INTERNATIONAL

Designation: ~~D7261 – 14~~ D7261 – 17

Standard Test Method for Determining Water Separation Characteristics of Diesel Fuels by Portable Separometer[1]

This standard is issued under the fixed designation D7261; 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 covers a rapid portable means for field and laboratory use to rate the ability of diesel fuels (both neat and those containing additives) to release entrained or emulsified water when passed through fiberglass coalescing material.

1.2 This test method is applicable to diesel fuels such as Specification D975 Grade No. ~~1~~1-D and Grade No. ~~2~~2-D of all sulfur levels, Specification D7467~~and MIL-F-16884,~~ biodiesel blends B6-B20, and MIL-DTL-16884, naval distillate fuel (NATO F-76).

NOTE 1—This test method is similar to Test Method D3948 which is applicable to aviation turbine fuels.

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~~ safety, health, and ~~health~~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]

D975 Specification for Diesel Fuel Oils
D1193 Specification for Reagent Water
D3948 Test Method for Determining Water Separation Characteristics of Aviation Turbine Fuels by Portable Separometer
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4176 Test Method for Free Water and Particulate Contamination in Distillate Fuels (Visual Inspection Procedures)
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D4306 Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination
D4860 Test Method for Free Water and Particulate Contamination in Middle Distillate Fuels (Clear and Bright Numerical Rating)
D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants
D6426 Test Method for Determining Filterability of Middle Distillate Fuel Oils
D7224 Test Method for Determining Water Separation Characteristics of Kerosine-Type Aviation Turbine Fuels Containing Additives by Portable Separometer
D7467 Specification for Diesel Fuel Oil, Biodiesel Blend (B6 to B20)

2.2 *Military Standard:*

~~MIL-F-16884~~MIL-DTL-16884 Fuel, Naval Distillate ~~(NATO F-76)~~[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 and Cleanliness~~on Stability, Cleanliness and Compatibility of Liquid Fuels.

Current edition approved ~~Dec. 1, 2014~~Dec. 1, 2017. Published ~~January 2015~~January 2018. Originally approved in 2006. Last previous edition approved in ~~2013~~2014 as ~~D7261 – 13.~~D7261 – 14. DOI: ~~10.1520/D7261-14.~~10.1520/D7261-17.

[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 Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.~~for electronic download at ASSIST Quick Search (http://quicksearch.dla.mil).

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

3. Terminology

3.1 For definitions of terms used in this test method that are not shown below, refer to Test Methods D3948 and D7224.

3.2 *Definitions:*

3.2.1 *reference fluid, n—in MSEP*[4] and DSEP[4], *[diesel separability] water separability tests* a reference fluid base to which a prescribed quantity of a known surface active agent has been added.

[4] MSEP and DSEP are registered trademarks of EMCEE Electronics, Inc, 520 Cypress Ave., Venice, FL 34285.

3.2.1.1 *Discussion—*

The known surface active agent is typically bis-2-ethylhexyl sodium sulfosuccinate, commonly referred to as AOT, dissolved in toluene.

3.2.2 *surfactant, n—in petroleum fuels*, surface active material (or surface active agent) that could disarm (deactivate) filter separator (coalescing) elements so that free water is not removed from the fuel in actual service.

3.2.2.1 *Discussion—*

Technically, surfactants affect the interfacial tension between water and fuel which affects the tendency of water to coalesce into droplets.

3.2.3 *strong surfactant, n—in petroleum fuels*, surface active material that disarms filter separator elements, allowing water to pass.

3.2.3.1 *Discussion—*

Strong surfactants can be refinery process chemicals left in the fuel or contaminants introduced during transportation of the fuel.

3.2.4 *weak surfactant, n—in petroleum fuels*, surface active material, typically certain types of additives such as static dissipator additive, that does not adversely affect the performance of filter separator elements in actual service.

3.3 *Definitions of Terms Specific to This Standard:*

3.3.1 *DSEP rating, n*—the diesel separability rating of diesel fuel as measured by this test method.

3.3.1.1 *Discussion—*

DSEP ratings are only valid within the range of 50 to 100, with ratings at the upper end of the range indicating a clean fuel with little or no contamination by surfactants, which is expected to show good water-separating properties when passed through a filter-separator (coalescing type filter) in actual service; see 14.1.

3.3.2 *reference fluid base, n*—a distillate diesel fuel that has been cleaned in a prescribed manner to remove all surface-active contaminants (agents), and having a minimum DSEP rating of 97.

3.3.2.1 *Discussion—*

The reference fluid base should be a diesel fuel typical of fuels to be tested.

3.4 *Abbreviations:*

3.4.1 *ac*—alternating current

3.4.2 *AOT*—Aerosol OT (see 8.1)

3.4.3 *C/S*—collect sample

3.4.4 *dc*—direct current

3.4.5 *DSEP*—diesel separability

3.4.6 *MSEP*—Micro-Separometer[5]

[5] Micro-Separometer is a trademark of EMCEE Electronics, Inc, 520 Cypress Ave., Venice, FL 34285.

4. Summary of Test Method

4.1 ~~A50 mL~~A 50 mL water/fuel sample emulsion is created in a syringe using a high-speed mixer. The emulsion is then expelled from the syringe at a programmed rate through a standard fiberglass coalescer and the effluent is analyzed for uncoalesced water by a light transmission measurement.

4.2 The results are reported on a 0-to-100 scale to the nearest whole number, however the effective range of the test equipment is from 50 to 100. High ratings indicate that water is easily coalesced, implying that the fuel is relatively free of surfactants.

4.3 A test can be performed in 5 min to 10 min.

5. Significance and Use

5.1 This test method provides a measure of the presence of surfactants in diesel fuels, and can be performed in the field or in a laboratory. Like Test Method D3948 used for jet fuel, this test method can detect traces of some refinery treating chemicals left in fuel. It can also detect surface active substances added to or picked up by the fuel during handling from point of production to point of use.

5.2 Certain additives, which can act as weak surfactants, give a slightly reduced DSEP rating. Other substances which are strong surfactants give much lower DSEP ratings.

5.3 ~~While filter separators have not been common in diesel fuel systems, they could become more prevalent with ULSD containing increased additive content to ensure clean, dry fuels in new engine designs.~~This test method recommends use of the D cell coalescer when testing ULSD that contains less than 1 % biodiesel content. The DB cell coalescer should be used when testing B1-B20 blends. Weak surfactants, with slightly reduced DSEP ratings, do not significantly affect the ability of filter separators to separate free water from the fuel. Strong surfactants give a much lower DSEP rating and adversely affect the ability of filter separators to separate free water from the fuel.

5.4 Results from this test method do not have a known relationship to the rate of water settling in tanks.

5.5 The Micro-Separometer instrument has a measurement range from 50 to 100. Values obtained outside of those limits are undefined and invalid.

NOTE 2—In the event a value greater than 100 is obtained, there is a good probability that light transmittance was reduced by material contained in the fuel used to set the 100 reference level. The material was subsequently removed during the coalescing portion of the test, thus, the processed fuel had a higher light transmittance than the fuel sample used to obtain the 100 reference level resulting in the final rating measuring in excess of 100.

6. Interferences

6.1 Any suspended particles, whether solids or water droplets or haze, in a fuel sample will interfere with this test method, which utilizes light transmission of a fuel sample after emulsification with water and subsequent coalescence.

6.2 ~~Non-hydrocarbon~~ Some non-hydrocarbon components such as oxygenates, especially alcohols, or emulsified water have not been verified for this test method and will likely interfere.

7. Apparatus

7.1 A *Micro-Separometer*[6,7] *instrument* is used to perform the test. The unit is portable and self-contained, capable of operating on an internal rechargeable battery pack or being connected to an ac power source using power cords which are available for various voltages. Connection to an ac power source will provide power to the unit and affect battery recharge. The accessories can be packed in the cover of the lockable case. There are two versions of the Micro-Separometer: the Mark V Deluxe and the upgraded version, Mark X.

NOTE 3—An extensive study was performed to verify that the Mark X Micro-Separometer gives equivalent results to the Mark V Deluxe Micro-Separometer. See Research Report RR:D02-1647.[8]

7.1.1 The Emcee Model 1140 Micro-Separometer Mark V Deluxe and associated control panel are shown in Fig. 1.

NOTE 4—Of the lettered (A-G) push buttons on the Mark V Deluxe, only the D push button is applicable to this test method.

7.1.2 The Emcee Model 1140 Micro-Separometer Mark X and associated control panel are shown in Fig. 2. Table 1 lists the manual and audio operating characteristics of the instrument.

NOTE 5—Of the lettered push buttons that select the test mode, only the DIESEL push button is applicable to this test method.

[6] The sole source of supply of the apparatus (Model 1140 Micro-Separometer, Mark V Deluxe and Mark X) known to the committee at this time is EMCEE Electronics, Inc., 520 Cypress Ave., Venice, FL 34285 www.emcee-electronics.com. 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] The Model 1140 Micro-Separometers Mark III and Mark V Standard versions may also be used, but they are no longer supported by the manufacturer. For operating procedures using these instruments, the user is referred to Test Method D3948–87.

[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1647.

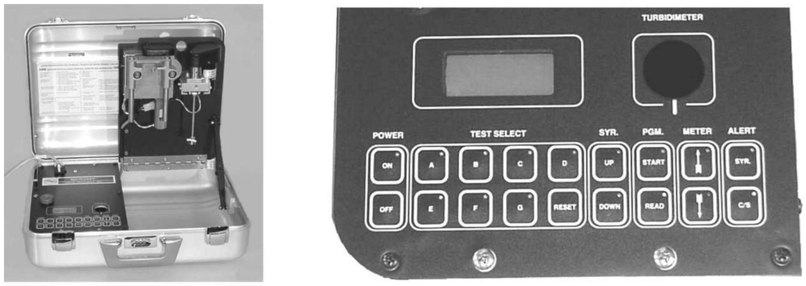

FIG. 1 Micro-Separometer Mark V Deluxe and Associated Control Panel

FIG. 2 Micro-Separometer Mark X and Control Panel

TABLE 1 Manual and Audio Operating Characteristics of the Emcee Model 1140 Micro-Separometer Instrument for Mode D/Diesel Operation

Available Test Mode(s) Function	Mark V Deluxe	Mark X
Test Mode - Select Mode D		
Depress	D push button	Diesel push button
Syringe Drive	Not required	Not required
Speed Selection	Not required	Not required
Clean Cycle		
Depress	START push button	CLEAN 1 CLEAN 2
Initiate Automatic Test Sequence		
Depress	START push button	RUN push button
Cancel Automatic Sequence		
Depress	RESET push button	RESET push button
1st Meter Read		
1st Meter Adjust	Depress ARROW push buttons	Not required
2nd Meter Read		
2nd Meter Adjust	Depress ARROW push buttons	Not required
Collect Sample	Short Tone and C/S Annunciator Lamp Illuminates	Short Tone and C/S Annunciator Lamp Illuminates
3rd Meter Read		
Record Measurement	Pulsed Tone Sounds 5 s into 3rd Meter Read	Steady tone

7.1.3 Both the Mark V Deluxe and Mark X Micro-Separometers have the *emulsifier* located on the right side of the raised panel and the *syringe drive mechanism* on the left side. The control panel containing the operating controls (push buttons) is mounted on the fixed panel in the left side of the case. A circuit breaker located on the control panel provides protection for the ac power circuit. The turbidimeter is located under the main control panel and consists of a well in which the sample vial is placed, a light source, and a photocell.

7.2 *Beaker, Catch Pan, or Plastic Container*—(Supplied with each Micro-Separometer) used to receive the waste fuel during the coalescence period of the test.

7.3 *Pipet*—An automatic ~~50-µL~~50 µL hand pipet (supplied with each Micro-Separometer) designed to accept a disposable plastic tip.

8. Reagents and Materials

8.1 *Aerosol OT,* (AOT), solid (100 % dry) bis-2-ethylhexyl sodium sulfosuccinate.

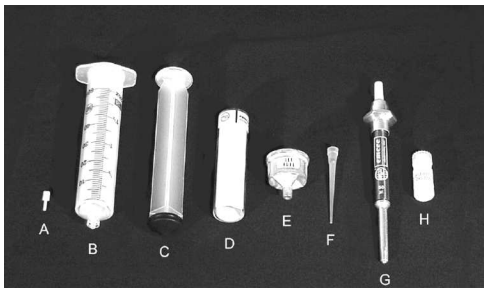

FIG. 3 Test Supplies and Small Parts

8.2 *Dispersing Agent*—Toluene solution (**Warning**—Flammable. Vapor harmful.) containing 1 mg of Aerosol OT per milliliter of toluene.

8.3 *Expendable Materials* needed to perform the test are shown in Fig. 3 and consist of the following:[9]

8.3.1 *Syringe Plug, (A)*—A plastic plug used to stopper the syringe during the clean and emulsion cycles.

8.3.2 *Syringe, (Barrel (B) and Plunger (C))*—A disposable 50 mL plastic syringe.

8.3.3 *Vials, (D),* 25 mm outside diameter vial premarked for proper alignment in the turbidimeter well.

8.3.4 *DCell*[10] *or DBCell Coalescer, (E)* an expendable, pre-calibrated aluminum coalescer cell with a tapered end to fit the syringe. It is labeled in a white background with black lettering:

DCELL®, DIESEL FUEL, D7261

DBCELL®, DIESEL FUEL, D7261

8.3.4.1 In order for a coalescer to be acceptable for this test method, it shall have ~~been manufactured using 2-grades of fiberglass and have~~ passed factory calibration tests for air flow and leakage.

8.3.5 *Disposable Plastic Pipet Tip (F)*—Used with an automatic 50 µL hand pipet (Fig. 3, G).

8.3.6 *Container (H)*—A clean container of double-distilled water (8.7).

8.4 *Reference Fluid Base*—A surfactant-free, clean, distillate diesel fuel which is used to verify proper operation and is prepared in the manner described in Annex A1 (see 3.3.2). (**Warning**—Flammable. Vapor harmful.)

8.5 *Reference Fluid*—(**Warning**—Flammable. Vapor harmful.) A fluid used for checking the operational performance of the Micro-Separometer instrument), consisting of increasing concentrations (0 mL/L to 1.6 mL/L) of dispersing agent added to the reference fluid base. The DSEP ratings for this range of concentrations appear in ~~Table 2~~Tables 2-5. The reference fluids are prepared and tested as described in Sections 12 and 13.

8.6 *Toluene,* ACS reagent grade. (**Warning**—Flammable. Vapor harmful.)

8.7 *Water,* clean, double-distilled and surfactant-free: D1193 Type IV reagent water, re-distilled. In practice, re-distillation of commercial distilled water has proven to be satisfactory.

8.7.1 Use of water other than double-distilled water (such as tap water) will render test results invalid.

9. Hazards

9.1 The primary hazard in this test method is the flammability of the fuels that are tested. Take suitable precautions to avoid sparks, flames or sources of ignition.

9.2 Minimize worker exposure to breathing fuel vapors.

10. Preparation of Apparatus

10.1 Locate the instrument on a clean workbench in an area where the temperature is between 18 °C and 29 °C and does not vary more than ±3 °C.

10.2 Open the case, and raise the right panel until completely vertical and locked in place.

10.2.1 If ac power is available, connect the power cord.

NOTE 6—The Micro-Separometer can be purchased with or without an internal battery pack.

10.2.2 If the internal battery power is used, ensure that the batteries are charged sufficiently to perform the desired number of tests.

[9] A new syringe, pipet tip, test sample vial, syringe plug, DCell coalescer or DBCell coalescer (trademarked) and double distilled water are used in each test. These expendable materials are available from Emcee Electronics, Inc. in a kit, termed the DCell or DBCell Micro-Separometer Six Pack (trademarked), containing supplies for six tests (Fig. 4).

[10] The term "DCell" "DBCell" and logo are registered trademarks of EMCEE Electronics, Inc, 520 Cypress Ave., Venice, FL 34285.

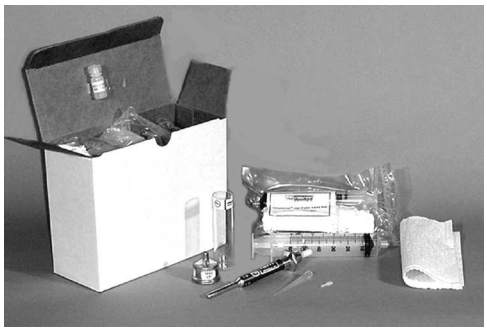

FIG. 4 Six Pack and Test Accessories

TABLE 2 Expected Performance for Reference ~~Fluids~~[A]

~~AOT mL/L~~	~~DSEP Rating~~	~~Std Dev~~	
	~~0.0~~		~~970.89~~
~~0.2~~	~~90~~	~~2.88~~	
~~0.4~~	~~85~~	~~2.58~~	
~~0.8~~	~~77~~	~~1.55~~	
~~1.6~~	~~65~~	~~1.75~~	

TABLE 2 Expected Performance for Reference Fluids—ULSD Using the D Cell

D Cell ULSD		
Concentration of AOT (mg/L)	Average DSEP Rating	Standard Deviation
0	96.4	1.93
0.3	90.3	2.21
0.6	82.4	4.08
0.9	75.2	4.83
1.2	72.6	3.29

~~[A] Expected range of values obtained by using increasing amounts of dispersing agent AOT used to verify instrument calibration. The values shown in Table 2 are the averages that were derived from an inhouse test study conducted in September 2005, by Emcee Electronics, Inc. One operator using one Micro-Separometer performed 6 successive tests on each reference fuel. The values in Table 2 are graphically shown in Fig. 10.~~

TABLE 3 Expected Performance for Reference Fluids—Marine Diesel using the D Cell

D Cell Marine Diesel		
Concentration of AOT (mg/L)	Average DSEP Rating	Standard Deviation
0	98.2	1.47
0.6	80.8	2.24
1.2	67.7	3.75

TABLE 4 Expected Performance for Reference Fluids—B5 using the DB Cell

DB Cell B5		
Concentration of AOT (mg/L)	Average DSEP Rating	Standard Deviation
0	99.6	0.73
0.3	97.5	1.68
0.6	91.2	3.19
0.9	85.3	3.77
1.2	83.5	4.79

NOTE 7—Low battery power on the Mark V Deluxe instrument is indicated when the power lamp does not illuminate. The Mark X will display an ERR-06 indicating a LO BAT condition, indicating that the battery is not sufficiently charged to run a test. To recharge the battery, connect the instrument to an ac power source for at least 16 h (full charge) prior to use. Approximately 25 tests can then be performed.

10.2.3 Turn the Mark V Deluxe and Mark X instruments on by depressing the switch (push button) marked ON.

NOTE 8—The on-power indicator light will alternately pulse on and off when the instrument is connected to an ac power source and will stay on