



Designation: D7619 – 22

# 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.

*This standard has been approved for use by agencies of the U.S. Department of Defense.*

## 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 and military specification fuels falling within the scope of this test method include Specifications: D975 grades 1D and 2D, D1655, D3699, D4814 (see 14.1.1.1), D6751, D7467, distillate grades of D396 and D2880, MIL-DTL-83133, and MIL-DTL-16884.

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 sizes 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  $\geq 14 \mu\text{m(c)}$ . All measurements are per millilitre.

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, health, and environmental 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, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.14 on Stability, Cleanliness and Compatibility of Liquid Fuels.

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<sup>2</sup> The sole source of the apparatus known to the committee at this time is SETA-AvCount, available from 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.

1.4 *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:<sup>3</sup>

- D396 Specification for Fuel Oils
- D975 Specification for Diesel Fuel
- D1655 Specification for Aviation Turbine Fuels
- D2880 Specification for Gas Turbine Fuel Oils
- D3699 Specification for Kerosine
- 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
- 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, Liquid Fuels, and Lubricants
- D6751 Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels
- D7467 Specification for Diesel Fuel Oil, Biodiesel Blend (B6 to B20)

### 2.2 ASTM Adjuncts:<sup>4</sup>

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

<sup>3</sup> 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.

<sup>4</sup> Out of print.

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

### 2.3 ISO Standards:<sup>5</sup>

**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)

### 2.4 U.S. Dept. of Defense Specifications:<sup>6</sup>

**MIL-DTL-83133** Turbine Fuel, Aviation, Kerosene Type, JP-8 (NATO F-34), NATO F-35, and JP-8+100 (NATO F-37)

**MIL-DTL-16884** Fuel, Naval Distillate

### 2.5 Energy Institute Standard:<sup>7</sup>

**IP 565** Determination of the level of cleanliness of aviation turbine fuel—Portable automatic particle counter method

## 3. Terminology

### 3.1 Definitions:

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

### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *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.

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

3.2.2.1 *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 millilitre.

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

3.2.4 *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.

3.2.5 *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  $\geq 14 \mu\text{m}(c)$ ,

3.2.5.1 *Discussion*—Automatic particle counters may also count the total number of particles per mL, in size bands, in addition to those in 3.2.5, up to  $\geq 100 \mu\text{m}$ .

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

3.2.7 *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 measurement 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 8**.)

5.7 This test method can also be used to estimate the total particulate counts excluding free water droplets in aviation turbine fuels by a chemical pretreatment of the fuel. See **Appendix X2**.

## 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

<sup>5</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

<sup>6</sup> Copies of these documents are available online at <https://assist.dla.mil/quicksearch/> or <http://assistdocs.com/> or from the Standardization Document Order Desk, 700 Robbins Avenue, Building 4D, Philadelphia, PA 19111-5094.

<sup>7</sup> Available from Energy Institute, 61 New Cavendish St., London, W1G 7AR, U.K., <http://www.energyinst.org>.

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>8</sup>—Containing ISO Medium Test Dust (MTD) as specified in specification ISO 12103-A3.

7.2 *Heptane*—Reagent grade filtered down to 0.45  $\mu\text{m}$ .

7.2.1 Prepare the heptane by filtering through a 0.45  $\mu\text{m}$  filter (see 6.4.1) contained in a filter apparatus (see 6.4) See 10.2.1. 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 [10.1.2](#).)

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

<sup>8</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.

the original sample container. [10.1.2](#) 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  $\geq 4 \mu\text{m(c)}$  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](#).)

10.1.2 Consistent agitation can be achieved by either: (a) tumbling 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) inverting the sample container back and forth for a minimum of 60 times at approximately 1 cycle per second; or (c) using a barrel roller and roll for a minimum of 60 rotations. Other ways of gently shaking the sample can 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.

10.2.1 The efficacy of cleaning of the test specimen container can 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 100 counts for the  $\geq 4 \mu\text{m(c)}$  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 7—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  $\geq 4 \mu\text{m(c)}$  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.

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

NOTE 8—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 9—This 60 s to 90 s includes time for fitting the test specimen input tube in place.

12.1.1 Consistent agitation can be achieved by either: (a) tumbling 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) inverting the test specimen container back and forth for a minimum of 60 times at approximately 1 cycle per second. Other ways of gently shaking the sample can be used provided a representative test specimen is achieved.

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 10—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.

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           |        |         |        |       |        |       |

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 11—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 12—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.

13.1.2 The sample identification.

13.1.3 The date of the test.

13.1.4 Particle size cumulative count for at least  $\geq 4 \mu\text{m(c)}$ ,  $\geq 6 \mu\text{m(c)}$ , and  $\geq 14 \mu\text{m(c)}$  all per mL.

13.1.5 ISO coding, according to ISO 4406 for  $\geq 4 \mu\text{m(c)}$ ,  $\geq 6 \mu\text{m(c)}$ ,  $\geq 14 \mu\text{m(c)}$ . (See Appendix X1.)

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

13.1.7 In cases of dispute also report the instrument model used and software version installed.

### 14. Precision and Bias

14.1 *The Precision Study:*

14.1.1 The precision values given in 14.2 and 14.3 were derived from a 2006 multi-product laboratory study which used 5 similar instruments and 5 operators at a single location, to test 15 samples in duplicate and in random order. The sample types comprised automotive diesel, biodiesel, biodiesel blend, gas oil, kerosine (Aviation Turbine Fuels Jet A-1 and JP-8), marine diesel, and gasoline. One of the samples was found to be over the coincidence error limit (see 3.2.1) and hence the results for this sample may not be representative and this sample was not included in the statistical analysis. A further sample was discarded as the particles were found to be unstable.

14.1.1.1 The two gasoline samples used in the ILS were not gasoline-alcohol blends.

14.1.2 The viscosity range of the samples used in the laboratory study was  $0.7 \text{ mm}^2/\text{s}$  to  $4.6 \text{ mm}^2/\text{s}$  at  $40^\circ\text{C}$ . Viscosities outside this range may be tested but the precision could be affected. Automatic particle counters can usually measure samples with much higher viscosities.

14.1.3 The precision was obtained by statistical examination of laboratory test results according to Practice D6300 using ADJ6300 D2PP. The degrees of freedom required ( $>30$ ) by Practice D6300-07 were met, however Practice D6300-07 now requires a minimum of 6 labs (if satisfactory pilot program data is not available) whereas only 5 participated in this 2006 interlaboratory study. (Practice D6300-06 only required a minimum of 5 labs.) Full details of the statistical analysis are given in the research report.<sup>9</sup>

14.1.4 As the precision was determined from results obtained at a single location the reproducibility may not be comparable when results obtained at different times and locations are compared, due to sampling, shipping, storage and environmental factors. In practice two results obtained from different locations would be acceptable if their difference did not exceed the published reproducibility. In the event of a dispute or concern regarding the particle counts of a sample, it is recommended that freshly obtained samples are tested by both parties at the bulk storage location using the same sampling procedures. This ensures that nominally identical samples are tested by either or both parties and the precision shown in 14.2 and 14.3 shall apply.

NOTE 13—The test samples used in the ILS were prepared and subdivided with care to ensure that they were nominally identical as required by 14.2 and 14.3. Sampling techniques that do not ensure the sample is representative could result in different results at different locations (see 14.1.4).

NOTE 14—Appendix X1 shows precision in tabular and graphical form relative to particle counts and ISO codes.

14.2 *Repeatability, r*—The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the value given in only one case in 20. Table 2 shows the repeatability and applicable ranges for particle sizes (cumulative count)  $\geq 4 \mu\text{m(c)}$ ,  $\geq 6 \mu\text{m(c)}$ , and  $\geq 14 \mu\text{m(c)}$ .

14.3 *Reproducibility, R*—The difference between two single and independent test results, obtained by different operators using different apparatus on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the value given in only one case in 20. Table 2 shows the reproducibility and applicable ranges for particle sizes (cumulative count)  $\geq 4 \mu\text{m(c)}$ ,  $\geq 6 \mu\text{m(c)}$ , and  $\geq 14 \mu\text{m(c)}$ .

14.4 *Bias*—This test method has no bias because the results of the test are defined only in terms of this test method.

<sup>9</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1696. Contact ASTM Customer Service at service@astm.org.

TABLE 2 Precision and Range for 1 mL

| Parameter Size Bands     | Range of Results | Repeatability, r | Reproducibility, R |
|--------------------------|------------------|------------------|--------------------|
| $\geq 4 \mu\text{m(c)}$  | 369-49633        | $0.6095X^{0.85}$ | $0.8542X^{0.85}$   |
| $\geq 6 \mu\text{m(c)}$  | 82-14013         | $0.6095X^{0.85}$ | $0.8542X^{0.85}$   |
| $\geq 14 \mu\text{m(c)}$ | 4-3568           | $0.74X^{0.75}$   | 0.5846X            |

where: X is the average of results being compared

## 15. Keywords

15.1 automatic particle counting; automotive diesel; bio-diesel; fuel cleanliness; gas oil; gasoline; kerosine; marine diesel

## ANNEX

### (Mandatory Information)

#### A1. AUTOMATIC PARTICLE COUNTING APPARATUS (APC)

A1.1 *General*—The apparatus is self contained and automatic, with microprocessor control of the pump, change-over valve and sensor for measurement and flushing sequences. Fig. A1.1 shows the configuration of the APC.

A1.1.1 *Principle*—The APC operates on the light obscuration principle and is suitable for counting and sizing particles within the range of  $4\ \mu\text{m(c)}$  to greater than  $14\ \mu\text{m(c)}$ .

A1.1.2 Cumulative results are provided for at least the  $\geq 4\ \mu\text{m(c)}$ ,  $\geq 6\ \mu\text{m(c)}$  and  $\geq 14\ \mu\text{m(c)}$  size bands and include the associated ISO 4406 codes. All results are per mL. Cumulative results for other bands up to  $\geq 100\ \mu\text{m}$  may be provided but precision for these other bands has not been determined.

A1.1.3 The APC apparatus has a coincidence error limit of 60 000 particles/mL as defined in 3.2.1 and as measured in ISO 11171. Counts at least as high as 100 000 are possible but the count accuracy and precision will be affected for particle counts over 60 000.

A1.1.4 Calibration shall be in accordance with ISO 11171 (see 11.2).

A1.1.5 Consult the manufacturer's APC specification to ensure the seals and tubing are compatible with the sample type

to be tested. For example samples such as Fatty Acid Methyl Esters (FAME) are known not to be compatible with some types of rubber and other materials.

A1.1.6 Results (per mL) shall be recorded electronically and/or printed (see 6.5).

A1.2 *Automatic Changeover Valve*—To enable the sample to flow through the optical measurement cell in a continuous way, in conjunction with the double pump, from input to output.

A1.3 *Double Pump*—Fixed volume (10 mL nominal per each stroke) driven by a constant speed motor under microprocessor control to ensure a fixed volume flows through the measurement cell at a constant nominal flow rate of 30 mL/min.

A1.4 *Laser*—5 mW laser diode light source, wavelength  $670\ \text{nm} \pm 5\ \text{nm}$ .

A1.5 *Test Specimen Delivery Tube Assembly:*

A1.5.1 Flexible transparent tubing for connection to the input connector.

A1.5.2 Adjustable length adaptor to enable the use of different dimension test specimen containers.

A1.5.3 Test specimen container cap to avoid dust ingress.

A1.6 *Calibration Software*—Integral auto software, with self checking facility, to enable on-site calibration in conjunction with the verification and calibration fluids (see 7.1) and the manufacturer's instructions.

A1.7 *Pre-Measurement Checks*—The following checks shall be carried out automatically:

A1.7.1 Laser source operation and intensity.

A1.7.2 Sensor status.

A1.7.3 Power supply voltages.

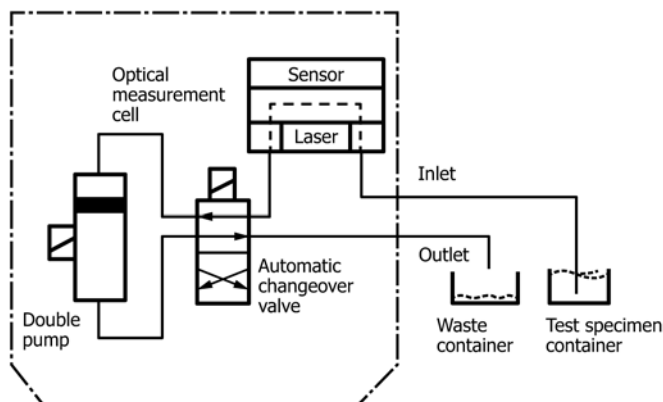


FIG. A1.1 Automatic Particle Counter

**APPENDIXES**
**(Nonmandatory Information)**
**X1. TABULAR AND GRAPHICAL REPRESENTATION OF PRECISION**

NOTE X1.1—Tables X1.1-X1.3 show the range of particle counts per mL represented by the ISO codes and the precision per mL (r repeatability and R reproducibility) for the minimum and maximum numbers of particles represented by each ISO code number.

NOTE X1.2—ISO Code 18/16/13 indicates that there are 1301 to 2500 particles of  $\geq 4 \mu\text{m(c)}$  (see Table X1.1), 321 to 640 particles of  $\geq 6 \mu\text{m(c)}$  (see Table X1.2) and 41 to 80 particles of  $\geq 14 \mu\text{m(c)}$  (see Table X1.3).

**TABLE X1.1 Tabulated Precision for  $\geq 4 \mu\text{m(c)}$** 

| $\geq 4 \mu\text{m(c)}$<br>Particles | ISO<br>CODE | r<br>Particle Counts | R<br>Particle Counts |
|--------------------------------------|-------------|----------------------|----------------------|
| 321–640                              | 16          | 82–148               | 115–207              |
| 641–1300                             | 17          | 148–270              | 208–379              |
| 1301–2500                            | 18          | 270–471              | 379–660              |
| 2501–5000                            | 19          | 471–849              | 661–1190             |
| 5001–10000                           | 20          | 850–1531             | 1191–2146            |
| 10001–20000                          | 21          | 1531–2760            | 2146–3868            |
| 20001–40000                          | 22          | 2760–4974            | 3868–6971            |
| 40001–80000                          | 23          | 4974–8966            | 6971–12566           |

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