



Designation: D7619 – 10

## Standard Test Method for Sizing and Counting Particles in Light and Middle Distillate Fuels, by Automatic Particle Counter<sup>1, 2</sup>

This standard is issued under the fixed designation D7619; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method uses a specific automatic particle counter<sup>2</sup> (APC) to count and measure the size of dispersed dirt particles, water droplets and other particles, in light and middle distillate fuel, and bio fuels such as biodiesel and biodiesel blends, in the overall range from 4  $\mu\text{m(c)}$  to 100  $\mu\text{m(c)}$  and in the size bands  $\geq 4 \mu\text{m(c)}$ ,  $\geq 6 \mu\text{m(c)}$ , and  $\geq 14 \mu\text{m(c)}$ .

NOTE 1—ASTM specification fuels falling within the scope of this test method include Specifications: D975 grades 1D and 2D, D3699, D4814 (see 14.1.1.1), D6751, D6985, D7467 and distillate grades of D396 and D2880.

NOTE 2—For the purposes of this test method, water droplets are counted as particles, and agglomerated particles are detected and counted as a single larger particle. Dirt includes biological particles. Although the projected area of a particle is measured, this is expressed as the diameter of a sphere for the purposes of this test method.

NOTE 3—The notation (c), used with particle sizes, is used to denote that the apparatus has been calibrated in accordance with ISO 11171. Strictly this only applies to particles up to 50  $\mu\text{m}$ .

NOTE 4—This test method may be used for particle size bands up to 100  $\mu\text{m(c)}$ , however the precision has only been determined for the size bands  $\geq 4 \mu\text{m(c)}$ ,  $\geq 6 \mu\text{m(c)}$ , and  $\geq 14 \mu\text{m(c)}$ .

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.*

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.14 on Stability and Cleanliness of Liquid Fuels.

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<sup>2</sup> The following equipment, as listed in RR:D02-1696, Seta-Avcount 91700-0 available from Stanhope-Seta, London Street, Chertsey, Surrey KT16 8AP UK was used to develop the precision statement. This listing is not an endorsement or certification by ASTM International.

### 2. Referenced Documents

#### 2.1 ASTM Standards:<sup>3</sup>

D396 Specification for Fuel Oils

D975 Specification for Diesel Fuel Oils

D2880 Specification for Gas Turbine Fuel Oils

D3699 Specification for Kerosine

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D4814 Specification for Automotive Spark-Ignition Engine Fuel

D5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants

D6751 Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels

D6985 Specification for Middle Distillate Fuel Oil—Military Marine Applications<sup>4</sup>

D7467 Specification for Diesel Fuel Oil, Biodiesel Blend (B6 to B20)

#### 2.2 ASTM Adjuncts:<sup>5</sup>

ADJ6300 D2PP Determination of Precision and Bias data for Use in Test Methods for Petroleum Products

#### 2.3 ISO Standards:<sup>6</sup>

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>4</sup> Withdrawn. The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

<sup>5</sup> For referenced ASTM adjuncts contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org).

<sup>6</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

**ISO 11171** Hydraulic Fluid Power—Calibration of Automatic Particle Counters for Liquids

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

**ISO 12103-A1** Specification for Ultra Fine Test Dust (UFTD)

**ISO 12103-A3** Specification for Medium Test Dust (MTD)

### 3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *particles, n*—solid particles and dispersed water droplets which are detected and counted by this test method.

3.1.2 *particle count, n*—the sum of the number of solid particles and dispersed water droplets.

3.1.3 *particle size,  $\mu\text{m}(c)$ , n*—the projected area equivalent diameter of spherical particles passing through the detecting cell in accordance with **ISO 11171**.

3.1.4 *particle size cumulative count, n*—the total number of particles per mL, in size bands,  $\geq 4 \mu\text{m}(c)$ ,  $\geq 6 \mu\text{m}(c)$ , and  $\geq 14 \mu\text{m}(c)$ ,

3.1.4.1 *Discussion*—Automatic particle counters may also count the total number of particles per mL, in size bands, in addition to those in 3.1.4, up to  $\geq 100 \mu\text{m}$ .

3.1.5 *ISO Codes, n*—a standard method for coding the level of contamination by particles.

3.1.5.1 *Discussion*—Results are expressed by ISO Codes as specified by **ISO 4406**. These codes are written in the form of  $x/y/z$ , where  $x$ ,  $y$  and  $z$  are ISO Codes equivalent to the cumulative counts, per mL, for particle size bands  $\geq 4 \mu\text{m}(c)$ ,  $\geq 6 \mu\text{m}(c)$ , and  $\geq 14 \mu\text{m}(c)$  respectively. An example of this is given in **Appendix X1**.

NOTE 5—All particle counts are per mL.

3.1.6 *coincidence error limit, n*—the highest concentration of ISO ultrafine test dust (**ISO 12103-A1** or ISO UFTD) that can be counted with an automatic particle counter with less than 5 % error resulting from the presence of more than one particle in the sensor/laser optical path at a time.

3.1.7 *test specimen, n*—an aliquot of the test sample. (See Section 10.)

### 4. Summary of Test Method <sup>2</sup>

4.1 The optical measurement cell comprises a light source and an optical sensor. The principle of operation is the measurement of laser light obscuration. Particles/droplets entrained within the test specimen cast shadows on the optical sensor causing a reduction of the output voltage of the sensor. The voltage drop is a function of the particle/droplet size. Each detected particle is counted, sized and recorded. Upon completion of the test the software calculates and displays the number of obscuration events for each of the predetermined size bands.

4.2 The test specimen is mixed in its container to suspend the particles. Upon initiation of a test, the automatic particle counter (APC) draws the test specimen directly from a test specimen container (see **Fig. A1.1**). The test sequence commences by flushing the optical measurement cell and pipework with 30 mL of the test specimen. This is immediately followed by the test of a 10 mL test specimen where particles in each of the specified size bands are counted. This flushing and mea-

surement is then repeated. If the size band  $\geq 4 \mu\text{m}(c)$ , per mL, measurements agree within either 10 % or 200 counts, the measurements for each of the size bands are averaged for each size band to give results, per mL, for each size band.

### 5. Significance and Use

5.1 This test method is intended for use in the laboratory or in the field for evaluating the cleanliness of distillate fuels, and liquid bio fuels. It is not applicable to on or in-line applications.

5.2 This test method offers advantage over traditional filtration methods in that it is a precise rapid test, and advantage over visual methods as it is not subjective.

5.3 An increase in particle counts can indicate a change in the fuel condition caused by storage or transfer for example.

5.4 High levels of particles can cause filter blockages and have a serious impact on the life of pumps, injectors, pistons and other moving parts. Knowledge of particle size in relation to the metallurgy can provide vital information especially if the hardness of particles is also known from other sources.

5.5 This test method specifies a minimum requirement for reporting measurements in particle size bands (see **A1.1.2**). Some specific applications may require measurements in other particle size bands.

5.6 Obtaining a representative sample and following the recommended sample and test specimen preparation procedures and timescales is particularly important with particle counting methods. (See Sections 8, 10, 14.1.4 and **Note 10**.)

### 6. Apparatus

6.1 *Automatic Particle Counter (APC)*<sup>2</sup>—Operating on the laser light obscuration principle, comprising an optical measurement cell, bi-directional double pump, electronics and software to analyze the test specimen, and display and print the particle measurement data. (See **Annex A1**.)

6.2 *Test Specimen Container*, cylindrical, made of glass or other suitable material, of at least 125 mL volume with provision for holding the test specimen input tube at least 10 mm above the bottom of the container, and a cap with a suitable inert internal seal.

NOTE 6—It is recommended that glass test specimen containers should be used to avoid any potential problems with particles adhering to the insides of the containers due to static electricity that could occur with some samples or some specimen containers.

6.3 *Waste Container*, for collecting the tested test specimen.

6.4 *Filter Apparatus*, general purpose for filtering heptane or other solvents.

6.4.1 *Filters*, cellulose, glass fiber or polycarbonate 0.45  $\mu\text{m}$ .

6.5 *Printer*, to record details of the measurements and results.

## 7. Reagents and Materials

7.1 *Verification and Calibration Fluids*<sup>7</sup>—Containing ISO Medium Test Dust (MTD) as specified in specification **ISO 12103-A3**.

7.2 *Heptane*—Reagent grade filtered down to 0.45 µm.

7.2.1 Prepare the heptane by filtering through a 0.45 µm filter (see 6.4.1) contained in a filter apparatus (see 6.4) See **Note 8**. Store in a container prepared in accordance with 10.2. (**Warning**—Extremely flammable, health hazard.)

## 8. Sampling

8.1 Unless otherwise specified, take a sample of at least 100 mL in accordance with Practices **D4057**, **D5854**, **D4177**, or other comparable sampling practices.

8.2 It is essential to take a representative sample, but avoid power mixing as this can modify the particles, break up agglomerated particles and entrain air. (See 14.1.4 and **Note 7**.)

8.3 Use sample containers that are capable of transporting the sample without contamination. Examples of these are fully epoxy-lined metal or amber colored glass containers with a threaded cap, fitted with an inert liner, forming a seal with the container.

8.4 Prior to taking the sample, rinse the sample containers with the product to be sampled at least three times. Each rinse shall use product equal to 10 to 20 % of the container volume. A rinse shall include closing and shaking the container for a minimum of 5 s and then draining the product.

8.5 Do not fill the sample container more than 90 % full. Overfilling affects the preparation of the test specimen as specified in 10.1.

8.6 Ensure that any aliquots or sub-division of the sample results in representative samples being taken and remaining in the original sample container. **Note 7** and 10.1 recommend suitable procedures regarding this particle counting test method.

## 9. Preparation of Apparatus

9.1 Ensure that the APC<sup>2</sup> is set up according to the instrument manufacturer's operating instructions and the verification and calibration requirements stated in both Section 11 and **A1.1.4**.

9.2 Ensure that the mode of operation, specified for this test method by the manufacturer, is selected.

9.3 Clean the outside of the test specimen input tube before each test sequence, by washing the outside in clean heptane or another filtered solvent.

9.4 At the start of any daily testing regime, initiate a test sequence using filtered heptane.

9.5 If a test specimen is tested that has a  $\geq 4$  µm(c) measurement of over 20 000 particles per mL, perform a complete test sequence (two flushes and two measurements as shown in Section 12) using filtered heptane to clean and flush

the measurement cell and the inside of the connecting tubing before testing other test specimens.

## 10. Test Specimen Preparation

10.1 Gently shake the sample in its container, for at least a minute, sufficiently to ensure that a representative test specimen can be drawn into the test specimen container.

10.1.1 It is essential to take a representative test specimen, but avoid power mixing or vigorous mixing as this can modify the particles, break up agglomerated particles and entrain air. (See 14.1.4 and **Note 17**.)

**NOTE 7**—To achieve a consistent agitation, equivalent to “gentle shaking” of the sample, it is recommended to either: (a) tumble the sample container, by hand or using a suitable automated mechanical tumbler, end over end for a minimum of 60 revolutions at approximately 1 revolution per second; (b) invert the sample container back and forth for a minimum of 60 times at approximately 1 cycle per second; or (c) use a barrel roller and roll for a minimum of 60 rotations.

Other ways of gently shaking the sample may be used, provided a representative test specimen is achieved.

10.2 Use a clean test specimen container, or flush a test specimen container by rinsing the inside of the container three times with the sample to be tested. Each rinse shall use product equal to 10 to 20 % of the container volume. A rinse shall include closing and shaking the container for a minimum of 5 s and then draining the product. Alternatively, the test specimen container may be cleaned by washing thoroughly with filtered heptane (see 7.2) and then allowed to dry in a clean environment.

**NOTE 8**—The efficacy of cleaning of the test specimen container may be checked by testing a sample of filtered heptane (see 7.2), in the cleaned test specimen container; this should give a count of less than 50 counts for the  $\geq 4$  µm(c) measurement.

10.3 Immediately after gently shaking, pour the mixed sample into the test specimen container and fit a clean cap. Ensure that the test specimen container is less than 90 % full.

**NOTE 9**—Over shaken or mechanically stirred samples can result in finely dispersed micro bubbles forming that will be counted as solid particles. Test specimens given ultrasonic treatment can result in the break-up of agglomerated particles into smaller ones that can affect the particle counts.

## 11. Apparatus<sup>2</sup> Verification and Calibration

### 11.1 Verification:

11.1.1 Verify the correct operation of the APC at least every 6 months or more frequently if required by local quality controls, by using the verification fluid (see 7.1) in accordance with 11.1.1.1 and Section 12. The result obtained shall be equal to or less than  $R/\sqrt{2}$  of the measurement plus the uncertainty of the verification fluid, from the certified  $\geq 4$  µm(c) value of the verification fluid, where R is the reproducibility of the test. If the result obtained is not within this figure, ensure the sample preparation is in accordance with the manufacturers instructions, check the verification fluid's validity date, and run a further test using the filtered heptane to confirm that the inlet tube and cell assembly are free from contaminants. Then repeat the verification. If the result is still not within the allowed tolerance, contact the manufacturer.

<sup>7</sup> The sole source of verification and calibration fluids known to the committee at this time is Stanhope-Seta, London Street, Chertsey, Surrey KT16 8AP UK. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration of a meeting of the responsible technical committee<sup>1</sup> which you may attend.

11.1.1.1 Follow the manufacturer’s instructions to prepare the verification fluid.

NOTE 10—Failure to correctly precondition the verification material can result in particle counts not meeting the verification criteria specified in 11.1.1 and 11.1.1.1.

11.2 *Calibration:*

11.2.1 The APC shall be calibrated according to **ISO 11171** at least every 12 months or more frequently if required by local quality controls or by the manufacturer.

11.2.1.1 Follow the manufacturer’s instructions regarding factory calibration or on site calibration using integral auto, self checking, calibration software.

11.2.2 The test specimen flow rate through the measurement cell shall be the same for calibration, verification and testing.

11.2.3 After calibration, verify the correct operation of the APC in accordance with 11.1.

**12. Procedure**

12.1 Before commencing a test, gently shake the test specimen, as prepared in 10.1-10.3, for a minimum of 1 min and then allow approximately 60 s before immediately starting the test. If the test is not started within approximately 90 s after shaking, gently re-shake the test specimen for a further minute.

NOTE 11—This 60 to 90 s includes time for fitting the test specimen input tube in place.

NOTE 12—To achieve a consistent agitation equivalent to “gentle shaking” of the test specimen it is recommended to either: (a) tumble the test specimen container, by hand or using a suitable automated mechanical tumbler, end over end for a minimum of 60 revolutions at approximately 1 revolution per second, or (b) invert the test specimen container back and forth for a minimum of 60 times at approximately 1 cycle per second.

12.2 It is permissible to test the sample directly from the sample container if the sample container, container closure and sample volume allow the test specimen to be drawn by the automatic particle counter, and the sample has been gently shaken as described in 10.1. However the remaining sample could then be unsuitable for carrying out other types of test methods due to possible cross contamination.

NOTE 13—Testing directly from the sample container reduces the possibility of introducing particle contamination into the test specimen.

12.3 Ensure the cleaned test specimen input tube is sufficiently below the level of the fuel to allow enough fuel (80 mL) to be used for the test sequences.

12.4 Ensure that the test specimen input tube is not touching the sides and is not within 10 mm of the bottom of the test specimen container, to avoid potential blockages.

12.5 Start the test sequence (see 12.1) in accordance with the manufacturer’s instructions. **Table 1** shows the test sequence.

12.6 The flow rate of the test specimen shall be fixed and not adjustable by the operator, and shall be the same for testing, verification and calibration.

12.7 The pump draws 30 mL of the test specimen through the test specimen input tube and directly into the optical measurement cell to flush and clean. One 10 mL test specimen is then drawn into the optical measurement cell for measurement. Record the measurements which shall be per mL.

12.8 Immediately, starting within approximately 15 s of the completion of the first test measurement, repeat the 30 mL flush and 10 mL test measurement.

12.9 Average the two  $\geq 4 \mu\text{m(c)}$  measurements from 12.7 and 12.8. If either of the criteria described in 12.9.1 and 12.9.2 are met, then the result is the average of each of the particle count size bands.

12.9.1 The difference between the two  $\geq 4 \mu\text{m(c)}$  measurements (per mL) from 12.7 and 12.8 is less than 10 % of their average.

12.9.2 The difference between the two  $\geq 4 \mu\text{m(c)}$  measurements (per mL) from 12.7 and 12.8 is less than 200 particles.

12.10 If the difference between the two  $\geq 4 \mu\text{m(c)}$  measurements from 12.7 and 12.8 does not meet either of the criteria described in 12.9.1 and 12.9.2, repeat the 30 mL flush and 10 mL measurement once or twice more until either criteria is met. If it is not possible to meet either criteria or if there is not enough test specimen, report the last measurement (12.8) and the non conformance, or repeat the whole test sequence with a fresh test specimen.

NOTE 14—Example: If the two  $\geq 4 \mu\text{m(c)}$  measurements from 12.7 and 12.8 are 500 particles and 600 particles, 10 % of their average would be 55 particles. As the difference between the measurements is 100 particles then the criteria in 12.9.1 would not be met, however as the difference between the two measurements is less than 200 particles, the second criteria in 12.9.2 is met and the result will be the average of the two particle counts viz 550 for the  $\geq 4 \mu\text{m(c)}$  size band.

NOTE 15—Filtered heptane (see 7.2) or another suitable solvent may be tested between test specimen testing to check that the APC and its test specimen delivery tube are free of contamination remaining from the previous tests.

12.11 Follow the manufacturer’s instructions regarding procedures when switching off the APC.

**13. Report**

13.1 Report the following information:

13.1.1 A reference to this test method.

**TABLE 1 Automated Test Sequence**

| Process                    | Time, seconds | Shake  | Prepare | Flush  | Count | Flush  | Count |
|----------------------------|---------------|--------|---------|--------|-------|--------|-------|
| Gently shake test specimen | >60           | xxxxxx |         |        |       |        |       |
| Prepare apparatus          | 60-90         |        | xxxxxx  |        |       |        |       |
| 30 mL flushing             | 60            |        |         | xxxxxx |       |        |       |
| 10 mL measurement          | 20            |        |         |        | xx    |        |       |
| Record measurements        | <15           |        |         |        |       | x      |       |
| 30 mL flushing             | 60            |        |         |        |       | xxxxxx |       |
| 10 mL measurement          | 20            |        |         |        |       |        | xx    |
| Record measurements        | <15           |        |         |        |       |        | x     |