Designation: D7261 – 22

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-D and Grade No. 2-D of all sulfur levels, Specification D7467 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, health, and 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

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

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

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, Liquid Fuels, and Lubricants

D6426 Test Method for Determining Filterability of Middle Distillate Fuel Oils

D7467 Specification for Diesel Fuel Oil, Biodiesel Blend (B6 to B20)

2.2 *Military Standard:*

MIL-DTL-16884 Fuel, Naval Distillate [3]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

3.1.2 *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.

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

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

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.14 on Stability, Cleanliness and Compatibility of Liquid Fuels.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available for electronic download at ASSIST Quick Search (http://quicksearch.dla.mil).

[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

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

3.1.4 *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.

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

3.1.5 *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.

3.2 *Definitions of Terms Specific to This Standard:*

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

3.2.1.1 *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.

3.2.2 *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.

3.2.2.1 *Discussion*—The reference fluid base should be a diesel fuel typical of fuels to be tested.

3.3 *Abbreviations:*

3.3.1 *ac*—alternating current

3.3.2 *AOT*—Aerosol OT (see 8.1)

3.3.3 *C/S*—collect sample

3.3.4 *dc*—direct current

3.3.5 *DSEP*—diesel separability

3.3.6 *MSEP*—Micro-Separometer[5]

4. Summary of Test Method

4.1 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 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 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

[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.

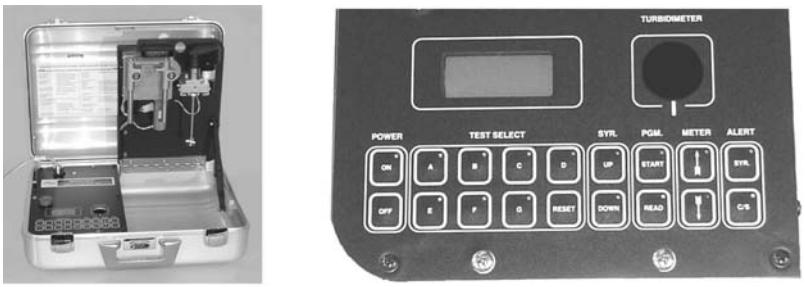

FIG. 1 Micro-Separometer Mark V Deluxe and Associated Control Panel

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.

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] *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 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 3.2.2). (**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 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] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1647. Contact ASTM Customer Service at service@astm.org.

[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. 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

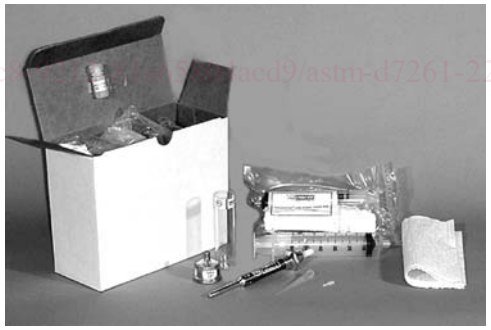

FIG. 4 Six Pack and Test Accessories

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.

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,

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

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

TABLE 5 Expected Performance for Reference Fluids—B20 using the DB Cell

DB Cell B20		
Concentration of AOT (mg/L)	Average DSEP Rating	Standard Deviation
0	99.4	0.73
0.3	95.8	1.65
0.6	88.8	3.47
0.9	81.4	4.41
1.2	72.4	5.59

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 or DBCell 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, 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 23 °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.

TABLE 6 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

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 push buttons 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 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 Refer to 5.3 for guidance on whether to use the D Cell or DB Cell. 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, and release and push again. With

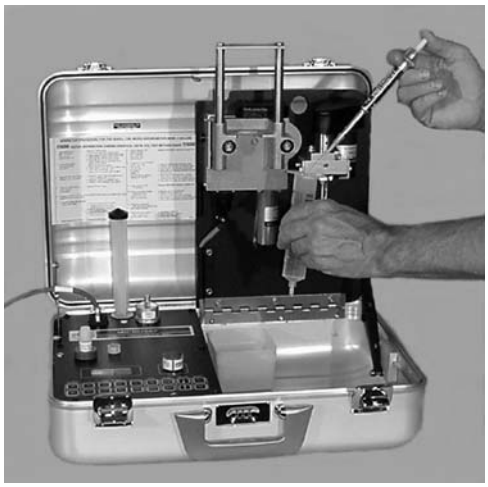

FIG. 5 Water Addition

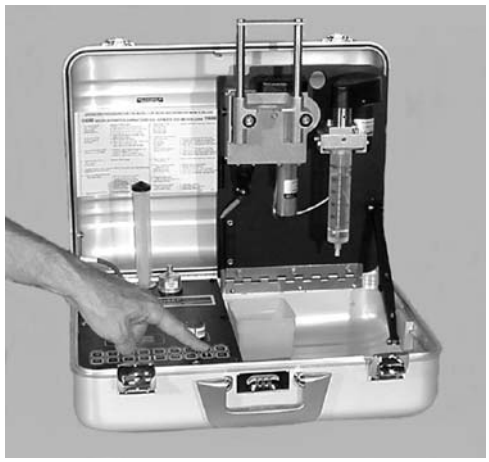

FIG. 6 Emulsification

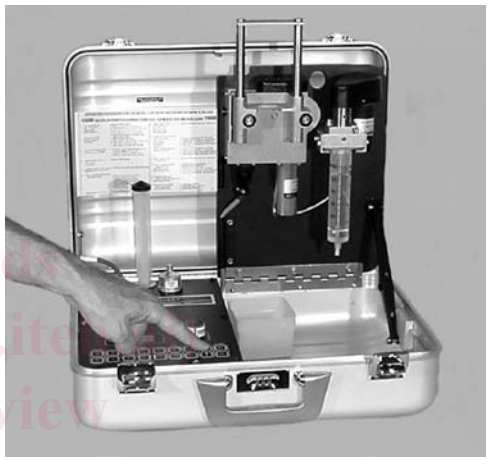

FIG. 7 Meter Adjustment

FIG. 8 Coalescence

plunger still down withdraw the pipet, and release the plunger. This ensures that all the water is in the sample.

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 (see Table 6).

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.

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