≥14 µm. ISO 4406 classifications are used as an option to report results for this test method.

3.2.4 *new oil, n*—oil taken from the original manufacturer's packaging, prior to being added to the machinery. **E2412**

3.2.5 *particle size, circular diameter, µm, n*—diameter of a circle with an area equivalent to the projected area of a particle passing through the direct imaging integrated tester flow cell.

3.2.6 *soft particles, n*—particles present in the sample that are related to undissolved oil additives or additive by-products. Without dilution, at room temperature these particles are likely to be counted by an optical particle counter in a similar manner to dirt and wear metal particles, air bubbles, and free water droplets. They are not considered contaminants as they are either purposefully left undissolved, or are not harmful to the fluid system, or both.

3.2.7 *soot, n—in internal combustion engines*, sub-micron size particles, primarily carbon, created in the combustion chamber as products of incomplete combustion. **D4175**

3.2.8 *wear, n*—damage to a solid surface, usually involving progressive loss or displacement of material, due to relative motion between that surface and a contacting substance or substances. **D4175, G40**

3.2.9 *wear particles, n*—particles generated from wearing surfaces of a machine or engine.

4. Summary of Test Method

4.1 Lubricant samples are acquired periodically from a machine or engine being monitored. Samples are taken using clean receptacles in order to avoid altering the sample by method or container.

4.2 Particles are counted and sized by processing a sample through an appropriate particle sizing instrument. Sample size is instrument dependent. The instrument determines the size and shape of each particle detected in the sample as described in Section 1 of this test method. Adjustable cell gap instruments are set at a fixed gap width that allows for comprehensive analysis. Gap of 100 µm to 300 µm is a common distance, however instruments may vary and other gap distances may be employed as long as there is no restriction of particle flow into the measurement zone. See Fig. 1.

4.3 The instrument calculates the shape of all particles ≥20 µm in size. The instrument software sorts particles into the following categories: cutting, fatigue, severe sliding, nonmetallic, fibers. Air bubbles and water droplets ≥20 µm must be eliminated from the particle counting results by analysis or treatment. Further information regarding wear particle shape recognition may be found in Anderson' report.[5]

4.4 Nonmetallic particles are recognized by their partial transparency. Nonmetallic particles, in thin sections, do not block light, as do metallic particles. Therefore, particles displaying transparent interior pixels are classified as nonmetallic. Nontransparent particles are sorted into one of three metallic categories, namely, cutting, sliding, and fatigue.

4.5 Cutting wear particles are recognized by their elongated, curved, or curly shape.

4.6 Sliding wear particles are recognized by being longer than wide, often with straight edges.

4.7 Fatigue particles are recognized by being more or less as long as they are wide and often with jagged, irregular edges.

4.8 Fibers are recognized by their elongated shape and by partial transparency indicating nonmetallic composition.

4.9 Air bubbles are dark round circles, either completely dark or with small bright centers.

4.10 Water droplets are dark round circles with large bright centers. The difference in appearance between air bubbles and water droplets is due to the much different refractive index of each. When present in oil, air bubbles refract much of the light passing through them away from the direction of transmission, whereas water droplets, having a refractive index more nearly equal to that of oil, allow much of the light incident upon them to transmit through them to the CCD video chip.

4.11 Soot is measured by performing an optical extinction measurement with reference to new oil. Absorbance of light is measured and calibration is made to diesel engine oil samples

[3] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.

[4] Available from SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://aerospace.sae.org.

[5] Anderson, D., *Wear Particle Atlas (Revised)*, Prepared for Advanced Technology Office, Support Equipment Engineering Department, Naval Air Engineering Center, Lakehurst, NJ, 08733, 28 June 1982, Report NAEC – 92 – 163, approved for public release, distribution unlimited.