



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 Extinction¹

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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 $\mu\text{m}_{(c)}$ to 200 $\mu\text{m}_{(c)}$ 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 @ 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 test method 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 appro-*

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

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

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 the purposes of this test method, the following definitions apply:

3.1.2 *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.

3.1.3 *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

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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>.

and chemically compatible with the apparatus used and easily soluble at room temperature with the sample lubricant or hydraulic fluid.

3.1.4 *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.

3.1.6 *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.

3.1.6.1 *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.

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

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

3.1.8 *particle size, $\mu\text{m}_{(c)}$, 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.

3.1.9 *particle size cumulative count, n*—total number of particles with sizes greater than a specified particle size (for example, $\geq 4 \mu\text{m}_{(c)}$, $\geq 6 \mu\text{m}_{(c)}$, $\geq 10 \mu\text{m}_{(c)}$, $\geq 14 \mu\text{m}_{(c)}$, $\geq 21 \mu\text{m}_{(c)}$, $\geq 38 \mu\text{m}_{(c)}$, 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.

3.1.11 *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](#).

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](#)) 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 to 10 000 W/m². 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/cm³, 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 [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 deserv-

ing lower dilution ratio is often evidenced when $ISO \geq 4 \mu m_{(c)}$ is equivalent to $ISO \geq 6 \mu m_{(c)}$ 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 $25 \pm 2 \%$ sample to $75 \pm 2 \%$ diluent was consistently used and the diluent was a blend of $33 \pm 2 \%$ lamp oil with $67 \pm 2 \%$ dipropylene glycol n-propyl ether (DPnB) for water-masking.

9.7 Take appropriate safety precautions when collecting samples.

10. Calibration and Verification

10.1 Calibration:

10.1.1 Calibration of the APC shall be done with an undiluted NIST-traceable calibration fluid in accordance with ISO 11171.

10.1.2 Calibration of the APC shall be done within the timeframe indicated by the particle counter manufacturer, or at least annually if no frequency is specified. Refer to reference in [10.2.1](#) to establish calibration frequency.

10.2 Verification:

10.2.1 Verify that the APC is holding its calibration by analyzing a primary or secondary calibration fluid allowing for sample-to-sample variation outlined in [Annex A2, Table A2.1](#) and allowing for calibration fluid batch-to-batch variation.

10.2.2 Verify that the APC is providing internally consistent results by analyzing diluent looking for expected results and allowing for sample-to-sample variation outlined in [Annex A2, Table A2.1](#).

10.2.3 Verification checks are recommended on a quarterly basis for instruments analyzing multiple samples regularly (such as weekly). Checks shall also be performed if an instrument has not been used for over three months or if the particle counter has been altered in such a way that could impact its calibration.

11. Preparation of Apparatus

11.1 The APC shall be set up according to the instrument manufacturer's operating manual.

11.2 Ensure that the ISO 4406 mode of operation is selected and displayed. If the APC has printing or electronic data output capabilities, ensure those are also set to ISO 4406.

11.3 Set the flow-rate to the setting at which the APC was calibrated.

11.4 If the APC is capable of running a pre-set program, set the APC to flush 10 to 25 mL of sample prior to analysis, then run three analyses consecutively of at least 5 mL per run. If the APC cannot run a preset flush, set it to run four analyses consecutively of at least 10 mL per run. When setting the amount of fluid to be analyzed per run, ensure that the total volume consumed by the APC does not exceed the volume of sample held in the sample specimen bottle.

11.5 If the APC has an inlet tube (also called pickup tube) use a clean, low-lint towel, wipe off sample inlet tube or any other materials of the APC that will directly contact the sample fluid. However do not allow any towel or surface to contact an

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.

unsealed disposable syringe if using that type APC. Alternatively, use a solvent wash to clean contaminated surfaces.

11.6 If the fluid last analyzed by the particle counter was excessively dirty or of unknown origin, flush or back-flush, or both, the unit according to the manufacturer's instructions with clean diluent or other compatible diluent recommended by the APC manufacturer. It is recommended that then a test sequence with the diluent be performed to confirm the cleanliness level is acceptable.

NOTE 6—Excessively dirty is a condition when counts in the particle counter at counts/mL $\geq 4 \mu\text{m}_{(c)}$ in excess of either 40 000 particles/mL or the coincidence error limit, whichever is less. Keep in mind the diluting factor—in order to have 40 000 particles/mL in the particle counter with 1:1 dilution, the in-service lubricant contains approximately 80 000 particles/mL.

11.7 Prior to beginning the procedure in Section 12 or 11.7, perform any preparatory steps required by the APC such as entering the sample name. The APC shall be ready for immediate analysis before beginning the procedure.

11.8 Clean diluent background analysis is performed to appropriately subtract particle counts contributed by diluent or water-masking diluent, or both.

11.8.1 The contributing particles from the diluent are excluded from the final sample results obtained by this method. A particle analysis of the diluent shall, at a minimum, be performed at the beginning of each day of testing.

11.8.2 Remove the lid from a clean specimen bottle and fill with diluent to be used for sample dilution, ensuring the specimen bottle is not filled beyond 80 % of its total capacity. The amount of diluent added shall be representative of the diluent to be used for sample dilution and the volume shall be sufficient to analyze at least three replicates with the APC including one 10-mL or larger flush or additional analysis, depending on the capability of the APC to perform a flush prior to analysis.

11.8.3 Analyze the sample diluent as described in 12.3-12.7.

11.8.4 Average the particle size cumulative count results for the three analyses (discarding the initial analysis if four are required). Retain this result so that these contributing particles can be removed when calculating the number of particles in the sample that is diluted with this diluent.

12. Procedure

12.1 Volumetric Dilution:

12.1.1 Use volumetric glassware that conforms to 7.6.

12.1.2 Shake the sample container and its contents in the mechanical shaker. For samples 200 mL or less, shake for one min. For samples 200 mL or larger, shake for three min. (Samples that are provided in containers larger than the capacity of the mechanical shaker may be shaken using a lateral shaker or other apparatus that provides sufficient agitation. If an alternate apparatus is used, a longer agitation time may be required.)

12.1.3 Remove the lid from a clean specimen bottle that can hold 120 mL of fluid (typical).

12.1.4 Remove the lid from the sample container and transfer the appropriate specimen volume of sample into that

sample specimen bottle from 12.1.3 to achieve the dilution ratio determined in 9.6 while not overfilling the sample container. For a sample specimen bottle that can hold 120 mL of fluid and a dilution ratio of 50 % sample specimen to 50 % diluent, add approximately 45 mL of sample specimen. If more than 90 s elapses after the shaking of the sample container in 12.1.2 before the addition of the sample specimen into the empty specimen bottle in 12.1.4, then the sample container shall be shaken again per 12.1.2 to re-homogenise the bulk sample before transferring the specimen. Record the volume of added sample specimen (V_s) to at least 1 mL (for example, 45 mL).

12.1.5 Replace the lid and return the sample container to an appropriate location for storage or further analysis.

12.1.6 Add the appropriate amount of the diluent selected in 9.5 into the specimen bottle of sample filled in 12.1.4 to achieve the dilution ratio determined in 9.6 while not overfilling the sample container. For a sample container that can hold 120 mL of fluid and a dilution ratio of 50 % sample to 50 % diluent, add approximately 45 mL of diluent. Record the volume of added diluent (V_d) to at least one mL (for example, 45-mL).

12.1.7 Replace the lid on the specimen bottle containing the diluted sample.

12.1.8 Use the following formula to calculate the dilution ratio with respect to the sample ($D_{r,s}$) and the dilution ratio with respect to the diluent ($D_{r,d}$):

$$D_{r,s} = \frac{V_s + V_d}{V_s} \quad (1)$$

$$D_{r,d} = \frac{V_s + V_d}{V_d} \quad (2)$$

where:

$D_{r,s}$ = dilution ratio with respect to the sample,

$D_{r,d}$ = dilution ratio with respect to the diluent,

V_s = volume of sample, and

V_d = volume of diluent.

12.2 Mass Dilution:

12.2.1 If glassware is used, it shall conform to 7.10. Pouring of sample or diluent, or both, into the specimen bottle used for the diluted sample created in this procedure is acceptable to change the mixing ratio.

12.2.2 Homogenize the incoming sample by shaking the sample container and its contents in the mechanical shaker. For samples 200 mL or less, shake for 1 min. For samples 200 mL or larger, shake for 3 min. (Samples that are provided in containers larger than the capacity of the mechanical shaker may be shaken using a lateral shaker or other apparatus that provides sufficient agitation. If an alternate apparatus is used, a longer agitation time may be required.)

12.2.3 If the density of the sample or diluent is unknown, determine and record the density using the density meter while ensuring that this process does not add contamination to the sample or diluent. Repeat 12.2.2 for the sample before proceeding to 12.2.4.

12.2.4 Remove the lid from a clean, empty bottle like the one that will be used for dilution and place it on the electronic balance. Tare the balance.

12.2.5 Remove the lid from the sample container and either weigh the sample container directly or transfer the appropriate volume of sample into a sample specimen bottle used to achieve the dilution ratio determined in 9.6 while not overfilling the sample specimen bottle. For example, in a sample specimen bottle that can hold 120 mL of fluid and a dilution ratio of 50 % sample to 50 % diluent, add approximately 40 g of sample. If more than 90 s elapse after the shaking of the sample container before the addition of the sample into the specimen bottle, the sample shall be shaken again per 12.2.2 before performing this step.

12.2.6 If dilution is not being done in the original sample container in which the sample was originally collected, replace the lid and return the sample container to an appropriate location for storage or further analysis.

12.2.7 Reweigh the specimen bottle that now includes sample from 12.2.4 and record the exact weight once the balance has stabilized. This weight is the weight of the added sample (M_s). Tare the balance.

12.2.8 Add the appropriate amount of the diluent selected in 9.5 into the specimen bottle of sample filled in 12.1.4 to achieve the dilution ratio determined in 9.6 while not overfilling the sample container. For example, in a sample container that can hold 120 mL of fluid and a dilution ratio of 50 % sample to 50 % diluent, add approximately 40 g of diluent.

12.2.9 Reweigh the sample specimen bottle now containing the sample and diluent and record the exact weight once the balance has stabilized. This weight is the weight of the added diluent (M_d).

12.2.10 Replace the lid on the specimen bottle containing the diluted sample.

12.2.11 Use the following formula to calculate the dilution ratio with respect to the sample ($D_{r,s}$) and the dilution ratio with respect to the diluent ($D_{r,d}$):

$$D_{r,s} = \frac{\frac{M_s + M_d}{P_s + P_d}}{\frac{M_s}{P_s}} \quad (3)$$

$$D_{r,d} = \frac{\frac{M_s + M_d}{P_s + P_d}}{\frac{M_d}{P_d}} \quad (4)$$

where:

- $D_{r,s}$ = dilution ratio with respect to the sample,
- $D_{r,d}$ = dilution ratio with respect to the diluent,
- M_s = mass of sample (in grams),
- M_d = mass of diluent (in grams),
- P_s = density of sample (in g/cm³), and
- P_d = density of diluent (in g/cm³).

12.3 Manually or mechanically agitate the diluted sample vigorously for 30 s.

12.4 Immediately degas the diluted sample by using vacuum chamber or ultrasonic bath or syringe degassing port or combination:

12.4.1 If using a vacuum chamber, remove the lid from the specimen bottle containing the homogenised diluted sample. Place it in the vacuum chamber. Evacuate the chamber and

observe until bubbles have risen to the surface. Vacuum degassing shall not exceed 30 s. Vent the vacuum chamber and remove the sample.

12.4.2 If using an ultrasonic bath, then degas by unscrewing ¼ turn or partially open the lid to the specimen bottle and placing the sample in the ultrasonic bath for 10 to 15 s, or until visible air bubbles have surfaced to the top of the sample. If needed, hold the sample in place to ensure it does not tip over in the ultrasonic bath. Sonication time shall not exceed 30 s. Remove the sample from the ultrasonic bath and wipe dry the exterior of the specimen bottle.

NOTE 7—The purpose of partially unscrewing or opening the lid is to promote quick degassing of the sample. It is acceptable for air bubbles to remain near the surface of the sample above the level of water in the ultrasonic bath. It may be noted that air bubbles appear to be generated on the interior walls of the sample specimen bottle while it is being sonicated. These created bubbles can continue indefinitely and shall not be of concern when determining whether or not the sample has been properly agitated.

12.4.3 If using syringe degassing port, open a sealed package containing the syringe, fill the syringe with diluted sample from the homogenised diluted sample, install a retaining spacer to prevent syringe plunger from retracting, insert Luer tip of syringe upward into degassing port and initiate vacuum suction through that port. Observe until bubbles rise to the top portion of the syringe. Vacuum degassing shall not exceed 30 s. After degassing, immediately remove the syringe from the degassing port, expel accumulated air, invert the syringe, and insert the Luer tip of the syringe into the sample testing port.

12.5 If necessary, perform any additional steps specific to the APC to be used for analysis, such as transferring the sample into a syringe and then degassing that sub-sample of diluted lubricant in preparation for injection into the APC. Immediately begin the processing of the sample on the APC that has been prepared according to Section 10. If more than 60 s elapse after the shaking of the diluted sample in 12.3 and the start of the automated analysis by the APC in 12.6, the procedure shall be restarted with shaking of sample in step 12.3. This is to ensure that suspended particles do not settle in the diluted sample.

12.6 Obtain three consecutive qualifying particle count results. If the APC is not capable of performing consecutive sampling as described in Section 11, run two or three additional analyses of the same sample, beginning at 12.3. The number of additional analyses to be run is dependent on the capability of the APC to perform a flush cycle with the sample prior to the very first analysis. If the unit can be flushed with at least 10 mL of the sample prior to the first analysis, three total analyses are necessary and are considered qualified particle count results. If the unit cannot be flushed with at least 10 mL of the sample prior to the first analysis, four total analyses are necessary. In this case, the particle count results from the first sample are to be discarded and the results from the next three analyses are considered qualified particle count results. This is to ensure that there are three analyses of the sample that do not include any cross contamination from a different sample previously analyzed.

12.7 Compare the particle count to the coincidence error limit specified by the APC manufacturer. If the sample exceeds this limit, the sample requires further dilution. Either a new sample shall be made from the original neat sample, beginning at 12.1, or the current sample with a known dilution ratio shall be further diluted beginning at 12.3.

12.8 Average the particle size cumulative count results for the three analyses (discarding the initial analysis if four are required). For the cumulative particle counts at the $\geq 4 \mu\text{m}_{(c)}$, $\geq 6 \mu\text{m}_{(c)}$, $\geq 14 \mu\text{m}_{(c)}$ size ranges, the results shall be rejected if the variation between the counts exceeds the limits in **Annex A2**. Retest may be performed provided there is sufficient diluted sample specimen remaining beginning either with the step to agitate the diluted sample specimen (12.3) or with the step to homogenise the neat sample in the original container (12.2).

12.9 Calculate the cumulative number of particles per milliliter at each particle size present in the original sample by using the following formula. Refer to **Annex A2** demonstrating this calculation:

$$C_{s,n} \geq n \mu\text{m} = D_{r,s} \left(C_{t,n} - \frac{C_d}{D_{r,d}} \right) \quad (5)$$

where:

- n = Particle size range of interest (for example, $\geq 4 \mu\text{m}_{(c)}$),
- $C_{s,n}$ = Cumulative particle counts per millilitre in the original sample at particle size range n ,
- $D_{r,s}$ = Dilution ratio with respect to the sample as calculated in 12.1.8 or 12.2.11,
- $C_{t,n}$ = Resulting cumulative particle counts per millilitre at particle size range $n \mu\text{m}$ from 12.8 of the analysis of the diluted sample,
- C_d = Cumulative particle counts per millilitre in the original diluent at size range n as determined by 11.8.4, and
- $D_{r,d}$ = Dilution ratio with respect to the diluent as calculated in 12.1.8 or 12.2.11.

12.9.1 If the contribution of particles from the diluent exceeds 12.5 % of the total number of particles counted (for example, $\geq 4 \mu\text{m}_{(c)}$), the sample shall be considered invalid. It shall either be reanalyzed after additional filtration of the diluent or a notation of this non-compliance shall be added to the report.

12.10 At the end of the testing day, flush the particle counter with clean diluent or an oil of a known cleanliness approximately equal to the clean diluent.

13. Report

13.1 Report should include the following information:

13.1.1 A reference to this test method.

13.1.2 The sample identification.

13.1.3 The date of the test.

13.1.4 Particle size cumulative counts reported by the instrument (for example, $\geq 4 \mu\text{m}_{(c)}$, $\geq 6 \mu\text{m}_{(c)}$, $\geq 14 \mu\text{m}_{(c)}$, $\geq 21 \mu\text{m}_{(c)}$, $\geq 38 \mu\text{m}_{(c)}$, etc.).

13.1.5 ISO code result, according to ISO 4406 for $\geq 4 \mu\text{m}_{(c)}$, $\geq 6 \mu\text{m}_{(c)}$, $\geq 14 \mu\text{m}_{(c)}$. (Other classification systems may also be used, for example, SAE AS4059.)

13.1.6 Any deviation, by agreement or otherwise, from the specified procedures.

13.1.7 In cases of dispute also report information related to the APC instrument and any settings that deviate from those given in this test method.

14. Precision and Bias

14.1 A temporary precision statement including preliminary repeatability and Reproducibility standard deviation is reported in **Table 1**. Data were collected on multiple instruments of the same type using mass dilution (12.2) and syringe degassing (12.4.3). Other instrument types, dilution and degassing options will be tested so that a final precision will be determined and available within five years after the publication of this test method.⁶

14.2 **Table 1** reports repeatability calculated from results of two samples tested thirty times each in the same laboratory within-laboratory variability. This table also reports Reproducibility calculated from the results of thirteen samples tested in seven different field laboratories.

15. Keywords

15.1 automatic particle counter; diluent; emulsified water; particle contamination; particle counting; soft particles; water-masking

⁶ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1646.

TABLE 1 Temporary Precision with 95 % Confidence

Parameter	Units	Repeatability	Reproducibility	Data Range	Data Range
		2.77 × std dev	2.77 × std dev	Low	High
Counts $\geq 4 \mu\text{m}_{(c)}$	counts/mL	30 %	113 %	150	110 000
Counts $\geq 6 \mu\text{m}_{(c)}$	counts/mL	30 %	76 %	60	60 000
Counts $\geq 14 \mu\text{m}_{(c)}$	counts/mL	73 %	135 %	6	22 000
ISO $\geq 4 \mu\text{m}_{(c)}$	code value	<1	1.7	14	24
ISO $\geq 6 \mu\text{m}_{(c)}$	code value	<1	1.2	13	22
ISO $\geq 14 \mu\text{m}_{(c)}$	code value	1.5	2	10	18

ANNEXES
(Mandatory Information)
A1. TYPICAL DILUENTS

A1.1 *Diluent List*—See [Table A1.1](#).

TABLE A1.1 Diluent List

NOTE 1—Precision statement for repeatability and reproducibility is based using diluents marked with asterisk (*).

Diluent	Is this a water-masking diluent?	MSDS Health Rating	MSDS Flammability Rating	MSDS Reactivity Rating
Stoddard solvent, also called Type 1 mineral spirits or white spirits	no	2	2	1
kerosene	no	1	2	0
lamp oil*	no	1	1	0
25 % 2-isopropanol / 75 % toluene	yes	3	3	0
dipropylene glycol n-propyl ether*	yes	0	0	0

A2. PROCEDURE FOR VERIFYING ACCEPTABLE ANALYSIS-TO-ANALYSIS VARIABILITY FOR APC SAMPLES

NOTE A2.1—This procedure and table are consistent with 6.3 of this test method and Table 8 in ISO 11171. If the total number of particles counted for the size range being considered is less than 100, this procedure is not required and the data can be considered valid.

A2.1 Average the three qualifying raw particle counts. Do not perform any calculations to correct for dilution if applicable.

A2.2 Calculate the percent difference (D_q) between the highest (C_{max}) and lowest (C_{min}) measured particle count for the particle size range (for example, $\geq 4 \mu\text{m}_{(c)}$, $\geq 6 \mu\text{m}_{(c)}$, or $\geq 14 \mu\text{m}_{(c)}$).

$$D_q = \frac{100(C_{max} - C_{min})}{\bar{C}} \quad (\text{A2.1})$$

where:

- D_q = percent difference,
- C_{max} = highest cumulative particle count for the size range obtained in the series of analyses,
- C_{min} = lowest cumulative particle count for the size range obtained in the series of analyses, and
- \bar{C} = the average of the cumulative particle count for the size range obtained in the series of analyses.

A2.3 Compare the calculated percent differences to those shown in [Table A2.1](#) for the corresponding average number of particle counts. If the calculated percent difference is less than or equal to the maximum allowable percent difference value in [Table A1.1](#), the data is considered valid. If the calculated percent difference exceeds the value given in [Table A1.1](#), the data shall be rejected and appropriate steps taken to correct for analytical errors.

TABLE A2.1 Maximum Allowable Percent Differences in Particle Counts Between Runs

Average Number of Total Particles Counted per Run, \bar{C}	Maximum Allowable Percent Difference
$10\,000 \leq \bar{C} < \infty$	11.0
$5000 \leq \bar{C} < 10\,000$	11.3
$2000 \leq \bar{C} < 5000$	11.9
$1000 \leq \bar{C} < 2000$	13.4
$500 \leq \bar{C} < 1000$	15.6
$200 \leq \bar{C} < 500$	19.3
$100 \leq \bar{C} < 200$	27.5
$50 \leq \bar{C} < 100$	37.4
$20 \leq \bar{C} < 50$	51.8

A3. EXAMPLE OF VARIABILITY ANALYSIS AND DILUTION CORRECTION CALCULATION OF AN OIL SAMPLE

A3.1 Sample Information A3.1

$V_s = 45.0$ -mL sample
 $V_d = 45.0$ -mL diluent
 Dilution ratio with respect to the sample, $D_{r,s} = 2.0$
 Dilution ratio with respect to the diluent, $D_{r,d} = 2.0$

A3.2 Analysis Information A3.2

15 mL flush followed by three consecutive analyses
 Volume/analysis – 10 mL

A3.3 Variability Analysis

A3.3.1 Sample Results:

Size Range	Cumulative Counts/mL				Total Particles Counted ^A	D_q^B	Max Allowable D_q^C
	Run 1	Run 2	Run 3	Average			
$\geq 4 \mu\text{m}_{(c)}$	1817.8	1819.7	1874.7	1837.07	18371	3.2 %	11.0 %
$\geq 6 \mu\text{m}_{(c)}$	387.5	373.6	395.8	385.63	3856	5.8 %	11.9 %
$\geq 10 \mu\text{m}_{(c)}$	106.7	98.9	115.2	106.93	1069	N/A	N/A
$\geq 14 \mu\text{m}_{(c)}$	41.5	38.9	45.2	41.87	419	15.0 %	19.3 %
$\geq 21 \mu\text{m}_{(c)}$	9.1	12.3	11.8	11.07	111	N/A	N/A
$\geq 38 \mu\text{m}_{(c)}$	1.2	1.3	1.2	1.23	12	N/A	N/A
$\geq 70 \mu\text{m}$	0.6	0.8	0.7	0.70	7	N/A	N/A

^A Product of average cumulative counts/mL multiplied by the total volume analyzed (10 mL).

^B Calculated according to A2.2.

^C Determined according to Table A2.1.

A3.4 Dilution Correction Calculation

A3.4.1 Diluent Cleanliness Results in accordance with 11.8:

Size Range	Cumulative Counts/mL			
	Run 1	Run 2	Run 3	Average
$\geq 4 \mu\text{m}_{(c)}$	50.2	48.6	51	49.93
$\geq 6 \mu\text{m}_{(c)}$	22.3	20.7	22.4	21.80
$\geq 10 \mu\text{m}_{(c)}$	10.1	9.6	10.4	10.03
$\geq 14 \mu\text{m}_{(c)}$	1.2	0.9	1.2	1.10
$\geq 21 \mu\text{m}_{(c)}$	0.4	0.3	0.5	0.40
$\geq 38 \mu\text{m}_{(c)}$	0.2	0.1	0.2	0.17
$\geq 70 \mu\text{m}$	0	0	0	0.00

A3.4.2 Cumulative particles per millilitre in the original sample, $\geq 4 \mu\text{m}_{(c)}$ (in accordance with 12.9):

$$C_{s,n} \geq n \mu\text{m} = D_{r,s} \left(C_{t,n} - \frac{C_d}{D_{r,d}} \right) \quad (\text{A3.1})$$

$$C_{s,4} \geq 4 \mu\text{m} = 2.0 \left(1837.07 - \frac{49.93}{2.0} \right) = 3624.21 \text{ counts/mL} \quad (\text{A3.2})$$

A3.4.3 Repeating this calculation for all other size ranges gives the following:

Size Range	Cumulative Counts/mL in the Original Sample
$\geq 4 \mu\text{m}_{(c)}$	3624.2
$\geq 6 \mu\text{m}_{(c)}$	749.5
$\geq 10 \mu\text{m}_{(c)}$	203.8
$\geq 14 \mu\text{m}_{(c)}$	82.6
$\geq 21 \mu\text{m}_{(c)}$	21.7
$\geq 38 \mu\text{m}_{(c)}$	2.3
$\geq 70 \mu\text{m}$	1.4

A3.4.4 The ISO code result, according to ISO 4406:99 for $\geq 4 \mu\text{m}_{(c)}$, $\geq 6 \mu\text{m}_{(c)}$, $\geq 14 \mu\text{m}_{(c)}$: 19/17/14.

A3.4.5 These numbers are to be reported according to Section 13.

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