Designation: D8166 – 22

Standard Test Method for Sizing and Counting Particulates in Middle Distillate Fuels and Biodiesel Blend (B6 to B20) Using Continuous Flow and Bottle Sampler Particle Contamination Monitors[1]

This standard is issued under the fixed designation D8166; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method uses specific particle contamination monitors (PCMs) to count and measure the size of dispersed dirt particles, water droplets and other particulates, in middle distillate fuel, in the overall range from 4 µm to 70 µm and in the size bands ≥4 µm, ≥6 µm, ≥14 µm, and ≥30 µm.

NOTE 1—The term particle contamination monitor, as used in this test method, is the same as that defined in ISO 21018-4; an instrument that automatically measures the concentrations of particles suspended in a fluid at certain sizes and cannot be calibrated in accordance with ISO 11171 whose output may be as a particle size distribution at limited sizes or as a contamination code.

1.2 This test method has interim repeatability precision only, see Section 14 for more information.

NOTE 2—ASTM specification fuels falling within the scope of this test method include Specifications: D975, D1655, D3699, D7467, MIL-DTL-83133, MIL-DTL-5624, and distillate grades of D396 and D2880.

NOTE 3—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 microbial particulates. Although the projected area of a particle is measured, this is expressed as the diameter of a circle for the purposes of this test method. The detector is unable to distinguish between dirt and water particles.

NOTE 4—This test method may be used for particle sizes bands up to 70 µm, however the interim repeatability has only been determined for the size bands ≥4 µm, ≥6 µm, and ≥14 µm. All measurements are counts per millilitre.

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

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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
D4306 Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination
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
D7467 Specification for Diesel Fuel Oil, Biodiesel Blend (B6 to B20)
D7619 Test Method for Sizing and Counting Particles in Light and Middle Distillate Fuels, by Automatic Particle Counter

2.2 *U.S. Dept. of Defense Specifications:*[3]

MIL-DTL-5624 Specification: Turbine Fuel, Aviation, Grades JP-4 (NATO F-40 and JP-5 (NATO F-44)

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.14 on Stability, Cleanliness and Compatibility of Liquid Fuels.

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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 online at ASSIST Quick Search (http://quicksearch.dla.mil).

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

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

2.3 *ISO Standards:*[4]

ISO 11171 Hydraulic fluid power—Calibration of automatic particle counters for liquids

ISO 11943 Hydraulic fluid power — Online automatic particlecounting systems for liquids — Methods of calibration and validation

ISO 12103 Road vehicles—Test contaminants for filter evaluation—Part 1: Arizona test dust

ISO 21018–4 Hydraulic fluid power — Monitoring the level of particulate contamination in the fluid —Part 4: Use of the light extinction technique

2.4 *Energy Institute Test Methods:*[5]

IP 564 Determination of the level of cleanliness of aviation turbine fuel – Automatic particle condition monitor

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

IP 577 Determination of the level of cleanliness of aviation turbine fuel – Automatic particle counter method using light extinction

3. Terminology

3.1 For definition 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 12103-1, A.1) that can be counted with less than a 5 % error resulting from the presence of more than one particle in the sensing volume at a time.

3.2.2 *particle count, n*—the sum of the number of solid particles and dispersed water droplets counted by this test method.

3.2.3 *particle size, µm, n*—the diameter of the circle of an area equivalent to that of the projected area of the particle passing through the detecting cell.

3.2.3.1 *Discussion*—Particles are not generally spherical in shape. The shadow cast by a particle on the detector will nevertheless have a measurable area. The particle size is here defined as the diameter of that circle equal in area to this projected area.

3.2.4 *particle size cumulative count, n*—the total number of particles per millilitre, in size bands, ≥4 µm, ≥6 µm, ≥14 µm, and ≥30 µm.

3.2.4.1 *Discussion*—The cumulative count in each (increasing) size band includes the counts from all larger sizes but excludes the counts in all previous (smaller) size bands. PCMs used in this method can also count the total number of particles per millilitre in other size bands additional to those indicated in 3.2.4, up to ≥70 µm.

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

3.3 *Abbreviations:*

3.3.1 *ACFTD*—air clean fine test dust

3.3.2 *MTD*—medium test dust

3.3.3 *PCM*—particle contamination monitor

3.3.4 *UFTD*—ultra fine test dust

4. Summary of Test Method

4.1 The optical measurement cell is comprised of 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 channels.

4.2 The principle of operation (4.1) for PCMs is the same for both continuous flow (Procedure A) and bottle sampler (Procedure B) PCMs. The instruments differ in the test specimen volume and the mode of operation (manual for continuous flow PCM, and automatic for bottle sampler PCM) (4.2) The only manual operation required by continuous flow PCM is during the initiation of each of the determinations, by turning a knob.

4.2.1 *Continuous Flow PCM*—The test specimen is manually mixed in its container to suspend the particles. Upon initiation of a test, the PCM draws the test specimen directly from a test specimen container (see Fig. A1.1). The test sequence is started manually. The test sequence comprises of four manual initiations. The first initiation and resulting determination is used as a flush. The optical measurement cell is flushed with 10 mL test specimen. The remaining three determinations, each initiated manually and using 10 mL of test specimen, are averaged to give the test result. PCM counts particles in the specified size bands.

4.2.2 *Bottle Sampler PCM*—The test specimen is manually mixed in its container to suspend the particles. Upon initiation of a test, the PCM draws the test specimen directly from a test specimen container (see Fig. A1.2). The test sequence commences by flushing the optical measurement cell and internal tubing known as the dead volume. This is immediately followed by the test of 25 mL test specimen. The PCM counts particles in the specified size bands. This is repeated automatically two more times. The results are averaged as in Procedure A.

4.3 Obtaining a representative sample and following the recommended sample and test specimen preparation procedures and timescales are particularly important with particle counting methods (see Sections 8 and 10).

5. Significance and Use

5.1 This test method is intended for use in the laboratory or in the field to evaluate the cleanliness of distillate fuels, and

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

[5] Available from Energy Institute, 61 New Cavendish St., London, W1G 7AR, U.K., http://www.energyinst.org. Available online at http://publishing.energyinst.org/ip-test-methods

liquid biofuels, such as biodiesel and diesel blends. This specific test method and the precision statement applies to off-line analysis.

NOTE 5—These PCMs can be used for high pressure on-line applications as well, however the repeatability (r) and reproducibility (R) for on-line application were not established.

5.2 An increase in particulate counts can indicate a change in the fuel condition caused for example by contamination during storage or transfer. Potential causes of particulates formation during storage could be "fuel-degradation products," as described in Specification D975, Appendix X3.

5.3 High levels of particles can cause filter blockages (especially when the particles are close in size to the filter porosity rating) 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.

5.4 This test method specifies a minimum requirement for reporting measurements in particle size bands (A1.2.1). Some specific applications may require measurements in other particle size bands. The particle count from the test should be carefully interpreted by the user as it can potentially over-state risk of abrasive damage or filter blocking due to counting water droplets as well as hard dirt particles.

5.5 In situations where there is a requirement for the calibration of the apparatus to be solely in accordance with ISO 11171, Test Methods D7619, IP 565, or IP 577 may be used.

6. Apparatus

6.1 *Particle Contamination Monitor (PCM)*[6]—Operating on the laser light obscuration principle, comprising an optical measurement cell, bi-directional pump, electronics, and software to analyze the test specimen, and display and print the particle measurement data (see ISO 21018 and Annex A1).

6.2 *Test Specimen Container,* cylindrical, made of glass or other suitable material, of at least 400 mL volume for Procedure A with provision for holding the test specimen input tube at least 10 mm above the bottom of the container, or 200 mL volume for Procedure B, and a cap with a suitable inert internal seal.

NOTE 6—Some containers cause particles to adhere to the walls of the container due to static electricity effects.

6.3 *Waste Container,* for collecting the tested test specimen and filtered solvent used for flushing.

6.4 *Filter Apparatus,* general purpose for filtering solvents.

6.4.1 *Filters,* cellulose, glass fiber or polycarbonate membranes, rated at 0.45 µm.

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

[6] The sole source of supply of the apparatus known to the committee at this time is Parker Hannifin Corporation, Hydraulic and Fuel Filtration Division, Colorado Springs, CO 80907. 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.

7. Reagents and Materials

7.1 *Verification and Calibration Fluids*[7]—containing ISO Medium Test Dust (MTD) as specified in specification ISO 12103-1, A.3.

7.2 *Shop Air*—optional for bottle sampler (Procedure B). (**Warning—**High pressure, health hazard.)

7.3 *Isopropanol*—reagent grade filtered down to 0.45 µm.

7.3.1 Prepare the isopropanol by filtering through a 0.45 µm filter (6.4.1) contained in a filter apparatus (6.4). Store in a container prepared in accordance with 10.2.

7.4 *Isopropanol*—HPLC grade, required only for procedure in Appendix X1. (**Warning—**Flammable, health hazard.)

8. Sampling

8.1 Unless otherwise specified, take a sample of at least 400 mL in accordance with Practices D4057, D4177, or other comparable sampling practices.

8.2 If collecting field samples, use sample containers that are capable of transporting the sample without contamination, follow Practice D4306. 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. For additional guidance on handling samples and transport containers consult Practice D5854.

8.3 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.

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

9. Preparation of Apparatus

9.1 Ensure that the PCM is set up according to the instrument manufacturer's operating instructions and the verification and calibration requirements stated in both Sections 11 and A1.2.3 have been performed.

9.2 Clean the outside of the test specimen input tube before each test sequence by washing the outside in clean isopropanol.

9.3 If a test specimen reports a ≥4 µm measurement of over 20 000 particles per millilitre, perform several test sequences using filtered isopropanol to clean and flush the measurement cell and the dead volume inside the unit before testing other test specimens.

10. Test Specimen Preparation

10.1 To prepare the test specimen, gently rotate the sample container end-over-end for 1 min at approximately 1 r/s by hand, or use a suitable automated mechanical agitator, being careful not to form air bubbles. See Note 7. This ensures that a representative test specimen can be drawn into the test specimen container.

[7] The verification and calibration fluids are available from the instrument manufacturer.

10.2 Use a clean test specimen container, or flush the test specimen container by rinsing the inside of the container three times with the sample to be tested. Each rinse should 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 product. Alternatively, the test specimen container may be cleaned by washing thoroughly with filtered isopropanol (7.3.1) and then allowed to dry in a clean environment.

10.3 The efficacy of cleaning of the test specimen container can be checked by testing a sample of filtered isopropanol (see 7.3.1), in the cleaned test specimen container; this should give a count of less than 200 counts/mL for the ≥4 µm measurement. If less than 200 counts/mL are not achieved after several runs, then re-filter the isopropanol and recheck.

10.4 After rinsing the test specimen container, re-agitate the sample by gently rotating the sample container end-over-end for 1 min at approximately 1 r/s by hand, or use a suitable automated mechanical agitator and immediately pour the re-agitated sample into the test specimen container. Ensure that the test specimen container is less than 90 % full.

NOTE 7—Over-shaken or mechanically stirred samples can result in entrain air bubbles 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 distribution.

11. Apparatus Verification and Calibration

11.1 *Verification:*

11.1.1 Follow the manufacturer's instructions to prepare verification fluid.

11.1.2 Verify the correct operation of the PCM 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 and 12.1 or 12.2. The result obtained shall be within the limits stated on the verification fluid certificate for the reported ≥4 µm channel count. If the result obtained is not within this figure, ensure the sample preparation is in accordance with the manufacturer's instructions, check the verification fluid's validity date, and run a further test using the filtered isopropanol 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.

NOTE 8—Failure to correctly precondition the verification material can result in particle counts not meeting the verification criteria published by the manufacturer.

11.2 *Calibration:*

11.2.1 The PCM shall be calibrated by the manufacturer, or manufacturer's appointed agent, according to ISO 21018 Section 7 procedures, on an ISO 11943 compliant test rig. Calibration shall be performed at least every 12 months minimum or more frequently if required by local quality controls or by the manufacturer.

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

12. Procedure

12.1 *Procedure A for Continuous Flow PCM:*

12.1.1 Immediately before commencing a test, gently rotate the specimen container end-over-end for 1 min at approximately 1 r/s by hand, or use a suitable automated mechanical agitator then immediately initiate the test as per manufacturer's instructions. If the test is not started within approximately 90 s after agitation, gently repeat sample rotation for a further minute.

12.1.2 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 agitated as described in 10.1, the test specimen may be drawn directly from the sample container, however the remaining sample could then be unsuitable for carrying out other types of test methods due to possible cross-contamination.

NOTE 9—Testing directly from the sample container reduces the possibility of introducing local contamination into the test specimen.

12.1.3 Ensure the cleaned test specimen input tube is sufficiently below the level of the fuel to allow enough fuel to be used for the entire test sequence, which requires 400 mL.

12.1.4 Enter sample identification as required.

12.1.5 Initiate the test in accordance with the manufacturer's instruction.

12.1.6 Conduct four determinations manually. Ignore the results of first determination and record the results of the subsequent three determinations in sequence. Average these three determinations to generate the result for the test specimen.

12.1.7 Once the test runs have been completed, the results can be collated and printed off the internal printer or downloaded to a desktop or laptop computer.

12.1.8 If the previous sample tested gave a result of more than 20 000 particles per millilitre at 4 µm band, flush the system with filtered isopropanol (7.3.1) by following 12.1.3 to 12.1.7 prior to testing the next sample. For sample identification mark it as "blank" to distinguish it from the test data. Carrying out this "blank" test will avoid cross-contamination between samples (see Note 10).

NOTE 10—Filtered isopropanol (7.3.1) may be tested between test specimen testing to check that the PCM and its test specimen delivery tube are free of contamination remaining from the previous tests.

12.1.9 Follow the manufacturer's instructions regarding procedures when switching off the PCM.

12.2 *Procedure B for Bottle Sampler PCM:*

12.2.1 Program PCM in accordance with the manufacturer's instructions.

12.2.2 Enter sample identification as required.

12.2.3 Immediately before commencing a test, gently rotate the specimen container end-over-end for 1 min at approximately 1 r/s by hand, or use a suitable automated mechanical agitator.

12.2.4 Place the specimen container inside the sample chamber and close the door.

12.2.5 Immediately after closing the door start the test sequence. If the test sequence is not started within approximately 90 s after agitation, gently repeat specimen rotation for a further minute (12.2.3).

12.2.6 The PCM will automatically carry out four determinations. The first determination is a flush and the result will be discarded. The following three determinations are recorded in sequence. The instrument averages these three determinations and this average becomes the final result reported for the test specimen.

12.2.7 Once the test runs have been completed, the results can be collated and printed off the internal printer or downloaded to a desktop or laptop computer.

12.2.8 If the previous sample tested gave a result of more than 20 000 particles per millilitre at 4 µm band, flush the system with filtered isopropanol (7.3.1) by following 12.2.2 to 12.2.6 prior to testing the next sample. For sample identification mark it as "blank" to distinguish it from the test data. Carrying out this "blank" test will avoid cross-contamination between samples (see Note 10).

12.2.9 Follow the manufacturer's instructions regarding procedures when switching off the PCM.

13. Report

13.1 Report the following information:

13.1.1 Reference to this test method.

13.1.2 Procedure A or Procedure B.

13.1.3 The sample identification.

13.1.4 The date of the test.

13.1.5 The sample temperature.

13.1.6 Particle size cumulative count for at least ≥4 µm, ≥6 µm, ≥14 µm and ≥30 µm (optional) all per millilitre.

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

13.1.8 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 Only an interim precision statement is available at this time. A full ILS will be completed by 2022.

14.1.2 The precision values given in 14.2 were derived from a 2016 interim repeatability study. As per Practice D6300, subsection 6.2.1, the study was conducted using two instruments for Procedure A and two instruments for Procedure B, 12 replicates under repeatability conditions and one operator. The sample types comprised automotive diesel, biodiesel blend (B20), and Aviation Turbine Fuel Jet A-1.

14.1.3 The study was conducted at laboratory setting with room temperature maintained at 19 °C to 21 °C.

14.1.4 The precision was obtained by statistical examination of laboratory test results according to Practice D6300, outlier identification was as per GESD for each sample. Practice D6300 ANOVA with no repeat was used for simultaneous analysis of all 9 samples for each of the three size bands (≥4 µm, ≥6 µm, and ≥14 µm). Based on the test results, it was determined that power transform was best suited for variance stabilization.

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.

14.2.1 Table 1 shows the repeatability and applicable ranges for particle sizes (cumulative count) ≥4 µm, ≥6 µm, and ≥14 µm for Procedure A. Table 2 shows the repeatability and applicable ranges for particle sizes (cumulative count) ≥4 µm, ≥6 µm, and ≥14 µm for Procedure B.

14.3 The full interlaboratory study (ILS) will be completed within five years of publication of this test method, at which time the interim repeatability values (Tables 1 and 2) will be updated with full repeatability and reproducibility (r&R) values for the test method as per Practice D6300.

15. Keywords

15.1 automatic particle counting; automotive diesel; aviation; biodiesel blends; fuel cleanliness; gas oil; kerosene; marine diesel; turbine fuel

TABLE 1 Interim Repeatability and Range for Procedure A (Continuous Flow PCM)

Parameter Size Bands	Range Results (counts/mL)	Repeatability, r^A
≥4 µm	58 to 18 129	$3.51X^{0.7}$
≥6 µm	16 to 7179	$2.45X^{0.7}$
≥14 µm	1 to 328	$0.88X^{0.8}$

[A] where X is the average of results being compared.