Designation: ~~D8049 – 19a~~ D8049 – 21

Standard Test Method for Determining Concentration, Count, and Size Distribution of Solid Particles and Water in Light and Middle Distillate Fuels by Direct Imaging Analyzer[1]

This standard is issued under the fixed designation D8049; 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 a direct imaging analyzer to count and measure the size and shape of dispersed solid particles and water droplets in light and middle distillate fuels in the overall range from 4 µm to 100 µm and in size bands of ≥4 µm, ≥6 µm, and ≥14 µm.

NOTE 1—Particle size data from 0.7 µm through 300 µm is available for use or reporting if deemed helpful.

NOTE 2—Shape is used to classify particles, droplets, and bubbles and is not a reporting requirement.

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.*

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:*[2]

D2276 Test Method for Particulate Contaminant in Aviation Fuel by Line Sampling
D3240 Test Method for Undissolved Water In Aviation Turbine Fuels
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
F658 Practice for Calibration of a Liquid-Borne Particle Counter Using an Optical System Based Upon Light Extinction (Withdrawn 2007)[3]

[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] The last approved version of this historical standard is referenced on www.astm.org.

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

2.2 *ISO Standard:*[4]
ISO 11171 Hydraulic Fluid Power—Calibration of Automatic Particle Counters for Liquids
ISO 12103-1 Road Vehicles—Test Contaminants for Filter Evaluation—Part 1: Arizona Test Dust
~~ISO 11171~~SRM 2806 (ISO Medium Test Dust in Hydraulic Oil): ~~Hydraulic Fluid Power—Calibration of Automatic Particle Counters for Liquids~~A Particle-contamination Standard Reference Material for the Fluid Power Industry

2.3 *MIL Standard:*[5]
MIL-PRF-5606 Hydraulic Fluid, Petroleum Base; Aircraft, Missile and Ordinance

3. Terminology

3.1 For definitions of terms used in this standard, refer to Terminology D4175.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *air bubble, n*—non-fuel, gaseous formations within the fuel, generally spherical in shape and visible as a heavy wall ring due to the diffraction of light around and through them.

3.2.2 *droplet, n*—non-fuel liquid formations within the fuel, generally spherical in shape and visible as a thin wall ring due to the diffraction of light around and through them.

3.2.3 *major particle diameter µm, n*—the maximum two-dimensional length of the particle measured.

3.2.4 *minor particle diameter µm, n*—the maximum two-dimensional length of the particle measured perpendicular to the *major particle diameter*.

3.2.5 *particle, n*—non-liquid, non-gaseous, solid objects in the fuel.

3.2.6 *projected equivalent particle diameter µm, n*—the diameter calculated from the projected area of a particle if that area formed a circle, and in equation form is:

$$\text{Projected Equivalent Particle Diameter} = \sqrt{(\text{area}/0.785)}$$

4. Summary of Test Method

4.1 The optical measurement cell comprises a light source and an optical sensor. The principle of operation is the illumination and digital capture of actual particle images which are then analyzed for size and shape by the system software. The visual capability of the instrument allows for the differentiation between solid, water, and air particles and thus the detection of water and elimination of air bubbles from the analysis.

4.2 This standard presents a preferred and an alternate method. In the preferred method fuel is delivered by a metering pump and in the alternate method fuel is delivered by gravity.

4.2.1 In the preferred method the test specimen is filled into an ultraclean specimen jar of approximately 100 mL volume. The specimen should fill the jar to the 80 % level. A clean suction tube is inserted into the container and fuel is delivered to the analyzer by the pump.

4.2.2 In the alternate method, the test specimen, approximately 4 L, is agitated in its container. The container is then fitted with a spigot to allow delivery to the analyzer. Fluid flows through the analyzer and is tested for solids and water content. Larger or smaller volume test specimen maybe used as appropriate for the instrument.

4.3 The method requires reporting of particle counts in the ≥4 µm, ≥6 µm, and ≥14 µm categories, however particle counts in the 0.7 µm to <4 µm size range may also be reported as well as additional ranges the user deems important. Particle size is determined per 3.2.6. Water volume content is also reported.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.
[5] For referenced MIL standards, visit the Defense Logistics Agency, Document Services website at http://quicksearch.dla.mil

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 fuels identified in the scope.

5.2 Detection of particles and water can indicate degradation of the fuel condition. Particles, whether inorganic or organic, can cause fouling of fuel filters and damage pumps, injectors, and pistons. Knowledge of particle size in relation to metallurgy can provide vital information, especially if the hardness of the solid particles are known from other sources.

NOTE 3—The method includes the detection of water, solids, and air bubbles. The air bubbles are screened out of the data prior to analysis of results, based on shape and transparency, and are not reported in the results.

6. Apparatus[6]

6.1 *Preferred ~~Configuration:~~Procedure:*

6.1.1 *Direct Imaging Analyzer*—Operating on visual imaging principles comprising a flow cell with camera and optics, a light source, a metering pump, test specimen container, instrument stand and software to analyze the test specimen and display the particle measurement data.

6.1.2 *Test Specimen Container*—A clean fuel container used to supply the metering pump with fuel for testing. ~~Recommend 100 mL bottles clean to a maximum of 2 particles per millilitre greater than 5 µm diameter~~

6.1.3 *Metering Pump,* capable of supplying fuel to the analyzer at a rate of ~~1 mL~~30 mL/min ~~to 100 mL~~ ± 1 mL/min ~~and controllable to ±1 mL/min~~ accuracy.

6.1.4 *Collection Container,* equivalent to test specimen container for capturing analyzed specimen.

6.2 ~~*Laboratory or Field Usage:*~~ *Alternate Procedure:*

6.2.1 *Direct Imaging Analyzer*—Operating on visual imaging principles, comprising a flow cell with camera/optics, light, test specimen container, and stand and software to analyze the test specimen and display the particle measurement data.

6.2.2 *Test Specimen Container*—A clean fuel container for sample storage, transport, and transfer into the instrument. An epoxy-lined container of approximately 5 L in volume has been found to be suitable, along with a nominal 19 mm or larger opening in the top lid for installation of a tube manifold assembly to allow fuel transfer to the instrument and air into the epoxy-lined container for venting.

6.2.3 *Tube Manifold Assembly*—Consists of a stopper or threaded cap, which inserts into the top opening in the test specimen container to seal it, and has through-holes which accept tubing for venting and tubing for flow of fuel to the instrument.

6.2.4 *Flow Restrictor*—The flow of fuel through the instrument is restricted by an orifice located in the outflow line to the collection container.

6.2.5 *Collection Container*—For collecting analyzed fuel specimen for possible retesting. Equivalent to the test specimen container.

7. Reagents and Materials

7.1 *Heptane*—~~Reagent-grade, filtered through 0.45 µm filter.~~Reagent-grade.

7.2 *Reticle*—~~NIST, or other widely recognized standards body, traceable, for calibration of system. A 19 mm diameter reticle~~ NIST traceable with 100 µm grids and 10 µm ~~subdivisions has been found to work well for use in calibrating the instrument.~~subdivisions.

[6] The sole source of supply of the apparatus known to the committee at this time is Jet Fuel InFlow available from J.M. Canty Inc, 6100 Donner Rd., Lockport, New York USA 14094. 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.

7.3 ~~*Partistan Resolution*~~*Particle Size Standard,* ~~mono disperse polymer beads,~~ monodisperse polymer beads with a nominal diameter between 8 µm and 12 µm, and a coefficient of variation <10 %.

7.4 ~~*Verification Standard Partistan 2806,*~~ *Particle Count Verification Standard,* ~~containing ISO Medium Test Dust, ISO 12103-A3 traceable to NIST.~~meeting NIST SRM 2806.

7.5 *Partistan Super Clean Fluid.*

8. Sampling

8.1 Sample into the test specimen container. Ensure it is new and unused, or in clean, new condition (see Practice D4306). Take precautions not to introduce contamination during the sampling process.

8.2 Take a representative sample. Refer to Practice D4057, Practice D4177, or other similar sampling practices.

8.3 Confirm that the container is approximately 80 % ~~filled (~4 L).~~filled.

9. Preparation of Apparatus

9.1 Ensure the instrument is set up according to manufacturer's instructions.

9.2 Ensure instrument and assembled components are clean and ready for use by flushing with ~~a filtered, fast-drying solvent such~~solvent (7.1 ~~as heptane. System cleanliness may~~). Cleanliness must be checked by running a sample of ~~filtered heptane~~solvent (7.1) through it. If the ~~test specimen has a ≥4 µm count in excess of 200 µm~~count in the ≥4 µm range~~/mL, the system~~ is greater than 200/mL, the instrument requires cleaning by continued flushing with ~~filtered heptane~~solvent (7.1) until the count falls below ~~200.~~200/mL.

10. Test Specimen Preparation

10.1 ~~Gently shake~~ Shake the test specimen in its container for ~~at least 1 min~~ 30 s to ensure that it is well mixed.

10.1.1 To achieve a consistent agitation ~~it is recommended to either: (a) tumble the test specimen container by hand or appropriate mechanical shaking device, (b)~~ invert the container back and ~~forth for a minimum of 60 times at approximately 1 Hz (cycle/second), or (~~forth, mechanically or bye~~) use a roller device and roll~~ hand, for a minimum of 60 ~~rotations. Other ways of gently shaking the container may be used provided a well-mixed test specimen is achieved.~~times at 2 Hz.

~~NOTE 4—Over-shaken or mechanically stirred samples can result in finely dispersed micro bubbles that may be counted as solid particles. Additionally, test specimens given ultrasonic treatment can result in the break-up of agglomerated particles into smaller ones that can affect the count.~~

11. Apparatus Verification and Calibration

11.1 Illumination level ~~should~~must be checked ~~daily~~ prior to use per manufacturer's operating manual.

11.2 *Calibration:*

11.2.1 ~~The instrument shall be calibrated per the manufacturer's operating manual. Calibration shall be done by referencing a reticle (see~~ A calibration reticle (7.2~~). Once calibrated, a direct imaging-type instrument remains in calibration as long as the camera and lens components remain unchanged and unmoved.~~) shall be installed in the analyzer light port opposite the camera per the manufacturer's operating manual.

11.2.2 The image of the reticle is captured and used to calculate the scale of the view.

11.2.3 The test specimen flow rate should be similar for calibration, verification, and operation. For gravity feed, an orifice is provided on the outflow of the analyzer to ensure this.

11.3 *Verification*—Verification shall be performed at least every six months.

11.3.1 *Particle Count*—Use the verification fluid ~~referenced in~~ (~~7.5~~7.4) to verify particle count. Test in accordance with Section 12. The per millilitre result obtained shall be ~~equal to or less than~~ within (*r*/1.414 + 2.8**S*~~of the measurement plus the uncertainty of the verification fluid from the~~) of the certified ≥4 µm(c) value of the ~~standard~~ verification fluid (7.4) where *r* is the repeatability of the test (see Section 14) and *S* is the standard deviation of the verification fluid (~~Appendix X1~~7.4)~~.~~) certified by the manufacturer in the ≥4 µm(c) range. If the result obtained is not within this figure, ensure the instrument and the sample preparation ~~is~~are in accordance with ~~the~~Sections 8~~manufacturer's~~, 9~~instructions,~~ , and 10 of this standard, check the verification ~~fluids~~fluid's validity date, and ~~run a further test using the filtered heptane~~flush the instrument per 9.2~~to confirm the inlet tube and cell assembly are free from contaminants. Repeat the verification. If the result is still not within the allowed tolerance, consult the operating manual or contact the manufacturer.~~.

NOTE 4—Failure to correctly precondition the verification fluid can result in particle counts not meeting the verification criteria specified by the fluid manufacturer.

11.3.2 *Particle Size*—Mono-disperse beads per 7.3~~, or similar,~~ shall be used to verify the operation of the instrument. ~~Dilute the beads, if required, to an appropriate volume using the super clean fluid. The result of the analysis shall be within 3 % of the average manufacturer certified particle size plus the specified standard deviation for the beads.~~

11.3.2.1 Add beads (7.3) to a sample container filled ~80 % with solvent (7.1). Mixture ratio ~0.2 gr. beads to 100 mL solvent.

11.3.2.2 Follow the procedure of 12.1.1 – 12.1.3.

11.3.2.3 Follow the manufacturer's instruction and select the calibration option displayed in the software interface. This will engage the analyzer to measure the bead diameters.

11.3.2.4 When the analysis is complete check the Dv50 result posted and confirm it is within 3 % of the certified bead size ± certified standard deviation. If not, recalibrate and reverify starting at 11.1.

12. Procedure

12.1 *Preferred Procedure:*

12.1.1 Ensure the instrument is set up as indicated in the manufacturer's instructions.

12.1.2 Prepare the sample in the test specimen container per Section 10.

12.1.3 ~~Insert~~Install the ~~clean tubing~~ container into the ~~test specimen container.~~instrument.

~~12.1.4 Start the pump.~~

12.1.4 ~~The instrument will fill with fuel. Allow the first 5 mL to flow through to clean the instrument. Start software analysis at this point.~~Start the analysis cycle per the manufacturer's instruction.

NOTE 5—The instrument will fill with fuel. The first 5 mL will flow through to clean the instrument. Fuel analysis will start at this point.

12.1.5 The test specimen is pumped from the specimen container through the instrument and the resultant particle counts (per ~~mL)~~millilitre) for the first 1000 frames ~~is~~are compared to the results from ~~a~~the second 1000 frames in the >1 µm category. If the count values ~~recorded~~ for these two results are within ~~either~~ the greater of 10 % or 200 particles/mL then an average of the two 1000 frame data sets is calculated and reported as the result.

12.1.6 If particle count values of ~~12.1.6~~12.1.5 are not within 10 % or 200 particles/mL, the results from a third 1000 frame data set will be automatically taken and compared to the results of the second 1000 frame data set. If the results are still not within the greater of 10 % or 200 particles/mL, repeat the ~~test.~~test one time starting at (12.1.1).

12.1.7 If the results of the second test (12.1.6) still are not within the greater of 10 % or 200 particles/mL, refer to manufacturer's instruction manual or contact manufacturer.

12.2 *Alternate Procedure:*

12.2.1 Ensure the instrument is set up as indicated in the manufacturer's instructions.

12.2.2 Prepare the sample in the test specimen container per Section 10, except when preparing the verification fluid for 11.3.1, follow the preparation instruction of the verification fluid manufacturer.

12.2.3 Insert the stopper into the opening of the sample container and attach tubing.

12.2.4 Ensure vent tube is within approximately 25 mm of the container bottom and tubing for inflow is approximately 25 mm inside the container.

12.2.5 Invert the container and position approximately 150 mm above the instrument, ensuring tubing is straight and not strained.

12.2.6 The instrument will fill and flow will start. Allow the first 500 mL to flow through to clean the instrument. Start software analysis at this point.

12.2.7 The test specimen is run from the container through the instrument and the resultant solid particle counts for the first 1000 frames is compared to the results for a second 1000 frames in the ≥1 µm category. If the count values recorded in the ≥1 µm category are within either 10 % or 200 particles, then an average of the complete results of the first 1000 frames and the second 1000 frames is calculated and reported as the result. The solid particle count and water droplet count results will be calculated based on the projected equivalent particle diameter.

12.2.8 If solid particle count values recorded at ≥1 µm are not within the specified error margin of 12.2.7, repeat the test.

NOTE 6—Volumes used in Section 12 may change according to the required total volume the particular instrument requires for the analysis.

13. Report

13.1 Report the following:

13.1.1 Reference this standard and the method used,

13.1.2 Sample identification,

13.1.3 Date of testing,

13.1.4 Instrument model and software version,

13.1.5 Solid particle cumulative counts per millilitre in the ≥4 µm, ≥6 µm, and ≥14 µm ranges. Additional ranges may be reported including 0.7 µm to <4 µm,

13.1.6 Water content in parts per million.

NOTE 7—Water droplets can stick to the inside of the container wall causing reported water droplet count to be lower than actual.

13.1.7 Solid particle size distribution (optional),

13.1.8 Droplet size distribution (optional), and

13.1.9 Any deviation from the method.

NOTE 8—With regard to 13.1.7 and 13.1.8 only, distributions can be reported based on count or volume, and by user preference of minor diameter, major

diameter, equivalent particle diameter, area, perimeter, or any other characteristic measured. Distribution by any measure other than equivalent particle diameter will be noted in the result report.

14. Precision and Bias

14.1 An interlaboratory study has yet to be completed. A temporary statement including repeatability is reported in Appendix X1 and Appendix X3 which contain descriptions and results of the testing done for this temporary precision statement. Full precision and bias statements based on interlaboratory round-robin testing will be determined within five years of the adoption of this standard. The temporary repeatability to be used in 11.3.1 for particle count verification for the preferred method is $r = 492$ in the ≥4 µm range, and for the alternate method is $r = 1088$ (10 %) in the ≥4 µm range when using verification fluid referenced in 7.5.

15. Keywords

15.1 middle distillate fuels; particle analyzer; particle counting; particle size; particulate contamination