Designation: ~~D7261 – 13~~ D7261 – 14

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 D975 Grade No. 1 and Grade No. 2 of all sulfur levels, and MIL-F-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 and health practices and determine the applicability of regulatory limitations prior to use.*

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)
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

2.2 *Military Standard:*

MIL-F-16884 Fuel, Naval Distillate (NATO F-76)[3]

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,~~tests* a reference fluid base to which a prescribed quantity of a known surface active agent has been added.

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum ~~Products~~ Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.14 on Stability and Cleanliness 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 Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

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

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

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]

4. Summary of Test Method

4.1 A50 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~~ 5 min to 10 min.

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

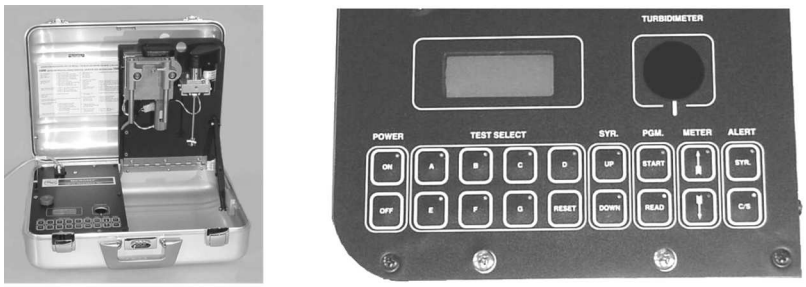

FIG. 1 Micro-Separometer Mark V Deluxe and Associated Control Panel

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. Weak surfactants, with slightly reduced DSEP ratings, do not 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 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. 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 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.

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~~25 mm outside diameter vial premarked for proper alignment in the turbidimeter well.

8.3.4 *DCell*[10] *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

[9] A new syringe, pipet tip, test sample vial, syringe plug, DCell 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 Micro-Separometer Six Pack (trademarked), containing supplies for six tests (Fig. 4).

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

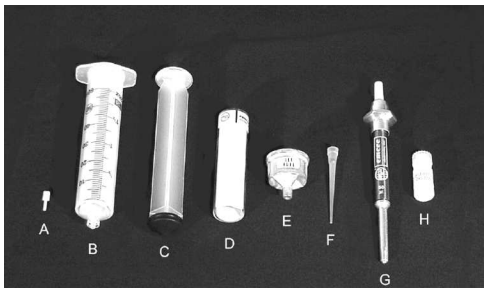

FIG. 3 Test Supplies and Small Parts

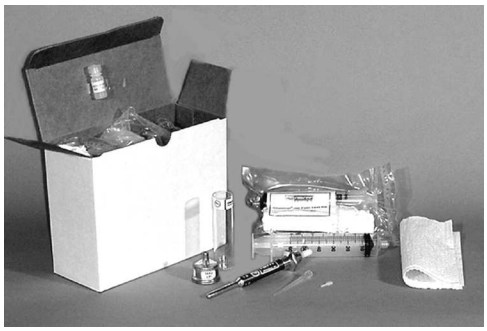

FIG. 4 Six Pack and Test Accessories

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~~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~~(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. 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 ~~1~~18 °C and ~~29°C~~29 °C and does not vary more than ~~±3°C.~~±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.

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.

TABLE 2 Expected Performance for Reference Fluids[A]

AOT mL/L	DSEP Rating	Std Dev
0.0	97	0.89
0.2	90	2.88
0.4	85	2.58
0.8	77	1.55
1.6	65	1.75

[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.

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 continuously when operated by the battery pack (dc power source). Flickering of the power indicator light, during any portion of a test sequence being performed using battery power, indicates that recharging is necessary.

10.3 Have ready a supply of syringes, syringe plugs, vials, DCell coalescers, the pipet and pipet tips, and a clean container of double-distilled water.

NOTE 9—Syringe drive travel times during the coalescing test period were initially calibrated at the factory for each mode of operation and have a significant bearing on the final test results. Syringe drive travel times exceeding the upper limit will cause the final results to measure high; conversely, travel times below the lower limit will cause the final results to measure low. Mark V Deluxe and Mark X instruments have self-check circuitry to detect out-of-tolerance syringe drive travel times. The Mark V Deluxe alert indicator lamp (marked SYR) illuminates and depending on the degree (more than 3 s) of the out-of-tolerance condition, three short (1-s) tones will also sound. The Mark X ERROR ALERT indicator illuminates and ERR-03 is displayed. An occasional out-of-tolerance alert may be experienced due to some intermittent condition, which probably will not be indicative of instrument failure. However, repeated alerts are cause for returning the instrument to the factory for adjustment.

11. Sampling and Sample Preparation

11.1 Rinse the sample container three times with the product to be sampled before collecting the sample. Collect a sample of at least ~~1 L,~~1 L, and preferably about ~~3 L.~~3 L, in a clean container in accordance with Practice D4057 or D4177.

NOTE 10—Test method results are known to be sensitive to trace contamination from sampling containers. For recommended sampling containers, refer to Practice D4306. Special precautions concerning sample containers and sampling technique are discussed in Appendix X1. Extreme care and cleanliness are required in taking samples either directly into the test syringe or into a sample container.

11.1.1 Before pouring the test sample from the container, wipe the container outlet thoroughly with a clean, lintless wiper; pour the test sample into a clean beaker or directly into the barrel of the test syringe.

11.2 (**Warning**—Do not, under any circumstances, prefilter the test fuel. The filter media can remove the very materials, surfactants, that the test method is designed to detect. If the test fuel is contaminated with particulate matter, allow such materials to settle out of the fuel before testing. Test methods such as D4176, D4860, and D6426 may be used to determine the quality and cleanliness of the sample.)

NOTE 11—If a sample does not clear up after being allowed to stand for a period of time, the sample cannot be tested by this test method (6.1).

11.3 If the sample is not within the test temperature limits of ~~18~~18 °C to ~~29°C,~~29 °C, allow the sample to stand or place the sample container in a water bath until the temperature is within the prescribed limits. The preferred temperature for testing is approximately ~~27°C.~~27 °C.

12. Calibration and Verification

12.1 The instrument is calibrated at the factory by using inhouse test equipment.

12.2 Instrument performance, especially for field use, may be verified by performing DSEP tests using a dilution of the dispersing agent (as prepared in 8.3), a reference fluid base (as prepared in Annex A1), and double-distilled water.

12.2.1 Prepare a 10:1 dilution by diluting ~~10 mL~~ 10 mL of dispersing agent (8.2) with ~~90 mL~~ 90 mL of toluene.

NOTE 12—Since ~~1 mL~~ 1 mL of dilution is equal to ~~0.1 mL~~ 0.1 mL of dispersing agent, ~~50 µL~~ 50 µL of dilution is equal to ~~0.1~~0.1 mL/~~mL/L~~ L when added to ~~50 mL~~ 50 mL of reference fluid base. The ~~0.1~~0.1 mL/~~mL/L~~ L of dispersing agent corresponds to even multiples of the concentration levels listed in Table 2.

12.2.2 Use the ~~50 µL~~ 50 µL pipet (8.3.5) to add increments of 0.1 mL/L of dispersing agent to reference fluid base.

12.2.3 Perform DSEP tests with several reference fluids and double-distilled water (8.7) according to Section 13.

12.2.4 Compare the DSEP ratings to the values listed in Table 2 for the particular concentration of dispersing agent used.

12.3 If the results do not fall within the range of limits shown in Table 2, the reference fluid shall be discarded and a fresh quantity of reference fluid prepared and the verification repeated.