Designation: D8049 – 17

Standard Test Method for Determining Concentration, Count, and Size Distribution of Solid Particles and Water in Light and Middle Distillate Fuels by Direct Imaging Particle 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 particle analyzer (DIPA) 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 and health 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]

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

2.2 *ISO Standard:*[3]

ISO 12103-1 Road Vehicles—Test Contaminants for Filter Evaluation—Part 1: Arizona Test Dust

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

2.3 *MIL Standard:*[4]

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:

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

[4] For referenced MIL standards, visit the Defense Logistics Agency, Document Services website at http://quicksearch.dla.mil

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

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

3.3 *Abbreviations:*

3.3.1 *DIPA*—Direct Imaging Particle Analyzer

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 The test specimen, approximately 4 L, is agitated in its container. The container is then fitted with a spigot to allow delivery to the direct imaging particle analyzer (DIPA). Fluid flows through the DIPA and is analyzed for solids and water content. Larger or smaller volume test specimen may be used as appropriate for the instrument.

4.3 The method requires reporting of particle and droplet 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 for this test method shall be determined using the definition per 3.2.6.

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[5]

6.1 *Laboratory or Field Usage:*

6.1.1 *Direct Imaging Particle Analyzer (DIPA)*—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.

6.1.2 *Test Specimen Container*—A clean fuel container for sample storage, transport, and transfer into the DIPA. 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 DIPA and air into the epoxy-lined container for venting.

6.1.3 *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 DIPA.

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

6.1.5 *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.

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

7.3 *Partistan Resolution Standard,* mono disperse polymer beads, coefficient of variation <10 %.

7.4 *Verification Standard Partistan 2806,* containing ISO Medium Test Dust, ISO 12103-A3 traceable to NIST.

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).

9. Preparation of Apparatus

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

9.2 Ensure instrument is clean and ready for use by flushing with a filtered, fast-drying solvent such as heptane. Clean sampling valve and tubing in the same manner. System cleanliness may be checked by running a sample of filtered heptane through it. If the test specimen has a ≥4 µm count in excess of 200 µm/mL, the system requires cleaning by continued flushing with filtered heptane until the count falls below 200.

10. Test Specimen Preparation

10.1 Gently shake the test specimen in its container for at least 1 min 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

[5] 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.

appropriate mechanical shaking device, (*b*) invert the container back and forth for a minimum of 60 times at approximately 1 Hz (cycle/second), or (*c*) use a roller device and roll for a minimum of 60 rotations. Other ways of gently shaking the container may be used provided a well-mixed test specimen is achieved.

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 be checked daily prior to use per manufacturer's operating manual.

11.2 *Calibration:*

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

11.2.2 The test specimen flow rate should be similar for calibration, verification, and operation. An orifice is provided on the outflow of the DIPA 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 to verify particle count. Test in accordance with Section 12. The per millilitre result obtained shall be equal to or less than *r*/1.414 of the measurement plus the uncertainty of the verification fluid from the certified ≥4 µm(c) value of the standard where *r* is the repeatability of the test (see Section 14 and Appendix X1). If the result obtained is not within this figure, ensure the sample preparation is in accordance with the manufacturer's instructions, check the verification fluids validity date, and run a further test using the filtered heptane 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 5—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 DIPA. 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.

12. Procedure

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

12.2 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.

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

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

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

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

12.7 The test specimen is run from the container through the DIPA 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.

12.8 If solid particle count values recorded at ≥1 µm are not within the specified error margin of 12.7, 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,

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 droplet 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,

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 which contains a description 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 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

APPENDIXES

(Nonmandatory Information)

X1. DETERMINATION OF TEMPORARY REPEATABILITY, *r*, FOR THIS TEST METHOD

X1.1 General

X1.1.1 The determination of temporary repeatability for this test method is described here along with the resultant data obtained from the testing which is then used to calculate the repeatability. The steps described follow the procedure as listed in Sections 8 – 12 of the test method.

X1.2 Procedure Used

X1.2.1 Obtain fuel samples in appropriate fuel containers for shipping.

X1.2.2 Prepare three 5 L fuel containers, either new or cleaned with filtered heptane. Pour approximately 4 L (80 % full) into the first clean fuel container.

X1.2.3 Calibrate the instrument and conduct verification per Section 11 (see Appendix X2).

X1.2.4 Prepare the instrument by flushing with filtered heptane until the particle count per millilitre in the ≥4 µm category is less than 200.

X1.2.5 Agitate the container for 60 s by turning over repeatedly.

X1.2.6 Place the stopper into the bung hole with the vent line and the feed line to the instrument already attached. Turn the container upside down and let the fuel feed into the instrument. Collect the drained fuel into the second cleaned/new fuel container.

X1.2.7 Allow 500 mL to pass through the instrument before engaging the software to start analysis of two consecutive 1000 frame data sets.

X1.2.8 If the per millilitre particle count in the ≥1 µm category for the two results is in agreement within either 10 % or 200 particles, then average the two sets of data to obtain the result for Specimen 1.

X1.2.9 Pour any remaining fuel from the feeding fuel container into the container collecting the fuel out the drain. The fuel in this second container will be designated Specimen 2. Use this second container to now repeat steps X1.2.5 – X1.2.8. Do this a third time to obtain results for three specimens tested in different containers.

X1.2.10 Use the averaged results for each specimen to calculate the repeatability of the test at each size category: ≥1 µm, ≥4 µm, ≥6 µm, and ≥14 µm.

X1.3 The results for this test for two different biofuels are listed in Tables X1.1-X1.4. An additional fuel sample was run spiked with ISO 12301–1 A test dust (see Table X1.5). The repeatability value of this data was used in 11.3.1 to confirm verification of particle count.

X1.3.1 The temporary repeatability of the test is 1088 in the ≥4 µm range.

TABLE X1.1 Fuel B100-25B (Particle Count)

Per/mL Data

	Specimen 1			Specimen 2			Specimen 3					
	1st data set	2nd data set	Average	1st data set	2nd data set	Average	1st data set	2nd data set	Average	Std Dev	*r*	% *r*
≥1	7568	7466	7517	7344	7283	7313.5	7995	7710	7852.5	272	533	7.06
≥4	1037	651	844	996	671	833.5	976	1037	1006.5	97	190	21.25
≥6	590	325	457.5	508	366	437	610	427	518.5	42	83	17.64
≥14	183	61	122	122	101	111.5	142	81	111.5	6	12	10.33

TABLE X1.2 Fuel B10-25A (Particle Count)

Per/mL Data	Specimen 1			Specimen 2			Specimen 3					
	1st data set	2nd data set	Average	1st data set	2nd data set	Average	1st data set	2nd data set	Average	Std Dev	*r*	%*r*
≥1	1281	1281	1281	1383	1220	1301.5	1078	1200	1139	88	173	14
≥4	223	183	203	122	184	153	142	184	163	26	52	30
≥6	122	81	101.5	82	101	91.5	121	82	101.5	6	11	12
≥14	41	0	20.5	0	20	10	0	0	0	10	20	198

TABLE X1.3 Fuel B100-25B (Water Droplets)

5 ppm Water	Specimen 1			Specimen 2			Specimen 3					
	1st data set	2nd data set	Average	1st data set	2nd data set	Average	1st data set	2nd data set	Average	Std Dev	*r*	% *r*
≥1	83	62	72.5	105	62	83.5	41	41	41	22	43	66
≥4	0	0	0	0	0	0	0	0	0	0	0	#DIV/0
≥6	0	0	0	0	0	0	0	0	0	0	0	#DIV/0
≥14	0	0	0	0	0	0	0	0	0	0	0	#DIV/0
10 ppm Water	Specimen 1			Specimen 2			Specimen 3					
	1st data set	2nd data set	Average	1st data set	2nd data set	Average	1st data set	2nd data set	Average	Std Dev	*r*	% *r*
≥1	146	167	156.5	105	124	114.5	146	124	135	21	41	30
≥4	42	83	62.5	83	42	62.5	62	42	52	6	12	20
≥6	21	42	31.5	21	21	21	21	21	21	6	12	48
≥14	0	0	0	0	0	0	0	0	0	0	0	#DIV/0
15 ppm Water	Specimen 1			Specimen 2			Specimen 3					
	1st data set	2nd data set	Average	1st data set	2nd data set	Average	1st data set	2nd data set	Average	Std Dev	*r*	% *r*
≥1	124	105	114.5	167	167	167	188	167	177.5	34	66	43
≥4	42	21	31.5	83	83	83	62	83	72.5	27	53	86
≥6	21	21	21	62	42	52	41	20	30.5	16	31	90
≥14	21	21	21	21	21	21	21	21	21	0	0	0

TABLE X1.4 Fuel B10-25A (Water Droplets)

5 ppm Water	Specimen 1			Specimen 2			Specimen 3					
	1st data set	2nd data set	Average	1st data set	2nd data set	Average	1st data set	2nd data set	Average	Std Dev	*r*	% *r*
≥1	41	41	41	41	62	51.5	62	41	51.5	6	12	25
≥4	0	0	0	0	0	0	0	0	0	0	0	#DIV/0
≥6	0	0	0	0	0	0	0	0	0	0	0	#DIV/0
≥14	0	0	0	0	0	0	0	0	0	0	0	#DIV/0
10 ppm Water	Specimen 1			Specimen 2			Specimen 3					
	1st data set	2nd data set	Average	1st data set	2nd data set	Average	1st data set	2nd data set	Average	Std Dev	*r*	% *r*
≥1	122	142	132	122	167	144.5	146	105	125.5	10	19	14
≥4	61	101	81	62	62	62	83	41	62	11	22	31
≥6	21	21	21	21	21	21	21	0	10.5	6	12	68
≥14	0	0	0	0	0	0	0	1	0.5	0	1	339
15 ppm Water	Specimen 1			Specimen 2			Specimen 3					
	1st data set	2nd data set	Average	1st data set	2nd data set	Average	1st data set	2nd data set	Average	Std Dev	*r*	% *r*
≥1	146	188	167	124	105	114.5	167	146	156.5	28	54	37
≥4	62	83	72.5	41	41	41	21	63	42	18	35	68
≥6	41	21	31	21	21	21	21	41	31	6	11	41
≥14	21	21	21	21	21	21	21	41	31	6	11	47

TABLE X1.5 Fuel B10-25A with ISO 12301–1 A Test Dust

Size range	Specimen 1			Specimen 2			Specimen 3			Std Dev	*r*	% *r*
	1st data set	2nd data set	Average	1st data set	2nd data set	Average	1st data set	2nd data set	Average			
≥1	37 947	38 542	38 244.5	39 046	41 232	40 139	37 550	40 090	38 820	971	1904	5
≥4	9675	9746	9710.5	10 562	11 020	10 791	9989	10 953	10 471	555	1088	10
≥6	4848	5030	4939	4943	5154	5048.5	5324	5666	5495	295	577	11
≥14	532	600	566	590	724	657	640	895	768	101	198	26

X2. COUNT VERIFICATION USING PARTISTAN 2806 CALIBRATION FLUID

X2.1 General

X2.1.1 The data presented here shows the verification counts per millilitre for solid particles detected in the particle count standard calibration fluid Partistan 2806,[6] which is equivalent to standards referenced in ISO 11171 for calibration of automatic particle count instruments.

X2.2 Method

X2.2.1 The Partistan 2806 calibration fluid was gravity fed through the clean DIPA instrument and collected in a clean specimen container.

X2.2.2 The software calculated the per mL particle count for two consecutive 1000 frame data sets.

Size Range	Average Particle Count Result
≥4	10 962

X2.2.3 The manufacturer's certified particle count per millilitre of the verification fluid used in this verification exercise in the ≥4 µm category was 10 911.

X2.2.4 The average particle count in the ≥4 µm range for the instrument and the certified count in the same category for the standard must agree within *r*/1.414 plus the standard deviation of the particle count.

$$10962 - 10911 < 1088/1.414,$$

therefore the count was verified.

[6] Fluid is traceable to NIST SRM 2806b.

SUMMARY OF CHANGES

Subcommittee D02.14 has identified the location of selected changes to this standard since the last issue (D8049 – 16a) that may impact the use of this standard. (Approved May 1, 2017.)

(1) Revised subsections 6.1.4, 11.3, and 11.3.2.

Subcommittee D02.14 has identified the location of selected changes to this standard since the last issue (D8049 – 16) that may impact the use of this standard. (Approved Dec. 15, 2016.)

(1) Revised subsection 12.2.