Designation: ~~D7647 – 10 (Reapproved 2018)~~ D7647 – 24

Standard Test Method for Automatic Particle Counting of Lubricating and Hydraulic Fluids Using Dilution Techniques to Eliminate the Contribution of Water and Interfering Soft Particles by Light Extinction[1]

This standard is issued under the fixed designation D7647; 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 and particle size distribution in new and in-service oils used for lubrication and hydraulic purposes.

1.2 Particles considered are in the range from 4 μm $_{(c)}$ to 200 μm $_{(c)}$ with the upper limit being dependent on the specific automatic particle counter being used.

NOTE 1—For the purpose of this test method, water droplets not masked by the diluent procedure are detected as particles, and agglomerated particles are detected and reported as a single larger particle.

NOTE 2—The subscript$_{(c)}$ is used to denote that the apparatus has been calibrated in accordance with ISO 11171. This subscript$_{(c)}$ strictly only applies to particles up to 50 μm.

1.3 Lubricants that can be analyzed by this test method are categorized as petroleum products or synthetic based products, such as: polyalpha olefin, polyalkylene glycol, or phosphate ester. Applicable viscosity range is up to 1000 mm^2/s at 40 °C. This procedure may be appropriate for other petroleum and synthetic based lubricants not included in the precision statement.

1.4 Samples containing visible particles may not be suitable for analysis using this test method.

1.5 Samples that are opaque after dilution are not suitable for analysis using this test method.

1.6 The test method is specific to automatic particle counters that use the light extinction principle and are calibrated according to the latest revision of ISO 11171.

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

1.8 *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] 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 D02.96.05 on In-Service Lubricants Particle Counting Practices and Techniques.

Current edition approved ~~April 1, 2018~~Feb. 1, 2024. Published ~~May 2018~~February 2024. Originally approved in 2010. Last previous edition approved in ~~2010~~2018 as ~~D7647 – 10.~~D7647 – 10 (2018). DOI: ~~10.1520/D7647-10R18.~~ 10.1520/D7647-24.

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

1.9 *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
D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D6786 Test Method for Particle Count in Mineral Insulating Oil Using Automatic Optical Particle Counters

2.2 *ISO Standards:*[3]

ISO 3722 Hydraulic fluid power—Fluid sample containers—Qualifying and controlling cleaning methods
ISO 4406 Hydraulic fluid power—Fluids—Method for coding level of contamination by solid particles
ISO 11171 Hydraulic fluid power—Calibration of automatic particle counters for liquids

3. Terminology

3.1 *Definitions:*

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

3.1.2 *free water, n*—water in excess of that soluble in the sample and appearing in the sample as a haze or cloudiness, as droplets, or as a separated phase or layer.

3.1.3 *soot-in-oil, n—in internal combustion engines*, <1 µm size particles, primarily carbon, created in the combustion chamber as products of incomplete combustion.

3.2 ~~*Definitions:*~~*Definitions of Terms Specific to This Standard:*

~~3.1.1 For the purposes of this test method, the following definitions apply:~~

3.2.1 *coincidence, n*—the presence of more than one particle in the sensing zone of a particle analyzer at the same time, causing incorrect sizing and incorrect counting of the particle present. The coincidence limit of the counter is determined by the maximum acceptable concentration of particles in the sensing zone and is supplied by the instrument manufacturer. Refer to Section 3.4 in ISO 11171.

3.2.2 *diluent, n*—a solvent listed in Annex A1, Table A1.1, having viscosity less than 10 mm^2/s at 40 °C that is physically and chemically compatible with the apparatus used and easily soluble at room temperature with the sample lubricant or hydraulic fluid.

3.2.3 *emulsified water, n*—water that exists in oil between the states of fully dissolved and phase-separated. An emulsifying agent in the oil causes the two immiscible liquids to coexist in a heterogeneous mixture.

~~3.1.5 *free water, n*—water that exists in a separate phase in an oil sample. This occurs when the water content of the oil exceeds the water holding capacity of the oil.~~

3.2.4 *interfering soft particles, n*—an undissolved, dispersed material (such as an additive) within an oil blend or substance that is formed during the service life of an oil blend.

3.2.4.1 *Discussion—*

When these substances are present in a sample and not completely solubilized, they 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.

3.2.5 *ISO Codes, n*—a standard classification for coding the level of contamination by solid particles.

[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] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

3.2.5.1 *Discussion—*
This code simplifies the reporting of particle count data by converting the number of particles per mL into three classes covering ≥4 µm $_{(c)}$, ≥6 µm $_{(c)}$ and ≥14 µm $_{(c)}$. ISO 4406 classifications are used as an option to report results for this test method.

3.2.6 *particle size, µm $_{(c)}$, n*—diameter of a circle with an area equivalent to the projected area of a particle passing through the detecting cell in accordance with ISO 11171.

3.2.7 *particle size cumulative count, n*—total number of particles with sizes greater than a specified particle size (for example, ≥4 µm $_{(c)}$, ≥6 µm $_{(c)}$, ≥10 µm $_{(c)}$, ≥14 µm $_{(c)}$, ≥21 µm $_{(c)}$, ≥38 µm $_{(c)}$, etc.).

NOTE 3—All particle counts are expressed on per 1 mL basis.

~~3.1.10 *soot-in-oil, n*—a sub-micron particulate product of incomplete combustion commonly found in in-service diesel engine crankcase oil.~~

3.2.8 *water-masking diluent, n*—a particular kind of diluent capable of dissolving otherwise immiscible substances such as water or soft particles in the sample lubricant or hydraulic fluid. ~~See Annex A1, Table A1.1.~~
3.2.8.1 *Discussion—*
In Annex A1, Table A1.1, 25 % isopropanol / 75 % toluene and dipropylene glycol n-propyl ether are the only water-masking diluents in Table A1.1.

4. Summary of Test Method

4.1 Inspect sample.

4.2 Agitate sample.

4.3 Obtain aliquot from homogeneous sample if not diluting in original container.

4.4 Dilute with appropriate diluent for the sample type.

4.5 Agitate diluted sample.

4.6 Degas sample.

4.7 Begin testing within 90 s (or repeat agitation and degassing).

4.8 Obtain particle counts in triplicate (for sample and method blank).

4.9 Analyze data and conduct validity checks.

4.10 Report results.

5. Significance and Use

5.1 This test method is intended for use in analytical laboratories including onsite in-service oil analysis laboratories.

5.2 Hard particles in lubricating or fluid power systems have a detrimental effect on the system as they cause operating components to wear and also accelerate the degradation of the oil. Hard particles in the oil originate from a variety of sources including generation from within an operating fluid system or contamination, which may occur during the storage and handling of new oils or via ingress into an operating fluid system.

5.3 High levels of contaminants can cause filter blockages and hard particles can have a serious impact on the life of pumps, pistons, gears, bearings, and other moving parts by accelerating wear and erosion.

5.4 Particle count results can be used to aid in assessing the capability of the filtration system responsible for cleaning the fluid, determining if off-line recirculating filtration is needed to clean up the fluid system, or aiding in the decision of whether or not a fluid change is required.

5.5 To accurately measure hard particle contamination levels, it is necessary to negate the particle counts contributed by the presence of small levels of free water. This method includes a process by which this can be accomplished using a water-masking diluent technique whereby water droplets of a size below the target level are finely distributed.

5.6 Certain additives or additive by-products that are semi-insoluble or insoluble in oil, namely the polydimethylsiloxane defoamant additive and oxidation by-products, are known to cause light scattering in automatic particle counters, which in turn causes falsely high counts. These and similar materials are commonly termed “soft particles” (see ~~3.1.6~~3.2.4) and are not known to directly increase wear and erosion within an operating system. The contribution of these particles to the particle size cumulative count is negated with this method.

5.7 The use of dilution in this test method counteracts viscosity effects for highly viscous oils that impact the accuracy of automatic optical particle counting results.

6. Interferences

NOTE 4—This section is consistent with the interferences described in Test Method D6786.

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

6.2 Air bubbles in the oil may be counted as particles giving false positive readings. Mixing or agitating the sample introduces bubbles into the oil, but these readily dissipate with sonication or vacuum degassing.

6.3 Suspended or free water in the oil will generally be counted as particles.

NOTE 5—Free or emulsified water interference presented can be negated by using the water-masking diluent as described in this test method.

6.4 Excessive concentrations of particles in the oil will cause coincidence or electronic saturation errors, or both. Limits are determined by ISO 11171 and are generally supplied by the instrument manufacturer. These errors may be avoided by increasing the dilution ratio with the diluent used in this test method.

6.5 Odd-shaped particles and fibers may be classified with incorrect calculated particle size, depending on their orientation as they pass through the sensing zone of the instrument.

6.6 Dye-in-oil is used by some lubricant manufacturers to distinguish certain lubricant types or brands. It is unusual for that dye to have a discernible impact on particle count data. Nonetheless, it is worthwhile to evaluate possible interferences for dye-in-oil by testing a sample of filtered, dyed, lubricant. If the automatic particle counter yields unusual results or if it reports an optical warning message, then this may be an indication of this type of interference.

6.7 Excessive soot-in-oil is an interference that makes it impractical to test in-service diesel engine lubricants, especially when soot level exceeds 1 %. This is not normally a problem for natural gas and gasoline engine oils. However heavy duty diesel engine oils typically produce excessive soot for most automatic particle counters. The coincident, opaque, soot particles reduce light transmission and produce very high false particle counts.

6.8 Solid lubricants, such as molybdenum disulfide or graphite are used in some lubricating oils, especially for extreme pressure applications. These materials are typically used at levels high enough to render the fluid opaque or to cause coincidence errors due to high particle concentrations in the detector. Even if these factors can be overcome with sufficient dilution, increases in particle counts are difficult to determine with adequate precision due to the inherently high particle counts in these fluids.

6.9 Specimen bottles shall not be reused. This is a source of cross-contamination interference.

7. Apparatus

7.1 *Liquid Automatic Particle Counter (APC),* liquid optical particle counter based on the light extinction principle. The instrument shall be capable of recording the size and number of particles as they pass across the detector. The particle counter shall include a sampling apparatus that automatically delivers a predetermined volume of specimen at a controlled flow rate to the sensing zone of the analyzer.

7.2 *Analytical Balance,* for mass dilution, calibrated, with a resolution of 100 mg.

7.3 *Mechanical Shaker,* paint shaker, table shaker, or other mechanical device to vigorously agitate sample containers.

7.4 *Ultrasonic Bath,* rated at 3000 W/m^2 to 10 000 W/m^2. This bath aids in the removal of air bubbles generated in the sample during the agitation process while also working to suspend particles in the sample and slow the settling process.

7.5 *Liquid Dispensers,* fitted with 0.8 µm or finer filter.

7.6 *Volumetric Pipette and Bulb,* if volumetric dilution or fluid transfer with a pipette is desired. Pipettes made of graduated glass or disposable polyethylene. Any glassware used shall be cleaned and verified in accordance with ISO 3722.

7.7 *Density Meter,* with an accuracy of 0.01 g/cm^3, if the mass dilution method is used.

7.8 *Filter Apparatus,* for filtering the diluent. There is no requirement for the apparatus itself but it shall be capable of producing acceptably clean diluent as necessary. Take appropriate safety precautions in handling low flash materials.

7.9 *Vacuum Degassing Apparatus,* capable of pulling full vacuum on the sample container in a vacuum chamber (per 12.4.1) or syringe degassing port (per 12.4.3) within time limit specified.

7.10 *Glassware,* any glassware used shall be cleaned and verified in accordance with ISO 3722.

7.11 *Sample Container,* a container used for collecting the neat sample per 9.1 and 9.2, or for diluting sample specimens.

7.11.1 Sample containers shall not be reused.

7.11.2 Recommended containers are cylindrical specimen bottles (or jars) typically made of polypropylene, polystyrene, PET, or glass with nominally flat bottoms, fitted with a suitable non-shedding threaded cap.

7.11.3 The dimensions and capacity of sample containers depends on specimen requirements and APC design. Sample containers often have an approximate capacity of 125 mL. However individual specimen requirements and APC design may call for substantially smaller or much larger sample containers.

7.11.4 After performing any cleaning procedures, the sample containers shall meet the cleanliness criteria of contributing less than 1 % of the total particles expected in the cleanest sample.

7.11.5 Sample containers shall be compatible with fluid and able to withstand the temperature of the fluid when collecting the sample. Sample containers with certified cleanliness levels (for example, “ultra clean”) may be used to collect samples for particle counting.

7.12 *Specimen Bottle,* or sample specimen bottle, a sample container used for diluting at least a portion of a sample. A specimen bottle shall meet the same criteria as the sample container (7.11.1 – 7.11.5).

7.13 *Filters,* to be used with filter apparatus (see 7.8). Recommended filters are cellulose or polycarbonate with a 0.8 µm or smaller pore size.

7.14 *Disposable, Single-Use Syringes,* uncontaminated and directly taken from individually sealed pouches are sometimes used instead of a sample inlet tube to deliver samples to automatic particle counters.

8. Reagents and Materials

8.1 *Calibration Fluid,* a suspension of ISO Medium Test Dust in oil or hydraulic fluid, using either a primary sample obtained directly from NIST (SRM 2806) or a secondary sample prepared in accordance to ISO 11171 and at least secondary traceable to NIST.

8.2 *Diluent,* from list in Annex A1, Table A1.1, shall be filtered to ensure it contributes less than 12.5 % of the total particles counted in the diluted sample tested according to this method.

8.3 *Water-Masking Diluent,*[4] ~~from~~ from Annex A1, Table A1.1, is either a volumetric mixture of toluene and 2-propanol (also called isopropanol or isopropyl alcohol), typically in 75:25 proportions, or dipropylene glycol n-propyl ether.[5] The water-masking diluent shall be filtered to ensure it contributes less than 12.5 % of the total particles counted in the diluted sample tested according to this method.

9. Sample Collection and Handling

9.1 Unless otherwise specified, take a representative sample in accordance with Practices D4057, D4177, or other comparable sampling practices using a clean and appropriate sample container. Containers previously holding a sample or any other type of fluid are not considered appropriate containers.

9.2 Ensure that enough sample is collected to perform all analysis methods of interest. The container shall not be filled beyond 80 % of its total capacity to allow volume for sufficient agitation.

9.3 Label the sample according to the expectations of the analyzing laboratory, including oil type at a minimum.

9.4 Upon receipt at the analyzing laboratory, the sample shall be inspected and any non-standard conditions noted. This includes inappropriate container, overfilled container, visible particulates, and free water. Recommend a re-sample if inappropriate container or overfilled container is noted.

9.5 Determine whether water-masking diluent (see 6.3) or diluent (non water-masking) is to be used for dilution based on indication or not of emulsified water-in-oil.

9.6 Determine the desired dilution ratio. The dilution ratio may vary depending on the viscosities of the sample and the diluent and the range of viscosities that can be accommodated by the APC.

9.6.1 A dilution ratio of ~50 % sample to ~50 % diluent is acceptable for most applications although a smaller sample-to-diluent ratio is often used.

9.6.2 If the sample is very dark or high particulate contamination is suspected, a lower sample to diluent dilution ratio is suggested. This sort of excessively high contamination deserving lower dilution ratio is often evidenced when ISO ≥4 µm $_{(c)}$ is equivalent to ISO ≥6 µm $_{(c)}$ indicating unusually small difference in particle counts between these size ranges.

9.6.3 For the temporary precision statement of reproducibility reported in Section 14, a mass dilution ratio of 25 % ± 2 % sample to 75 % ± 2 % diluent was consistently used and the diluent was a blend of 33 % ± 2 % lamp oil with 67 % ± 2 % dipropylene glycol n-propyl ether (DPnB) for water-masking.

---

[4] A water-masking method for counting light obstructing particles in a test oil sample containing a substantially immiscible fluid, and where the test oil sample is mixed with a masking fluid that is soluble with the oil and with the substantially immiscible fluid, is covered by US Patent 6,064,680 issued May 16, 2000.

[5] The sole source of manufacturer of the diluent known to the committee at this time is DOWANOL DPnB, a registered trademark of Dow Chemical Company, Abbott Rd., Midland, MI 48640. Dow Chemical Company supplies 1–L bottles of DOWANOL DPnB through Sigma-Aldrich Corp., St. Louis, MO, www.sigma-aldrich.com. 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.