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INTERNATIONAL

Designation: ~~D7596 – 10~~ D7596 – 14

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~~ 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 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~~ 70 µm with the upper limit dependent upon passing through a ~~100 µm~~ 100 µm mesh inlet screen.

1.1.3 Particle concentration measured may be as high as ~~5,000,000~~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.

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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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

ASTM D7596-14

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, Petroleum Products, 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~~ Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.96 on In-Service Lubricant Testing and Condition Monitoring Services.

Current edition approved ~~May 1, 2010~~June 1, 2014. Published ~~August 2010~~July 2014. Originally approved in 2010. Last previous edition approved in 2010 as D7596 – 10. DOI: ~~10.1520/D7596-10.~~10.1520/D7596-14.

[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**

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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 dustVehicles—Test Dust for Filter Evaluation—Part 1: Arizona Test Dust

ISO 4406 Hydraulic fluid power – Fluids – Method for coding level of contamination by solid particlesFluid 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 of Terms Specific to This Standard:*

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

3.1.2 *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.

3.1.3 *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 ≥4 µm, ≥6 µm, and ≥ 14 ≥14 µm. ISO 4406 classifications are used as an option to report results for this test method.

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

3.1.5 *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.

3.1.6 *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.

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

3.1.8 *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**

3.1.9 *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 drawing oil through a flow cell. Seeprocessing a sample through an Fig. 1. The cell is illuminated by a pulsed laser. The duration of the pulse is sufficiently fast to freeze the motion of the particles in the cell. The pulse frequency is 30appropriate 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 Hz. Images of the particles flowing through the cell are magnified by 4 using a lens between the cell and the CCD video chip onto which the images of the particles are focused. Software counts and sizes each particle. Sizing is done by comparison to objects of known linear dimension. The number of particles per mL is determined by dividing particle counts by the volume of oil examined. Each image taken corresponds to a small volume of oil equal to the image area, which is 1600 × 1200 µm, multiplied by the cell thickness, nominally 100 µm. The actual cell thickness, to the closest µm for each cell, is provided by the manufacturer and is entered into the software for theof this test method. Adjustable cell gap instruments are set at a fixed gap width that allows for comprehensive analysis. Gap of 100 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. 1purpose of calculating the volume of oil examined for each sample. The total oil volume examined is the volume per image multiplied by the number of images collected..

4.3 The direct imaging integrated tester software performs particle shape recognition instrument calculates the shape of all particles ≥ 20 µm by using a neural network. An algorithm ≥20 µm in size. The instrument software sorts particles into the following categories: cutting, fatigue, severe sliding, nonmetallic, fibers, air bubbles and water droplets. fibers. Air bubbles and

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[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.

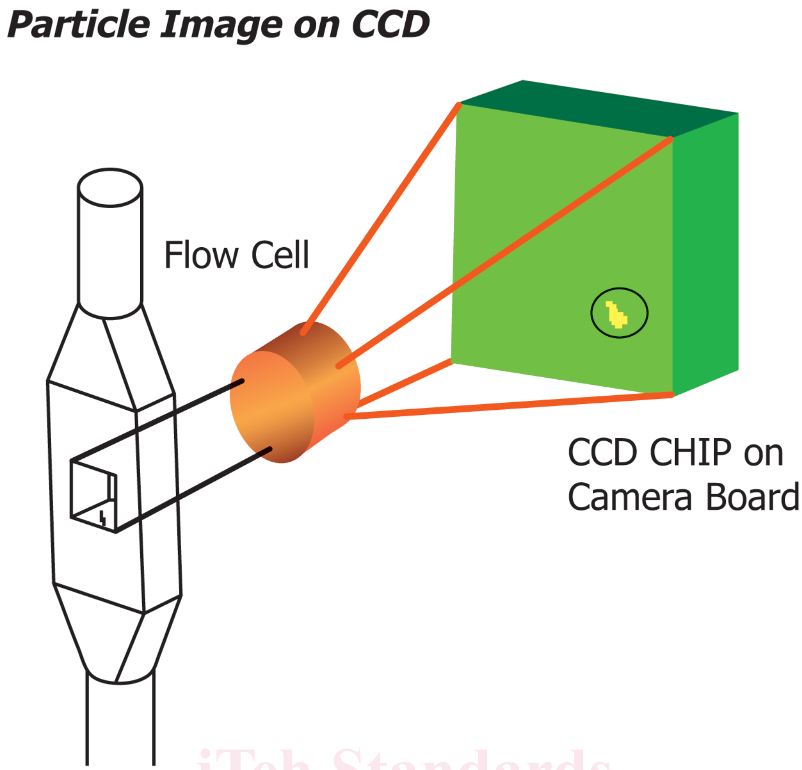

FIG. 1 Schematic of Direct Imaging Integrated ~~Tester~~Tester

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water droplets ~~≥ 20 µm are~~ ≥20 µm must be eliminated from the particle counting ~~results.~~ results by analysis or treatment. Further information regarding wear particle shape recognition may be found in ~~Anderson's~~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 ~~the laser~~ light is ~~calculated~~measured and calibration is made to diesel engine oil samples with known percentage of soot as determined by thermal gravimetric analysis in accordance with Test Method D5967, Annex A4.

4.12 Condition alerts and alarms, based on trend and level, can be issued for the system being monitored according to particle count, size distribution, types of particles recognized and soot content.

5. Significance and Use

5.1 This test method is intended for use in analytical laboratories including on-site in-service oil analysis laboratories. Periodic sampling and analysis of lubricants have long been used as a means to determine overall machinery health. Atomic emission

[5] ~~Anderson, D.P., 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.~~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.

spectroscopy (AES) is often employed for wear metal analysis (Test Methods D5185 and D6595). A number of physical property tests complement wear metal analysis and are used to provide information on lubricant condition (Test Methods D445, D2896, D6304, and D7279). Molecular spectroscopy (Practice E2412) provides direct information on molecular species of interest including additives, lubricant degradation products and contaminating fluids such as water, fuel and glycol. The direct Direct imaging integrated tester provides testers provide complementary information on particle count, particle size, particle type, and soot content.

5.2 Particles in lubricating and hydraulic oils are detrimental because they increase wear, clog filters and accelerate oil degradation.

5.3 Particle count may aid in assessing the capability of a filtration system to clean the fluid, determine if off-line recirculating filtration is needed to clean the fluid, or aid in the decision whether or not to change the fluid.

5.4 An increase in the concentration and size of wear particles is indicative of incipient failure or component change out. Predictive maintenance by oil analysis monitors the concentration and size of wear particles on a periodic basis to predict failure.

5.5 High soot levels in diesel engine lubricating oil may indicate abnormal engine operation.

6. Interferences

6.1 Dirty environmental conditions and poor handling techniques can easily contaminate the sample. Care must be taken to ensure test results are not biased by introduced particles.

6.2 Air bubbles < 20 µm may be counted as particles giving false positive readings. Air bubbles ≥ 20 µm are recognized and automatically eliminated from the count. Mixing or agitating the sample introduces air bubbles into the oil, but these readily dissipate with ultra-sonication or vacuum degassing.

6.3 Water droplets < 20 µm may be counted as particles giving false positive readings. If water droplets ≥ 20 µm are detected in a sample by the direct imaging integrated tester, there is reason to suspect water droplets < 20 µm are present and have spuriously increased particle count. Small amounts of water in the sample may be negated by the use of water masking solvent. See Appendix X1.

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6.4 Certain additives or additive by-products that are not fully dissolved in the oil, most notably polydimethylsiloxane defoamant additive, are known to be present as soft particles that are not contaminants in the fluid system, but are counted as particles by the direct imaging integrated tester. These may be negated by use of a diluting solvent. See Appendix X1.

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6.5 Samples with viscosity greater than approximately 150 mm150 mm^2/s at 40°C when processed by the direct imaging integrated tester at room temperature (approximately 20°C) may flow through the tester too slowly causing the same particle to be imaged twice. This effect may be negated by diluting the sample with clean solvent to lower viscosity. The tester software makes provision for input of the dilution factor so that particle counts are adjusted and reported for undiluted sample.

6.6 Soot levels above approximately 1.5 % by weight cause insufficient laser light to reach the CCD video detector. The software provides an error message and the sample may be diluted with clear, particle free oil and reprocessed. The tester software makes provision for input of the dilution factor so that particle counts are adjusted and reported for undiluted sample.

6.7 No correction for oil sample density or for soot density is made for the soot calculation.

6.8 High particle concentrations, in excess of approximately 5,000,000/mL, may cause reporting errors. The software provides an error message and the sample may be diluted with clear, particle free oil and reprocessed. The tester software makes provision for input of the dilution factor so that particle counts are adjusted and reported for undiluted sample.

6.9 The software categorizes particles into one of three possible metallic types. These are cutting, sliding or fatigue. The software was trained using particles generated by controlled wear modes under simulated conditions in a laboratory using wear testing equipment. Particles from actual in-service oil samples that appear similar in shape are classified into one of the direct imaging integrated tester's categories even though the nature of the particles may be different. For example, nonmetallic particles may be classified into one of the three metallic classes if the silhouette image captured by the CCD video camera contains no transparent interior pixels. The software assumes particles are nonmetallic if the image allows light to pass through interior pixels. Small, nonmetallic particles may deflect light so that they appear to be metallic. Fatigue platelets, generated by rolling contact fatigue, produce metallic platelets that are more or less as long as they are wide. There are, however, several other particle types with similar shape characteristics. These might be metallic sliding wear particles that are not long enough to be classified as sliding wear particles, or they may be something else entirely such as molybdenum disulfide flakes, coal dust particles, carbon seal material or dark metallo-oxides from sliding wear. Further examination of particles by other means may be necessary to determine their exact type.

6.10 Further investigation by microscopy, such as examination of particles on a membrane filter patch or analytical ferrography, may be necessary to better determine the exact particle types present.

6.10 A discrepancy may occur when comparing wear particle counts measured by a direct imaging integrated tester with the counts from a conventional automated light blockage particle counter. The metal particles, being solid, will block more light