Designation: 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*

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.*

1.9 *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:*

[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.

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

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

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 of Terms Specific to This Standard:*

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.

3.2.4 *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.

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.

3.2.8 *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.

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 3.2.4) 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.