Designation: 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* a reference fluid base to which a prescribed quantity of a known surface active agent has been added.

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.

[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 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**

3.2.4 *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 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. 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,

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

[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.

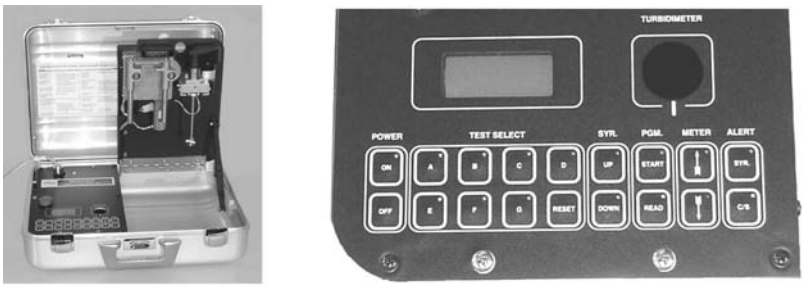


FIG. 1 Micro-Separometer Mark V Deluxe and Associated Control Panel

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.

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 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

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. 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.

[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. 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

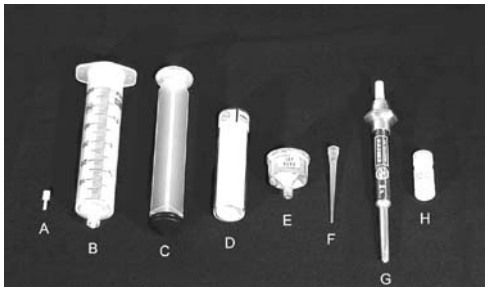


FIG. 3 Test Supplies and Small Parts

FIG. 4 Six Pack and Test Accessories

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.

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

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.

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, and preferably about 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 °C to 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.

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 of dispersing agent (8.2) with 90 mL of toluene.

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

12.2.2 Use the 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.

12.4 If repeated verification tests give out-of-tolerance test results, return the instrument to the factory for adjustment and recalibration.

13. Procedure

13.1 Select Mode D (Mark V Deluxe) or DIESEL (Mark X) operation.

13.1.1 Depress push button D or DIESEL for Mode D operation.

NOTE 13—Sequential illumination of the pushbuttons will cease and the depressed push button will stay lit. The correct syringe drive speed is set automatically.

13.2 To remove any contaminants from the syringe barrel and stirrer, run two 50 mL portions of the fuel to be tested through the mixing system in clean cycles, as follows.

13.2.1 Remove the plunger from a new 50 mL syringe and wipe the tip using a clean, lintless wipe to remove any sheen caused by excess lubricant. Insert a plug into the exit hole of the syringe barrel, add 50 mL ± 1 mL of fuel, and place the syringe barrel on the emulsifier mount, turning to lock in place.

13.2.1.1 To mitigate the buildup of static charge, only nitrile gloves are recommended for use while handling the syringe barrel.

13.2.2 Ensure that the syringe barrel is properly aligned concentrically with the mixer shaft and is not touching the propeller. Proper alignment can be verified by grasping the syringe barrel and moving the same until the propeller on the end of the mixer shaft is free and not touching.

NOTE 14—Misalignment can cause plastic shavings to form and collect on the coalescer filter material resulting in erroneous test results. This applies to all instruments manufactured prior to July 1988 that have not been serviced by EMCEE Electronics, Inc. since that date. Since July 1988, with ASTM approval, all new instruments and those returned for service have had a standoff installed on the mixer shaft to prevent the syringe barrel from coming into contact with the mixer blades.

13.2.3 Initiate the clean cycle by depressing the START push button on the Mark V or the CLEAN 1 push button on the

TABLE 3 Test Sequence (Mode D Operation)

Micro-Separometer Action	Operator Activity	Time min:s	
		Test Sequence (Time)	Elapsed Time
Start sequence	Depress start switch	0	0
Pulsed tone	Prepare for meter read	0:04	0:04
Meter on	Full-scale adjustment 1	0:10	0:14
Emulsifier on	Observe emulsification	0:30	0:44
No activity	Place emulsified sample into syringe drive	0:30	1:14
Pulsed tone	Prepare for meter reading	0:04	1:18
Meter on	Full-scale adjustment 2	0:10	1:28
Syringe drive	Coalescence period	0:45	2:13
Starts down	Collect sample		
No activity	Place sample into turbidimeter well	0:56	3:09
Steady tone	Prepare for meter reading	0:04	3:13
Meter on	Read results	0:05	3:18
One second tone	Record results	0:05	3:23

Mark X, as designated by the annunciator light. (**Warning**—Do not operate the mixer without having a syringe with fuel in place. The mixer bearings depend on the fuel for lubrication.)

13.2.4 At the end of the first clean cycle, when the mixer motor stops, remove the syringe barrel from the emulsifier, discard the fuel, and drain the syringe thoroughly.

13.2.5 Add 50 mL ± 1 mL of fresh fuel into the syringe and place the syringe barrel on the emulsifier mount (turn to lock in place). Visually inspect that the syringe barrel is properly aligned concentrically with the mixer shaft and is not touching the propeller.

13.2.6 Add about 15 mL to 20 mL of the fuel to be tested into a new vial. Wipe the outside of the vial with a clean, lintless wiper, and insert the vial into the turbidimeter well, aligning the black mark on the vial at 90° from the white line on the front of the turbidimeter well. Rotate the vial until the black mark on the vial aligns with the white line on the front of the turbidimeter well (Fig. 1). (This vial of clean fuel is required for setting the meter reading to 100 in 13.8.)

13.2.7 Initiate the second clean cycle on the Mark V by pressing the RESET and START push buttons sequentially. Initiate the second clean cycle on the Mark X by pressing the CLEAN 2 push button, as designated by the annunciator light.

13.3 At the end of the second clean cycle, when the mixer motor stops, remove the syringe barrel from the emulsifier, discard the fuel, and drain the syringe thoroughly.

13.4 Add 50 mL ± 1 mL of fresh fuel sample into the syringe.

13.4.1 Handle the syringe in such a manner as to minimize warming of the fuel sample by body heat.

13.5 Using a fresh plastic tip on the hand pipet, add 50 µL of double-distilled water (8.7) to the fuel sample as follows: Holding the pipet in hand, give a slight twist to the plastic tip to ensure a tight seal, push in the plunger, immerse the tip just below the water surface, release the plunger, and withdraw from the water slowly to avoid water drops adhering to the outside of the tip. Immerse the tip of the pipet just below the fuel surface in the center of the syringe (Fig. 5) to ensure the water drops break away cleanly and fall to the bottom, push and hold in the plunger, withdraw the pipet, and release the plunger.

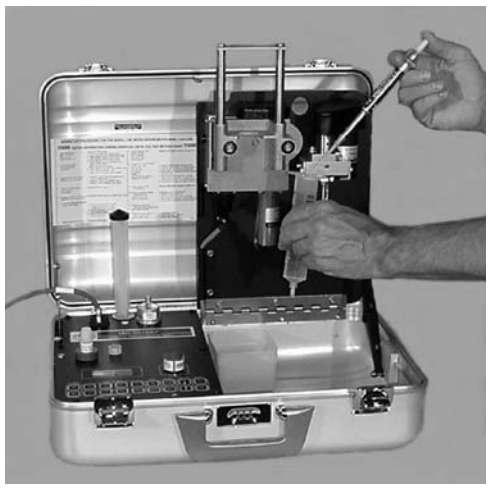


FIG. 5 Water Addition

13.6 Place the syringe barrel on the emulsifier mount, turning to lock in place.

13.7 With the syringe in place, initiate the automatic portion of Test Mode D on the Mark V by depressing the START push button (Fig. 6) or the RUN switch (Fig. 2) on the Mark X.

NOTE 15—If for any reason it is desired to interrupt the sequence and start over, the reset push button will cancel the test in progress and reset the program to the beginning of the clean segment of the test cycle.

13.8 The automatic program for the Mark V starts with a read meter indication (four short tones) followed by a 10 s full-scale adjustment period. During this period, depress the illuminated arrow push buttons on the Mark V to adjust the meter to read 100 (Fig. 7). The 100 reading is automatically set on the Mark X and does not require any adjustment. After the full-scale adjustment period, the mixer motor activates and the emulsion process is initiated.

NOTE 16—If the Mark V meter adjustment cannot be completed at this time, final adjustment may be accomplished during the second meter adjust period occurring later in the test sequence.

NOTE 17—A few drops of fuel can seep from the hole in the emulsifier head during the high-speed mixing operation. This should not affect the test results.

NOTE 18—If the emulsifier speed of the Mark X is outside of acceptable limits, the ERROR ALERT indicator on the Mark X will illuminate and ERR-05 will be displayed. The Mark V does not have an error alert.

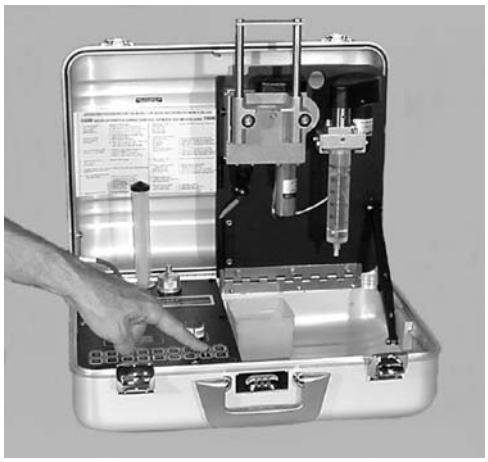


FIG. 6 Emulsification

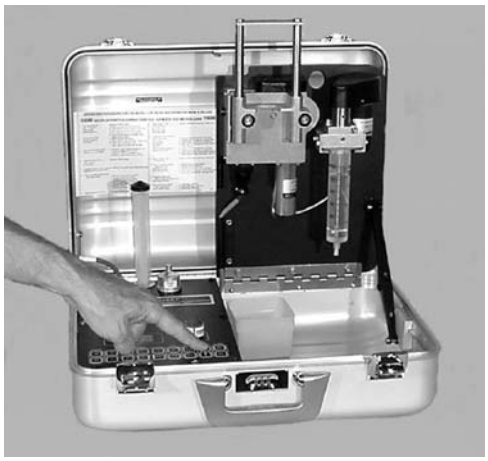


FIG. 7 Meter Adjustment

13.9 When the mixer stops (after emulsification), remove the syringe barrel from the emulsifier and partially insert the plunger to seal the open end of the syringe. Invert the syringe (exit hole up), remove the plug, and exhaust the entrapped air in the syringe barrel without significant fuel loss by carefully inserting plunger to the 50 mL mark. (Use a clean wipe over the exit hole to capture the small amounts of fuel which may be extruded as foam.) Affix a new coalescer (8.3.4) to the end of the syringe barrel. **Warning**—Use of an incorrect coalescer, such as a jet fuel coalescer, will give erroneous results.

13.9.1 Place the entire syringe assembly into the syringe drive mechanism (Fig. 8). To minimize the effect of plunger resistance (drag) in the syringe barrel, align the syringe assembly vertically in the syringe drive mechanism with the end of the syringe plunger parallel with pushbar of the syringe drive mechanism. Position a waste container beneath the coalescer to collect the unwanted portion of the processed fuel sample during the coalescing period.

13.9.2 Electrically bond the coalescer to the Micro-Separometer to prevent buildup of an electrostatic charge that could result in ignition of flammable test fluids.

13.9.2.1 Fasten the alligator clip to the coalescer and insert the plug in the chassis ground jack (Fig. 8). Other suitable grounding methods may be used for previous models.

FIG. 8 Coalescence

NOTE 19—Each Micro–Separometer instrument is furnished with a ground lead that has an alligator clip on one end and a banana plug on the other.

13.10 The second meter adjust period for the Mark V will occur after four short tones. Adjust the meter reading to 100, if necessary. The Mark X does not require any adjustment. The syringe drive mechanism will start down at the end of the meter-adjust period forcing the water/fuel emulsion through the coalescer (Fig. 8). During this operation, remove the vial from the turbidimeter well and discard the fuel.

13.11 Collect the last 15 mL of fuel sample being processed from the coalescer (Fig. 9) when the collect sample annunciator lamp (marked C/S) illuminates and a pulsed tone of short duration sounds. To lessen the amount of air introduced into the fuel during this operation, position the vial at a slight angle and allow the fuel to flow down the inner surface. Remove the vial just prior to when the last amount of sample is expelled from the coalescer.

13.12 Wipe the outside of the vial with a clean, lintless wiper to remove any fingerprints and fuel. Place the sample vial into the turbidimeter well aligning the marks on the vial and on the control panel in front of the well.

NOTE 20—At the end of the settling time (1 min), a steady (4 s duration) tone will alert the operator that the meter is about to activate. At the end of the tone, the meter will automatically activate for approximately 10 s.

13.13 Read the DSEP rating (displayed as a whole number) at the midpoint of the 10 s meter read cycle indicated by a short 1 s tone.

NOTE 21—Numerical values obtained outside the measurement range from 50 to 100 are undefined and invalid (see 5.2 and Note 2).

14. Interpretation of Results

14.1 DSEP ratings are only valid within the range of 50 to 100. Ratings at the upper end of the range indicate a clean fuel with little or no contamination by surfactants. Thus a fuel with a high DSEP rating is expected to show good water-separating properties when passed through a filter-separator (coalescing-type filter) in actual service. Conversely, fuels that give low

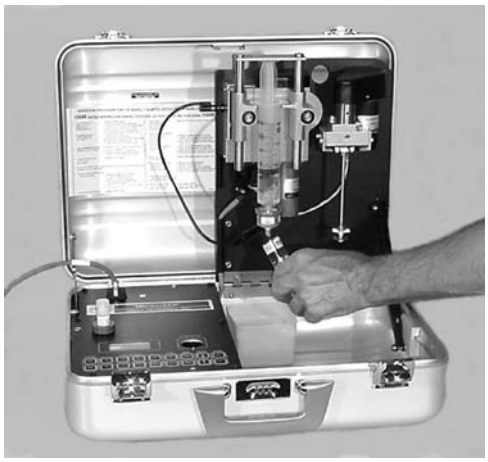


FIG. 9 Taking Sample

DSEP ratings likely contain strong surfactants that can disarm filter-separators and allow dispersed water droplets to pass through the filters.

15. Report

15.1 Report the results obtained in 13.13 as the DSEP rating by ASTM D7261.

16. Precision and Bias

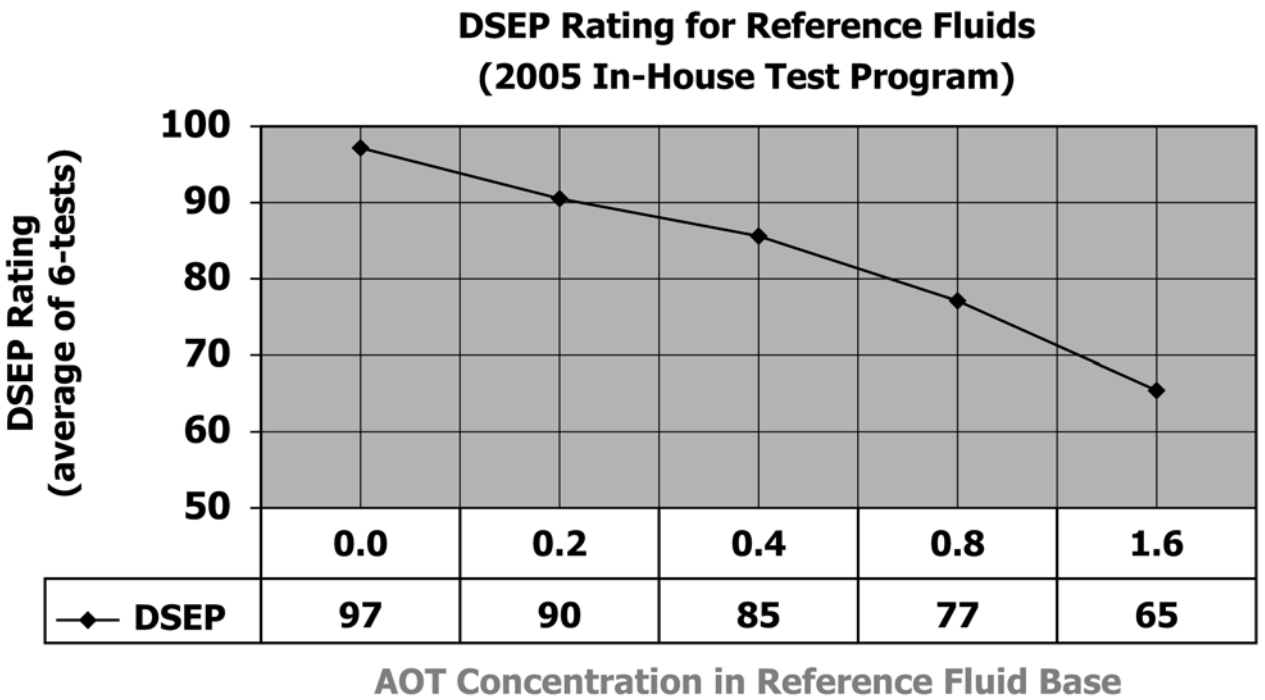
16.1 *Repeatability—Reference Fluids*—The results of 6 replicate measurements of DSEP ratings for five reference fluids are shown in Table 2 and Fig. 10 (Note 22).

NOTE 22—These results, which are based on an inhouse study performed by Emcee Electronics, Inc. in September 2005, using five reference fluids prepared in accordance with Annex A1, are given for information purposes only. A formal interlaboratory study using at least 9 reference fuels and field samples, to be tested by at least 6 operator/instrument pairs, will be performed within 5 years of approval of this test method to generate proper precision data. The data will be used to generate both repeatability and reproducibility tables and graphs.

16.2 *Bias*—The procedure in this test method has no bias, because the value of DSEP is defined only in terms of this test method.

17. Keywords

17.1 coalescence; diesel fuel; diesel fuel coalescer; DSEP rating; surfactant; water separation



This graph shows the data in Table 2 that was obtained from an inhouse program performed by Emcee Electronics, Inc. using a reference fluid (diesel fuel) prepared in accordance Annex A1 containing varying amounts of AOT dispersant. The averages of six tests that were performed successively by one operator using a single Micro-Separometer are shown. This figure will be replaced by the data obtained from the formal ILS.

FIG. 10 Reference Fluids—DSEP

ANNEX

(Mandatory Information)

A1. PREPARATION OF REFERENCE FLUID BASE

A1.1 Scope

A1.1.1 This procedure describes the preparation within an 8 h day of a 20 L lot of reference fluid base having a minimum rating of 97.

A1.2 Summary of Procedure

A1.2.1 A fuel is flowed at a constant rate through a fresh column of granular clay and collected in a clean storage receiver. The fuel should be in accordance with Specification D975. Additional processing, such as water washing followed by flowing the fuel through a salt bed prior to clay treating, may be required to attain a minimum 97 DSEP rating and the AOT standard ratings shown in Table 2.

A1.3 Apparatus

A1.3.1 *Glass Column,* containing a sealed-in coarse fritted glass disk near the bottom and with a 4 mm metering type TFE-fluorocarbon stopcock outlet at the bottom. The inside diameter of the column is 55 mm to 65 mm, and the length above the fritted disk shall be at least 1 m.

A1.3.2 *Siphon,* glass tubing having an outside diameter of 5 mm to 10 mm with the legs 100 mm to 150 mm apart. The suction leg shall be 380 mm to 400 mm long to reach the bottom of the feed container. The other leg shall be 50 mm to 100 mm longer.

A1.3.3 *Feed Container,* a standard square or round 20 L can in which the sample is obtained.

A1.3.4 *Receiver Can,* a new 20 L epoxy-lined can or one which has been used only with clay-filtered fuel. Plastic containers shall not be used.

A1.3.5 *Funnel,* with a 10 mm to 20 mm outlet.

A1.3.6 *Graduated Cylinder,* of 0.5 L to 1 L capacity.

A1.3.7 *Graduated Cylinder,* of 50 mL to 100 mL capacity.

A1.3.8 *Beaker,* 2 L capacity.

A1.4 Materials

A1.4.1 *Attapulgus Clay,* 30/60 mesh, LVM (calcined) grade or equal. Store the clay protected from atmospheric moisture and avoid handling that will cause particle size segregation.

A1.4.2 *Fine Glass Wool.*

A1.4.3 *Isopropyl Alcohol,* 90 %.

A1.4.4 *Toluene,* in a squeeze bottle. (**Warning**—Flammable. Vapor harmful.)

A1.4.5 *Water,* preferably distilled.

A1.4.6 *Salt,* rock salt or equivalent.

A1.5 Preparation of Apparatus

A1.5.1 Mount the column vertically.

A1.5.2 Measure approximately 500 mL of clay in the graduated cylinder, tapping gently to settle.

A1.5.3 Place the funnel on top, the column with its outlet centered. Quickly pour the clay into the funnel, aiming the funnel so that the clay falls in the center of the column. Remove the funnel and tap the column gently all around to settle and level the clay bed. Tamp a fist-sized wad of glass wool carefully down on top of the bed.

A1.5.3.1 When water washing of the fuel is required, place approximately 12.5 mm to 15.0 mm of salt on top of the wad of glass wool and then another wad of glass wool on top of the salt.

A1.6 Filtration Procedure

A1.6.1 Position a full 20 L feed container with its opening level with the top of the column. Remove the cap and insert the siphon, short leg in the can, longer leg in the column.

A1.6.2 Place the 2 L beaker under the column.

A1.6.3 Make sure the column stopcock is wide open. Put slight air pressure in the feed can to start the siphon.

NOTE A1.1—The glass wool packing should prevent the clay bed from being disturbed at startup.

NOTE A1.2—In a well-prepared column, the fuel may be seen to advance down the column in a nearly horizontal plane; no bubbles will rise through the clay. If the advancing front is tilted more than 45° or there is much bubbling, the quality of the percolation may be impaired.

A1.6.4 As soon as the fuel is flowing through the column outlet, adjust the metering screw to attain a rate of 50 mL/min to 60 mL/min. Check by measuring with the small graduated cylinder for 1 min or 2 min intervals.

A1.6.5 When at least 1 L has been collected, turn off the stopcock without disturbing the metering screw setting. Remove the beaker and support the 20 L receiver can under the column so that the outlet tube extends about 10 mm into the opening. Open the stopcock. Protect the opening from dirt.

NOTE A1.3—When percolating flammable fuel, seal between the outlet and receiver opening with aluminum foil, ground the receiver, and purge it with dry nitrogen before starting flow into it. A similar purge of the column before the step in A1.6.3 is desirable.

A1.6.6 Recycle the beaker of filtrate to the feed can or discard it.

A1.6.7 When the level of fuel has dropped nearly to the top of the clay bed, turn off the stopcock, remove, and cap the receiver can.

A1.6.8 For lengthy storage, purge the receiver can with dry-nitrogen.

NOTE A1.4—At the specified flow rate, the 20 L percolation will be complete in 6 h to 6.5 h running time.

A1.7 Clean the Column

A1.7.1 Drain the column.

A1.7.2 Dismount the column, open it over a solid waste can, and with the stopcock wide open, blow out the clay.

A1.7.3 With the column inverted over a liquid waste receiver, run alcohol from the squeeze bottle into the outlet. Tilt the column to rinse the entire disk area and the entire inside of the column. When the clay residue has been entirely rinsed out, disassemble and rinse the stopcock parts, dry and reassemble them, and blow the entire assembly dry.

A1.7.4 If the column still appears dirty, rinse it thoroughly with hot water, then with distilled water. Invert it and rinse as in A1.7.3 with alcohol, then with acetone and blow dry. This should seldom be necessary.

A1.8 Water Washing the Base Fuel

A1.8.1 *Scope*—Occasionally it may become necessary to further process the base fuel to prevent interaction between AOT and fuel additives not removed by the clay treatment. Base fuels containing icing inhibitors typically require this type of processing. The problem is not with the interaction with the AOT and fuel additives, it is with the ability to process a base reference fluid to a minimum DSEP rating of 97. From experience, DiEGME in jet fuel can only be adequately removed by water washing the sample that is intended to be the base reference fluid. This may not be a problem in the preparation of a reference fluid for diesel fuel. In fact, if the Microseparometer is calibrated using a jet fuel as the reference fluid, it is not necessary to perform a calibration test using a diesel fuel base as the reference fluid.

A1.8.2 *Summary of Procedure*—A given amount of water is mixed with the base fuel and is then allowed to stand for a period of time to let the fluids separate into layers. The water is then removed and the fuel is processed as described in A1.6.

A1.8.3 *Procedure:*

A1.8.3.1 Mix 1 L of water (see A1.4.5) thoroughly with 19 L of base fuel by agitating by any convenient means.

A1.8.3.2 Allow the container to stand for sufficient time to allow the water to completely settle out to the bottom of the container.

A1.8.3.3 Remove the water from the bottom of the container using a pump, pipet, or any other means available.

A1.8.3.4 Repeat A1.8.3.1 – A1.8.3.3 as required, to ensure removal of all water-soluble substances and then proceed to the filtration process.

A1.8.3.5 Initiate the filtration process of A1.6 using a filtration media prepared as described in A1.5.3.1.

APPENDIX

(Nonmandatory Information)

X1. SAMPLING TECHNIQUE

X1.1 For any test that seeks the presence of trace constituents, steps must be taken to ensure testing of a representative sample. The interlaboratory precision study for these test methods showed that flushing of the sampling container was most important. This indicates that trace amounts of surfactant material in diesel fuels can be absorbed on, or desorbed from, metal surfaces. A suggested technique for taking separometer samples follows; it has been found to give representative samples. Any similar approach should be satisfactory. The technique is shown here only as a guide to good practice.

X1.2 *Sample Container*—This should be a scrupulously clean metal can, preferably epoxy-lined. The size will be governed by the number of replicate tests to be run.

NOTE X1.1—New cans, not epoxy-lined, are sometimes coated with surfactant-type roll oils or solder flux residues, which can affect DSEP test results. Epoxy cans also have mold release or similar residues which can also affect the DSEP test result. Such cans can usually be cleaned by three consecutive rinses with the fuel to be sampled prior to taking the sample for test. Preferably the sample container should be filled with the same grade of fuel to be sampled (filtered through at least a 0.8 mm membrane filter) and allowed to stand for at least 24 h. The fuel should then be disposed of and the sample container flushed with the fuel to be tested prior to collecting the sample.

X1.2.1 *Sample Source*—Draw the sample from a moving stream of fuel whose source is removed from tank water bottoms by as great a distance as feasible.

X1.2.2 *Sample Line*—The line may consist of a short 6.4 mm to 12.7 mm diameter tube with its open end facing the moving stream. The other end (outside the pipe) should be equipped with a suitable shutoff valve and spout. In turbulent fuel streams, it has been determined that sampling taps flush with the pipe wall are satisfactory.

X1.2.3 *Taking the Sample*—Flush the sample line with at least 1 L of the fuel to be sampled. Open and close the sample valve several times. Rinse the sample can with three separate 1 L amounts of the fuel to be sampled (for a 4 L can). Include the cap and inner seal, if used, in the rinsing. Draw the sample and put the cap in place.

SUMMARY OF CHANGES

Subcommittee D02.14 has identified the location of selected changes to this standard since the last issue (D7261 – 13) that may impact the use of this standard. (Approved Dec. 1, 2014.)

(1) Subsection 13.2.1.1 was added with a recommendation to wear nitrile gloves to mitigate the build-up of electrostatic charge when handling plastic syringes.

(2) Subsection 13.2.6 was modified to clarify how the vial is to be inserted into the turbidimeter well.

Subcommittee D02.14 has identified the location of selected changes to this standard since the last issue (D7261 – 12) that may impact the use of this standard. (Approved May 1, 2013.)

(1) Terminology 3.1 was added to reference Test Methods D3948 and D7224.

(2) Added 3.4.6.