



# Standard Test Method for Determining Water Separation Characteristics of Aviation Turbine Fuels by Portable Separometer<sup>1</sup>

This standard is issued under the fixed designation D 3948; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense.*

## 1. Scope

1.1 This test method provides a rapid portable means for field and laboratory use to rate the ability of aviation turbine fuels to release entrained or emulsified water when passed through fiberglass coalescing material.

1.2 The procedure section of this test method contains two different modes of test equipment operation. The primary difference between the modes of operation is the rate of fuel flow through the fiberglass coalescing material. Test method selection is dependent on the particular fuel to be tested.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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.* For specific hazard statements, see 7.2, 7.3, 7.4, and 10.3.

## 2. Referenced Documents

### 2.1 ASTM Standards:

- D 1655 Specification for Aviation Turbine Fuels<sup>2</sup>
- D 2550 Test Method for Water Separation Characteristics of Aviation Turbine Fuels<sup>2</sup>
- D 3602 Test Method for Water Separation Characteristics of Aviation Turbine Fuels<sup>3</sup>
- D 4306 Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination<sup>3</sup>

### 2.2 Military Standards: <sup>4</sup>

- MIL-T-5624 (Grade MIL JP 4 and MIL JP 5) Turbine Fuel, Aviation Grade JP 4 and JP 5/JP 8 ST
- MIL-T-38219 (Grade MIL JP 7), Turbine Fuel, Low Volatility, JP-7
- MIL-T-83133 (Grade MIL JP 8) Turbine Fuel, Aviation, Kerosene Type, Grade JP 85 and NATO F 34 and F 35

## 3. Terminology

### 3.1 Definitions of Terms Specific to This Standard:

3.1.1 *micro separometer rating (MSEP)*—a numerical value indicating the ease of separating emulsified water from fuel by coalescence as affected by the presence of surface active materials (surfactants).

3.1.1.1 *Discussion*—MSEP ratings obtained using Test A and Test B are termed MSEP-A and MSEP-B, respectively. The MSEP rating is comparable to the Water Separometer Index, Modified (WSIM) and the Minisonic Separometer Surfactants (MSS) of Test Method D 2550 and field Test Method D 3602, respectively.

3.1.1.2 *Discussion*—The results of precision programs with the Micro-Separometer and its correlation with other rating methods (Test Methods D 2550 and D 3602) are discussed in Appendix X3.<sup>5</sup>

3.1.2 *reference fluids*—fuels that have been, as a minimum, clay treated and, as required, subjected to a water wash process and passed through a filter separator; and to which prescribed quantities of a known surface active agent (typically bis-2-ethylhexyl sodium sulfosuccinate in toluene) have been added.

## 4. Summary of Test Method

4.1 A 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 fiber-glass coalescer and the effluent is analyzed for uncoalesced water by a light transmission measurement. The results are reported on a 0-to-100 scale to the nearest whole number. High ratings

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.J0 on Aviation Fuels.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 05.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 05.02.

<sup>4</sup> Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

<sup>5</sup> A report of the data and conclusions are on file at ASTM Headquarters. Request RR:D02-1050.

indicate the water is easily coalesced, implying that the fuel is relatively free of surfactant materials. A test can be performed in 5 to 10 min.

## 5. Significance and Use

5.1 This test method provides a measure of the presence of surfactants in aviation turbine fuels. Like Test Methods D 2550 and D 3602, this test method can detect carryover traces of refinery treating residues in fuel as produced. They can also detect surface active substances added to or picked up by the fuel during handling from point of production to point of use. Certain additives can also have an adverse affect on the rating. Some of these substances affect the ability of filter separators to separate free water from the fuel.

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

5.3 Test Mode A function of the separometer will give approximately the same rating for Jet A, Jet A-1, MIL JP 5, MIL JP 7, and MIL JP 8 fuels as Test Methods D 2550 and D 3602. Using Mode A water separation characteristic ratings of Jet B and MIL JP 4 fuels will not necessarily be equivalent to Test Method D 2550 but will give approximately the same rating as Test Method D 3602. All Micro-Separometers have test Mode A capability.

5.4 The Test Mode B option is used to determine water separation ratings for MIL JP 4 fuels containing fuel system corrosion and icing inhibitors. These ratings are approximately the same as those obtained using Test Method D 2550.

5.5 Selection of Mode A or Mode B depends on the specific fuel and specification requirement. Table 1 identifies the recommended test method for various fuels.

5.6 The basic difference between Modes A and B is the flow rate at which the water/fuel emulsion is forced through the standard fiberglass coalescer cell. The lapsed time required to force the emulsion through the coalescer cell in Mode A is  $45 \pm 2$  s; whereas, Mode B requires  $25 \pm 1$  s.

**TABLE 1 Applicable Test Mode for Various Fuels**

Fuel	Available Test Mode(s)
	Applicable Test Mode
Jet A	A
Jet A-1	A
Jet B	A
MIL JP 5	A
MIL JP 7	A
MIL JP 8	A
MIL JP 4	B

## 6. Apparatus

6.1 A Micro-Separometer<sup>6,7</sup> is used to perform the test. The unit is completely portable and self-contained, capable of operating on an internal rechargeable battery pack or being connected to an a-c power source using power cords which are available for various voltages. Connection to an a-c power source will provide power to the unit and effect battery recharge. The accessories as well as the expendable materials for six tests can be packed in the cover of the lockable case.

6.2 The Micro-Separometer Mark V Deluxe and associated control panel is shown in Fig. 1. The emulsifier is on the right side of the raised panel and the syringe drive mechanism is on the left side. The control panel containing the operating controls is mounted on the fixed panel in the left side of the case. Table 2 lists the manual and audio operating characteristics of the instrument.

6.2.1 All of the controls are located in a pushbutton array on the control panel. The pushbuttons illuminate when depressed thus indicating operational status. A circuit breaker located on the control panel provides protection for the a-c power circuit.

6.2.2 By depressing the ON pushbutton, the electronic circuits are energized. The ON pushbutton pulses on and off when the instrument is being operated by an a-c source and constantly remains on when the battery (d-c) pack is used. The lettered pushbuttons will sequentially illuminate on and off indicating READY operational status.

NOTE 1—Of the lettered (A-G) pushbuttons, only the A and B pushbuttons are applicable to this test method.

6.2.3 The RESET pushbutton can be depressed at any time to cancel the test in progress and restore the program to the initial start mode. The lettered pushbuttons commence to sequentially illuminate, thus indicating a READY operational status enabling test mode selection.

6.2.4 Selection of test Mode A or test Mode B programs is accomplished by depressing either the A or B lettered pushbutton. The depressed pushbutton illuminates and the sequential illumination of the other lettered pushbuttons ceases. The START pushbutton also illuminates.

6.2.5 The START pushbutton, when depressed initially, initiates the CLEAN cycle causing the syringe drive mechanism to travel to the UP position and the emulsifier motor to operate for the cleaning operation.

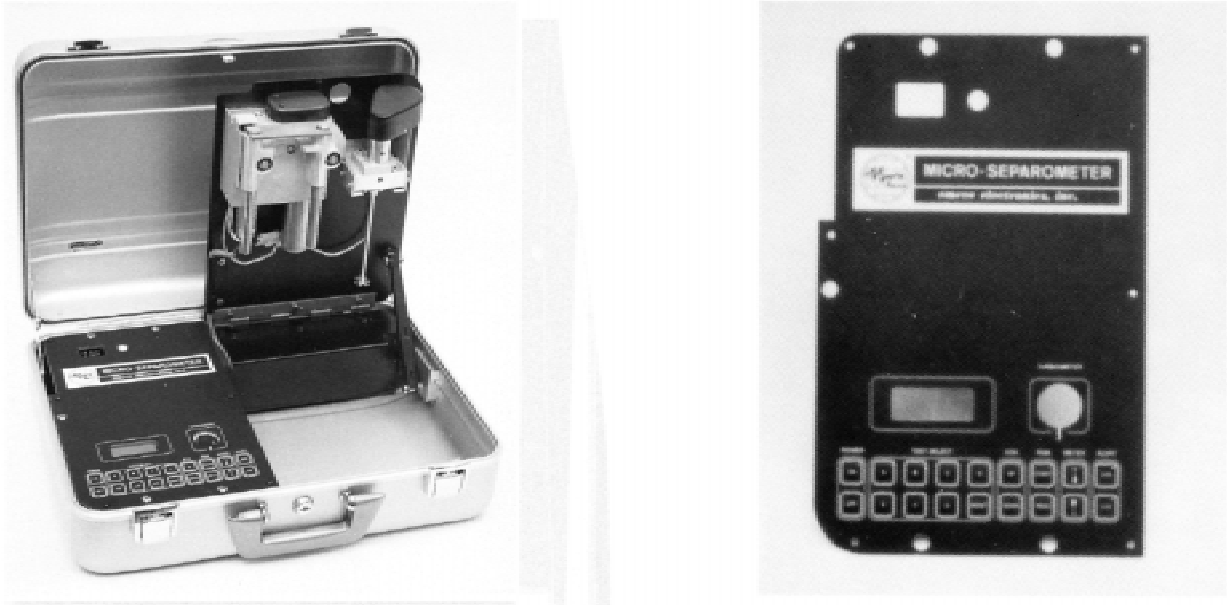
6.2.6 The START pushbutton, when depressed after the CLEAN cycle initiates the automatic program sequence causing the read indicator and the two ARROWED pushbuttons to illuminate, indicating that a full-scale adjustment period is in effect. A numerical value also appears on the meter.

6.2.7 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.

6.2.8 By depressing the appropriate ARROWED pushbutton, the displayed value on the meter can be increased or

<sup>6</sup> The Model 1140 Micro-Separometer Mark V Deluxe is available from EMCEE Electronics, Inc., 520 Cypress Ave., Venice, FL 34292.

<sup>7</sup> 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 D 3948 – 87.



(Micro-Separometer, Mark V Deluxe)

FIG. 1 Micro-Separometer Model and Associated Control Panel

TABLE 2 Manual and Audio Operating Characteristics of the Various Model 1140 Micro-Separometer Instruments

Available Test Mode(s)	Deluxe A and B
Function	
Test Mode Select	Pushbutton
Mode A	Depress A
Mode B	Depress B
Syringe Drive	Not required
Speed Selection	
Clean Cycle	START
Depress	
Pushbutton	
Automatic Sequence	
Initiate	START
Cancel	RESET
1st Meter Read	
1st Meter	Depress
Adjust	ARROWED Pushbuttons
2nd Meter Read	
2nd Meter	Depress
Adjust	ARROWED Pushbuttons
Collect Sample	Short Tone and C/S Annunciator Lamp Illuminates
3rd Meter Read	
Record	Pulsed Tone Sounds 5 s
Measurement	into 3rd Meter Read

decreased, as required, to attain the 100 reference level for the vial of fuel sample in the turbidimeter.

6.3 Accessory equipment and expendable materials needed to perform the test are shown in Fig. 2 and consist of the following:

6.3.1 *Syringe Plug, (A)*—A plastic plug used to stopper the syringe during the CLEAN and EMULSION cycles.

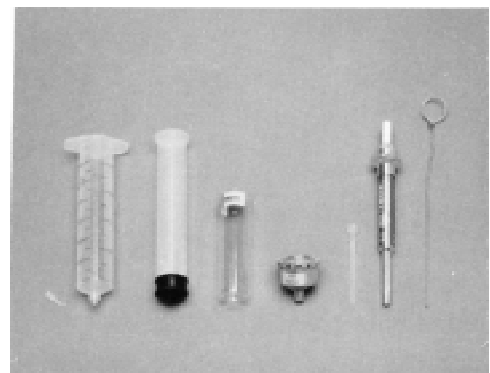
6.3.2 *Syringe, (Barrel (B) and Plunger (C))*—A disposable plastic syringe.

6.3.2.1 Use of syringes other than those demonstrated to be free of surfactant contamination in a precision program such as described in Section 12 will render test results invalid.

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

6.3.4 *Alumicel,<sup>8</sup> Coalescer, (E)* labeled for use with jet fuel, an expendable, precalibrated aluminum coalescer cell with a tapered end to fit the syringe.

<sup>8</sup> A registered trademark of EMCEE Electronics, Inc.



(A) (B) (C) (D) (E) (F) (G) (H)  
FIG. 2 Test Supplies and Small Parts

6.3.5 *Pipet, (G) with Plastic Tip (F)*—An automatic hand pipet with a disposable plastic tip. A pipet is supplied with each Micro-Separometer.

6.3.6 *Wire Aid, (H)*—A piece of wire with a loop on one end, used during test to release the air trapped in the barrel of the syringe when the plunger is being inserted. A wire aid is supplied with each Micro-Separometer.

6.3.7 *Water Container*—A clean container for distilled water (not shown or supplied).

6.3.8 *Beaker, Catch Pan, or Plastic Container*—Supplied with each Micro-Separometer may be used to receive the waste fuel during the coalescence period of the test (not shown).

6.4 A new syringe, pipet tip, test sample vials, syringe plug and Alumicel coalescer are used in each test. These expendable materials are available in a kit containing supplies for six tests. This kit termed Micro-Separometer Six Pack is designed to fit inside the top lid of the Micro-Separometer (Fig. 3).<sup>9</sup>

## 7. Reagents

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

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

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

7.4 *Reference Fluid Base*—A surfactant-free clean hydrocarbon material which is used to verify proper operation and is prepared in the manner described in Appendix X1. (**Warning**—Flammable. Vapor harmful.)

7.5 *Reference Fluids*—(**Warning**—Flammable. Vapor harmful.) (for checking the operational performance of the Micro-Separometer instrumentation) consist of increasing concentrations (0 to 1.2 mL/L) of dispersing agent added to the reference fluid base. The MSEP-A ratings for this range of concentration appear in Table 3 for Jet A, Jet A-1, MIL JP 5, MIL JP 7, and MIL JP 8 fuels and Table 4 for Jet B fuels using Mode A. The MSEP-B ratings for MIL JP 4 fuels using Mode B are shown in Table 5. The reference fluids are tested as described in Section 10 using the applicable Mode of opera-

<sup>9</sup> A kit containing six each of these test expendables is available from EMCEE Electronics, Inc., 520 Cypress Ave., Venice, FL 34292.

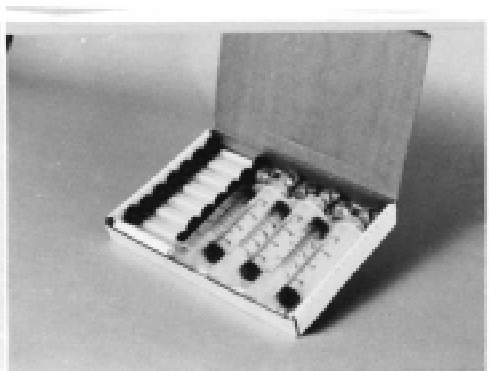


FIG. 3 Package (Six-Pack) of Supplies

TABLE 3 Expected Performance with Jet A, Jet A-1, MIL JP 5, MIL JP 7, or MIL JP 8 Reference Fluid Containing a Dispersing Agent Using Mode A Operation

Concentration of Dispersing Agent, mL/L	Standard Rating	Limits for Acceptable Performance <sup>A</sup>	
		Min	Max
0	99	97	100
0.2	89	82	94
0.4	80	69	88
0.6	72	59	83
0.8	65	51	77

<sup>A</sup>Expected range of values obtained by using increasing amounts of dispersing agent used to verify instrument calibration.

TABLE 4 Expected Performance with Jet B Reference Fluid Containing a Dispersing Agent Using Mode A

Concentration of Dispersing Agent, mL/L	Standard Rating	Limits for Acceptable Performance <sup>A</sup>	
		Min	Max
0	99	96	100
0.3	92	86	97
0.6	88	81	95
0.9	86	78	94
1.2	79	69	90

TABLE 5 Expected Performance with MIL JP 4 Reference Fluid Containing a Dispersing Agent Using Mode B

NOTE 1—Standard ratings are based on actual averages and the limits are based on  $\pm 1/2$  calculated reproducibility value from the average. A standard rating of 99 was used for the base reference fuel instead of the actual average since this value is preferred.

Concentration of Dispersing Agent, mL/L	Standard Rating	Limits for Acceptable Performance <sup>A</sup>	
		Min	Max
0	99	93	100
0.2	88	83	93
0.4	81	76	86
0.6	74	69	79
0.8	69	64	74
1.0	64	59	69
1.2	60	55	65

<sup>A</sup>Expected range of values obtained using increasing amounts of dispersing agent used to verify instrument calibration. Developed using the data from eight of the ten laboratories of the 1983 test program. Refer to RR:D02-1274.

tion. If the results do not fall within the range of limits shown in Table 3, Table 4, or Table 5, the reference fluid shall be discarded and a fresh quantity of reference fluid prepared and the check repeated. Repeated out of tolerance test results are cause for returning the instrument to the factory for adjustment and calibration.<sup>10</sup>

NOTE 2—The reference fluid base should have standard MSEP rating of 99+ without any dispersing agent; otherwise, the results may not be indicative of the accuracy of the instrument. Values obtained outside the measurement range from 50 to 100 are undefined and invalid.

7.5.1 Reference fluid shall be prepared by adding dispersing agent as described in 7.3 to a suitable quantity of reference fuel base contained in a properly equilibrated container or graduate.

<sup>10</sup> A Micro-Separometer Operation Manual is furnished with each instrument.

7.5.1.1 If a new or nonequilibrated container is used, the additive may adsorb on the walls and the MSEP ratings may improve significantly. To equilibrate the container surface, an additive blend should be held for a minimum of 24 h, discarded, and replaced with a fresh blend.

7.5.1.2 For field use, instrument calibration can be verified by performing MSEP tests using a dilution of the dispersing agent (as prepared in 7.3), a reference fluid base (as prepared in Appendix X1), and distilled water. A 10:1 dilution is prepared by diluting 10 mL of dispersing agent with 90 mL of toluene (7.2). Since 1 mL of dilution is equal to 0.1 mL of dispersing agent, 50  $\mu$ L 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 3, Table 4, and Table 5. This facilitates using the 50  $\mu$ L pipet (6.5.5) to add increments of 0.1 mL/L of dispersing agent, as well as the distilled water required for the MSEP test. Depending on the type of reference fluid base, the MSEP ratings are compared to the values listed in the applicable table for the particular concentration of dispersing agent used.

7.6 *Water*, clean, distilled, and surfactant free.

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

## 8. Preparation of Apparatus

8.1 Locate the instrument on a clean workbench in an area where the temperature is between 18 and 29°C (65 and 85°F) and does not vary more than  $\pm 3^\circ\text{C}$  (5°F).

8.2 Open the case and remove the six-pack box from the lid. Raise the right panel until completely vertical and locked in place. If a-c power is available, connect the power cord and turn the instrument on. If the internal battery power is used, assure that the batteries are charged sufficiently to perform the desired number of tests. Low battery power is indicated when the power lamp does not illuminate. Connect the instrument to an a-c power source for at least 16 h (full charge) prior to use. Approximately 25 tests can then be performed.

8.2.1 Mark V Deluxe instruments are turned on by depressing the switch (pushbutton) marked ON. The ON power indicator light will alternately pulse on and off when the instrument is connected to an a-c power source and will stay on continuously when operated by the battery pack. Flickering of the power indicator light, during any portion of a test sequence being performed using battery power, indicates that recharging is necessary.

8.3 Have ready a supply of syringes, vials, Alumicel coalescers, syringe plugs, and pipet tips, as well as a clean container with distilled water. All of the items except the container and water are furnished in the six-pack provided with the instrument. In addition, have the wire aid and pipet readily available.

8.4 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.

NOTE 3—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.

8.4.1 Mark V Deluxe instruments have self-check circuitry to detect out of tolerance syringe drive travel times. The 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. 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.

## 9. Sample Preparation

9.1 Under no circumstances shall test fuel be prefiltered as 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.

9.2 Special precautions concerning sample containers and sampling technique are discussed in Appendix X2. Extreme care and cleanliness are required in taking samples either directly into the test syringe or into a sample container. 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.

NOTE 4—Test method results are known to be sensitive to trace contamination from sampling containers. For recommended sampling containers refer to Practice D 4306.

9.3 If the sample for test is not within the test temperature limits, 18° to 29°C (65° to 85°F), allow the sample to stand until an in tolerance temperature is attained.

## 10. Procedure

10.1 Select either Mode A or B operation. (Refer to Table 1 for applicable Mode for a specific fuel.)

NOTE 5—Only JP 4 requires Mode B operation.

10.1.1 Depress either pushbutton A or B for Mode A or B operation, respectively. Sequential illumination of the pushbuttons will cease and the depressed pushbutton will stay lit. The correct syringe drive speed is set automatically.

10.2 Remove a 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  $50 \pm 1$  mL of fuel, and place the syringe barrel on the emulsifier mount, turning to lock in place. Ensure that the syringe barrel is properly aligned concentrically with the mixer shaft and is not touching the propeller.

10.3 Initiate the CLEAN cycle by depressing the START pushbutton 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.)

10.4 At the end of the first clean cycle, when the mixer motor stops, press the RESET push-button, remove the syringe barrel from the emulsifier, discard the fuel, and drain the syringe thoroughly. Add  $50 \pm 1$  mL of fresh fuel into the syringe and place (see Note 4) 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. Select Test Mode A by pressing A push-button.

10.5 Initiate the second CLEAN cycle by pressing the START push-button, as designated by the annunciator light.

10.6 Add about 15 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 with the line on the front panel.

10.7 At the end of the clean cycle, when the mixer motor stops, remove the syringe barrel from the emulsifier, discard the fuel, and drain the syringe thoroughly. Add  $50 \pm 1$  mL of fresh fuel sample into the syringe.

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

10.8 Using a fresh plastic tip on the hand pipet, add 50  $\mu$ L of distilled water 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. 4) 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.

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

10.9.1 Assure that the syringe barrel is properly aligned concentrically with the mixer shaft. 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. Misalignment may cause plastic shavings to form and collect on the coalescer filter material resulting in erroneous test results. This applies to all instruments manufactured and those which have not been serviced by EMCEE Electronics, Inc. since July of 1988. Since that date, 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.

10.10 With the syringe in place, depress the START push-button (Fig. 5) to initiate the automatic portion of the applicable test mode program listed in Table 6. If for any reason it is desired to interrupt the sequence and start over, the RESET

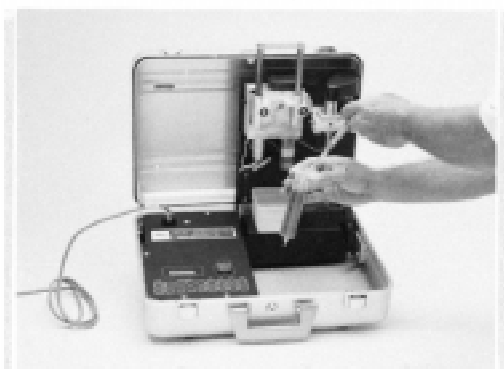


FIG. 4 Water Addition

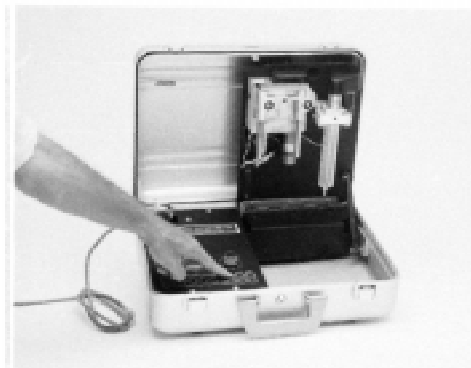


FIG. 5 Emulsification

pushbutton will cancel the test in progress and reset the program to the beginning of the CLEAN segment of the test cycle.

10.11 The automatic program starts with a read meter indication (four short tones) followed by a 10-s full-scale adjustment period. During this period, the ARROWED push-buttons will illuminate and can be depressed to adjust the meter to read 100 (Fig. 6). If the adjustment cannot be completed at this time, final adjustment can be accomplished during the second meter adjust period occurring later in the test sequence.

10.12 After the full-scale adjustment period the mixer motor activates and the emulsion process is initiated.

NOTE 6—A few drops of fuel may seep from the hole in the emulsifier head during the high-speed mixing operation. This should not affect the test results.

10.13 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 (Fig. 8). 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 (Fig. 9). Affix a new Alumicel coalescer to the end of the syringe barrel.

10.13.1 Place the entire syringe assembly into the syringe drive mechanism (Fig. 7). 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 Alumicel coalescer to collect the unwanted portion of the processed fuel sample during the coalescing period.

10.13.2 Electrically bond Alumicel coalescers to the Micro-Separometer to prevent buildup of an electrostatic charge that could result in ignition of flammable test fluids. Each Mark V instrument is furnished with a ground lead that has an alligator clip on one end and a banana plug on the other. Fasten the alligator clip to the Alumicel coalescer and insert the plug in the chassis ground jack (Fig. 7). Other suitable grounding methods may be used for previous models.

10.14 Four short tones will indicate the second meter adjust period. If required, the operator should adjust the meter reading to 100. The syringe drive mechanism will start down at the end

TABLE 6 Test Sequence

Micro-Separator Action	Operator Activity	Time			
		Sequence (s)		Elapsed (min:s)	
		Mode	Mode	Mode	Mode
Start sequence	depress start switch	A 0	B 0	A 0	B 0
Pulsed tone	prepare for meter read	4	4		
Meter on	full-scale adjustment 1	10	10	0:14	0:14
Emulsifier on	observe emulsification	30	30	0:44	0:44
No activity	placed emulsified sample into syringe drive	30	30	1:14	1:14
Pulsed tone	prepare for meter reading	4	4	1:18	1:18
Meter on	full-scale adjustment 2	10	10	1:28	1:28
Syringe drive	coalescence period	45	25	2:13	1:53
Starts down	collect sample				
No activity	place sample into turbidimeter well	56	56	3:09	2:49
Steady tone	prepare for meter reading	4	4	3:13	2:53
Meter on	read results	5	5	3:18	2:58
One second tone	record results	5	5	3:23	3:03

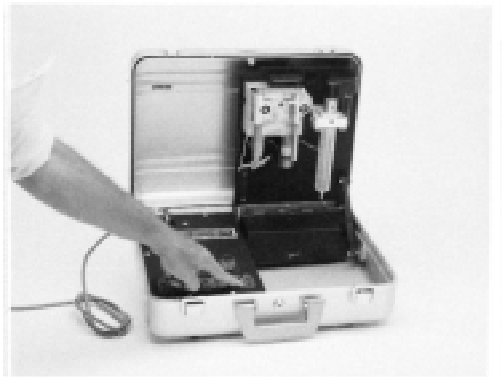


FIG. 6 Meter Adjust

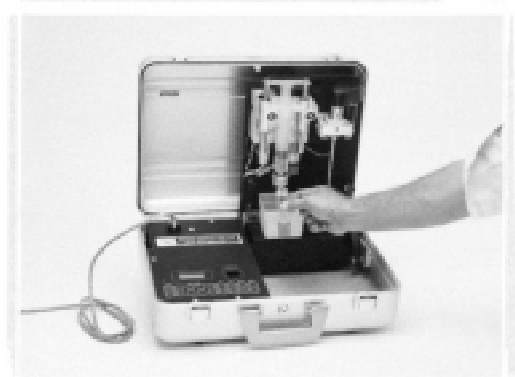


FIG. 8 Taking Sample

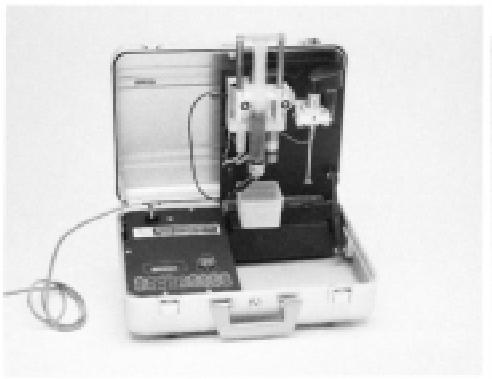


FIG. 7 Coalescence

of the meter adjust period forcing the water/fuel emulsion through the Alumicel coalescer (Fig. 7). During this operation remove the vial from the turbidimeter well and discard the fuel.

10.15 Collect the last 15 mL of fuel sample being processed from the Alumicel coalescer (Fig. 8) 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 Alumicel coalescer.

10.16 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. 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.

10.17 At the end of the tone, the METER will automatically activate for approximately 10 s.

10.17.1 MSEP ratings are read at the midpoint of the 10-s meter read cycle indicated by a short 1-s tone.

NOTE 7—Numerical values obtained outside the measurement range from 50 to 100 are undefined and invalid (see 5.2).

## 11. Report

11.1 Report the results obtained in 10.17.1 as the MSEP-A rating for Mode A operation or MSEP-B rating for Mode B operation.

12. Precision and Bias

12.1 MSEP-A ratings obtained using Mode A operation have precision as determined by the ten operator/instrument pairs on identical fuel samples at a common site, as follows:

12.1.1 *Repeatability*—The difference between successive measured MSEP-A ratings obtained by the same operator with the same Micro-Separometer under constant operating conditions on identical test material would, in the long run and in the normal and correct operation of the test method, exceed the following values for only one case in twenty (Note 8).

NOTE 8—These results are based on a 1983 ten laboratory cooperative test program using reference fuels and field samples. The repeatability and reproducibility values were estimated from results obtained at the same location and on several consecutive days, by different operator/instrument pairs testing identical samples. Results, particularly for reproducibility, obtained at different times and locations may, therefore, not be comparable according to these estimates, since they may contain errors due to sampling and environmental factors.<sup>11</sup>

In practice, two results obtained at different laboratories (locations) would be acceptable if their difference did not exceed the published reproducibility. In the event that the difference did exceed the reproducibility there would be no means of testing whether the results were acceptable or not.

The need for additional cooperative testing to establish reproducibility when samples are shipped between laboratories (and therefore may not be identical at the time of testing) is being studied.

12.1.1.1 *Reference Fuels*—The repeatability of MSEP-A ratings for Jet A, Jet A-1, MIL JP 5, MIL JP 7, and MIL JP 8 reference fuels is shown in Fig. 9 (Note 9). These data were obtained using Mode A of the Mark V instrument.

NOTE 9—This result is based on a 1978, eight laboratory cooperative test program using reference fluids prepared in accordance with Appendix X1.

12.1.1.2 *Field Samples*—The repeatability of MSEP-A ratings for Jet A, Jet A-1, MIL JP 5, MIL JP 7, and MIL JP 8 field samples, obtained by using test Mode A of the Mark V instruments, is shown in Fig. 10 (Note 10).

NOTE 10—These results are based on a 1983, ten laboratory cooperative test program using Type A (field samples of MIL JP 5) fuels containing fuel system corrosion and icing inhibitors. Due to the large number of additive combinations and concentrations, there may be field samples other than those used to develop the precision of this test method, that may not respond within the expected limits.<sup>12</sup>

12.1.2 *Reproducibility*—The difference between two single and independent measurements of MSEP-A rating obtained by different operator/instrument pairs at the same location on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values for only one case in twenty (Note 8).

12.1.2.1 *Reference Fuels*—The reproducibility of MSEP-A ratings for Jet A, Jet A-1, MIL JP 5, MIL JP 7, and MIL JP 8 reference fuels is shown in Fig. 9 (Note 9).

12.1.2.2 *Field Samples*—The reproducibility of MSEP-A ratings for Jet A, Jet A-1, MIL JP 5, MIL JP 7, and MIL JP 8 field samples using test Mode A of the Mark V instruments is shown in Fig. 9 (Note 10).

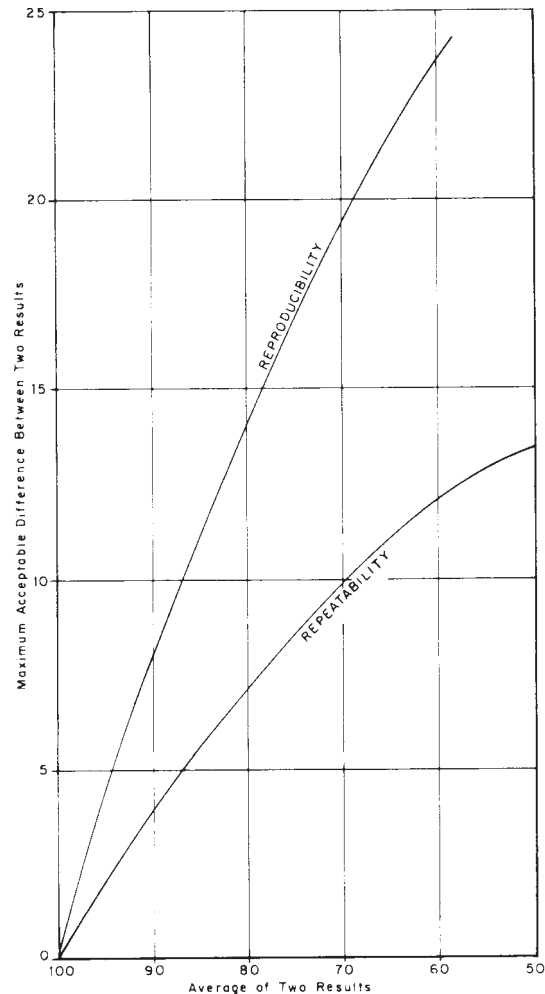


FIG. 9 Reference Fuels—MSEP-A (Mode A Operation) Variation of Repeatability and Reproducibility of MSEP-A Ratings Obtained for Reference Fuels (Jet A, Jet A-1, MIL JP 5, MIL JP 7, and MIL JP 8) Containing a Dispersing Agent

12.2 MSEP-B ratings obtained using Mode B operation have precision as determined by the ten operator/instrument pairs on identical fuel samples at a common site, as follows:

12.2.1 *Repeatability*—The difference between successive measured MSEP-B ratings obtained by the same operator with the same Micro-Separometer under constant operating conditions on identical MIL JP 4 test material would, in the long run and in the normal and correct operation of the test method, exceed the following values for only one case in twenty (Note 8).

12.2.1.1 *Reference Fuels*—The repeatability of MSEP-B ratings for MIL JP 4 reference fuels is nine (maximum difference between two consecutive measurements obtained by the same operator using the same instrument) (Note 11).

12.2.1.2 *Field Samples*—The repeatability of MSEP-B ratings for MIL JP 4 field samples is 16 (maximum difference between two consecutive measurements obtained by the same operator using the same instrument) (Note 12).

12.2.2 *Reproducibility*—The difference between two single and independent measurements of MSEP-B ratings obtained by different operator/instrument pairs at the same location on

<sup>11</sup> A report of the data and conclusions for the 1983 ten laboratory test program are on file at ASTM Headquarters. Request RR:D02-1274.



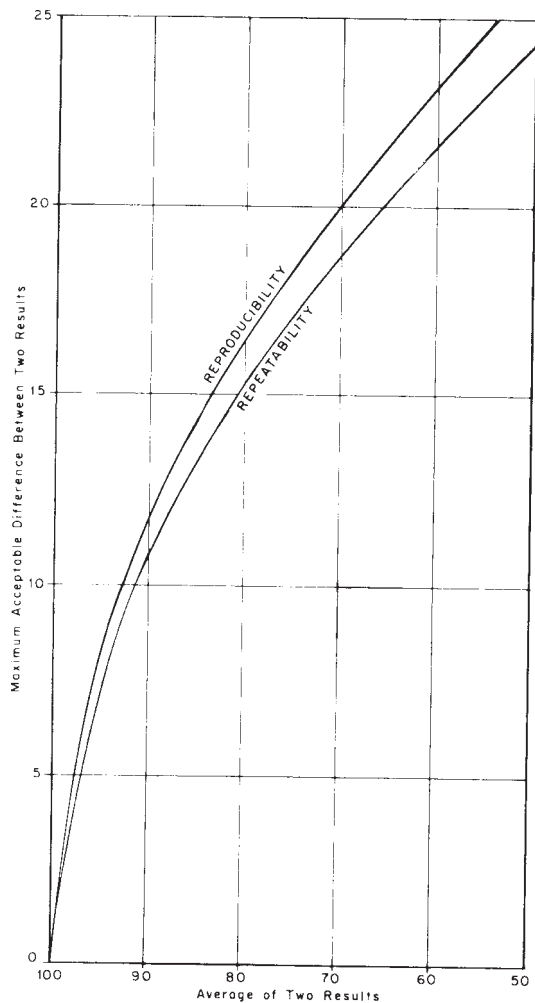


FIG. 10 Field Samples—MSEP-A (Mode A Operation) Variation of Repeatability and Reproducibility of MSEP-A Ratings Obtained for Field Samples (Jet A, Jet A-1, MIL JP 5, MIL JP 7, and MIL JP 8)

identical MIL JP 4 test material would, in the long run, in the normal and correct operation of the test methods, exceed the following values for only one case in twenty (Note 6).

12.2.2.1 *Reference Fuels*—The reproducibility of MSEP-B ratings for MIL JP 4 reference fuels is ten (maximum difference between two measurements obtained by two different operators on the identical test sample using two different instruments at the same test site) (Note 11).

12.2.2.2 *Field Samples*—The reproducibility of MSEP-B ratings for MIL JP 4 field samples is 19 (maximum difference between two measurements obtained by two different operators on the identical test sample using two different instruments at the same test site) (Note 12).

NOTE 11—This result is based on a 1983 ten-laboratory cooperative test program using MIL JP 4 fuels as a reference fuel prepared in accordance with Appendix X1.

NOTE 12—This result is based on a 1983 ten-laboratory cooperative test program using field samples of MIL JP 4 fuels containing fuel system corrosion and icing inhibitors. The fuels did not contain any significant amounts of static dissipator additives (SDA). Due to the large number of additive combinations and concentrations, there may be field samples other than those used to develop the precision of this test method, on which data could be generated, using this test method correctly, that may not fall within the expected limits.

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

13. Keywords

13.1 aviation turbine fuel; coalescence; jet fuel coalescer; MSEP rating; surfactant; water separation

APPENDIXES

(Nonmandatory Information)

X1. PREPARATION OF REFERENCE FLUID BASE

X1.1 Scope

X1.1.1 This procedure describes the preparation within an 8-h day of a 20-L (5-gal) lot of reference fluid base. This procedure has been found to give a filtrate having 100 MSEP Rating.

X1.2 Summary of Procedure

X1.2.1 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 conform to Test Specification D 1655 Jet A, Jet A-1, or Jet B and the aromatic content should be between 10 and 20 volume percent. Reference fluids may also be of MIL JP 4, MIL JP 5, MIL JP 7, and MIL JP 8 fuel origin. 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 standard 99+ rating and, the AOT Standard Ratings shown in Table 3, Table 4, and Table 5, or both.

X1.3 Apparatus

X1.3.1 *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 to 65 mm, and the length above the fritted disk shall be at least 1 m.

X1.3.2 *Siphon*, glass tubing having an outside diameter of 5 to 10 mm with the legs 100 to 150 mm apart. The suction leg shall be 380 to 400 mm long to reach the bottom of the feed container. The other leg shall be 50 to 100 mm longer.

X1.3.3 *Feed Container*, a standard square or round 20-L (5-gal) can in which the sample is obtained.

X1.3.4 *Receiver Can*, a new 20-L (5-gal) epoxy-lined can or one which has been used only with clay-filtered fuel. Plastic containers shall not be used.

X1.3.5 *Funnel*, with a 10 to 20-mm outlet.

X1.3.6 *Graduated Cylinder*, of 0.5 to 1-L capacity.

X1.3.7 *Graduated Cylinder*, of 50 to 100-mL capacity.

X1.3.8 *Beaker*, 2-L capacity.

#### X1.4 Materials

X1.4.1 *Attapulugus 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.

X1.4.2 *Fine Glass Wool*.

X1.4.3 *Isopropyl Alcohol*, 90 %.

X1.4.4 *Toluene*, in a squeeze bottle.

NOTE X1.1—**Warning:** Flammable. Vapor harmful. See Annex A1.2 .

X1.4.5 *Water*, preferably distilled.

X1.4.6 *Salt*, rock salt or equivalent.

#### X1.5 Preparation of Apparatus

X1.5.1 Mount the column vertically.

X1.5.2 Measure approximately 500 mL of clay in the graduated cylinder, tapping gently to settle.

X1.5.3 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.

X1.5.3.1 When water washing of the fuel is required, place approximately 12.5 to 15.0 mm (0.5 to 0.6 in.) of salt on top of the wad of glass wool and then another wad of glass wool on top of the salt.

#### X1.6 Filtration Procedure

X1.6.1 Position a full 20-L (5-gal) 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.

X1.6.2 Place the 2-L beaker under the column.

X1.6.3 Make sure the column stopcock is wide open. Put slight air pressure in the feed can to start the siphon. The glass wool packing should prevent the clay bed from being disturbed at startup.

NOTE X1.2—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.

X1.6.4 As soon as the fuel is flowing through the column outlet, adjust the metering screw to attain a rate of 50 to 60 mL/min. Check by measuring with the small graduated cylinder for 1 or 2-min intervals.

X1.6.5 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 (5-gal) 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 X1.3—**Warning:** 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 X1.6.3 is desirable.

X1.6.6 Recycle the beaker of filtrate to the feed can or discard it.

X1.6.7 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.

X1.6.8 For lengthy storage, purge the receiver can with dry-nitrogen.

NOTE X1.4—At the specified flow rate, the 20-L (5-gal) percolation will be complete in 6 to 6½ h running time.

X1.7 *Clean the Column.*

X1.7.1 Drain the column.

X1.7.2 Dismount the column, open it over a solid waste can, and with the stopcock wide open, blow out the clay.

X1.7.3 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.

X1.7.4 If the column still appears dirty, rinse it thoroughly with hot water, then with distilled water. Invert it and rinse as in X1.7.3 with alcohol, then with acetone and blow dry. This should seldom be necessary.

#### X1.8 Water Washing the Base Fuel

X1.8.1 *Scope*

X1.8.1.1 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.

X1.8.2 *Summary of Procedure*

X1.8.2.1 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 X1.6.

#### X1.9 Procedure

X1.9.1 Mix 0.95 L (1 qt) of water (X1.4) thoroughly with 19 L (5 gal) of base fuel by agitating by any convenient means.

X1.9.2 Allow the container to stand for sufficient time to allow the water to completely settle out to the bottom of the container.

X1.9.3 Remove the water from the bottom of the container using a pump, pipet, or any other means available.

X1.9.4 Repeat X1.9.1, X1.9.2, and X1.9.3, as required, to assure removal of all water soluble substances and then proceed to the filtration process.

X1.9.5 Initiate the filtration process of X1.6 using a filtration media prepared as described in X1.5.3.1.

## X2. SAMPLING TECHNIQUE

X2.1 For any test that seeks the presence of trace constituents, steps must be taken to assure testing of a representative sample. The round-robin 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 aviation turbine 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.

X2.2 *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 X2.1—New cans, not epoxy-lined, are sometimes coated with surfactant-type roll oils or solder flux residues which can affect MSEP test results. Epoxy cans also may have mold release or similar residues which can also affect the MSEP 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 an 0.8- $\mu$ m 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 taking the sample.

X2.2.1 *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.

X2.2.2 *Sample Line*—The line may consist of a short 6.4 to 12.7-mm ( $\frac{1}{4}$  to  $\frac{1}{2}$ -in.) 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.

X2.2.3 *Taking the Sample*—Flush the sample line with at least 0.95 L (1 qt) of the fuel to be sampled. Open and close the sample valve several times. Rinse the sample can with three separate 0.95-L amounts of the fuel to be sampled (for a 1-gal can). Include the cap and inner seal, if used, in the rinsing. Draw the sample and put the cap in place.

## X3. CORRELATION OF MICRO-SEPAROMETER MSEP-A RATINGS

### X3.1 Introduction

X3.1.1 The program to establish the precision of the Micro-Separometer involved cooperative testing by ten laboratories using seven Jet A blends and seven Jet B blends and parallel testing with Test Method D 3602. The correlation of MSEP-A Ratings is shown below.

X3.1.2 Earlier programs with Test Method D 3602 involved cooperative testing in conjunction with Test Method D 2550. Correlations of MSS Ratings with WSIM Ratings established at that time have been extended to MSEP-A Ratings using the MSS rating as the common reference. These correlations appear in RR: D02-1050 on file at ASTM Headquarters.

### X3.2 Precision of MSEP-A Ratings

X3.2.1 Using fuels that simulated field experience by addition of corrosion inhibitors, etc., as well as reference blends prepared in accordance with 7.5, the precision of the Micro-Separometer was shown to be equivalent to the precision of Test Method D 3602 Minisonic Separometer.

X3.2.2 Analysis revealed that precision of MSEP-A Ratings varies with rating levels as shown in Fig. 9, which was developed originally for Test Method D 2550 and confirmed with Test Method D 3602.

### X3.3 Correlation of MSEP-A and MSS Ratings

X3.3.1 The relationship between the MSEP-A and MSS Ratings is a function of fuel type as described in the following equations:

$$MSEP-A (\text{Jet A}) = 1.276 (MSS) - 23.805 \quad (X3.1)$$

$$MSEP-A (\text{Jet B}) = 1.338 (MSS) - 32.366$$

$$MSEPA (\text{both fuels}) = 1.247 (MSS) - 22.952$$

### X3.4 MSEP-A Ratings Level Versus Fuel Type

X3.4.1 The Micro-Separometer Rating level for Jet B fuel is higher than Jet A fuel using either the standard dispersing agent (7.3) or other additives typical of fuels in the field. In Test Method D 3602 the relationship for Jet B fuel is shown in Note 7 in terms of Test Method D 2550 WSIM Ratings. Using Test Method D 3602 MSS Ratings as the common reference, the MSEP-A relationship for Jet B fuel containing standard dispersing agents is as follows:

$$MSEP-A (\text{JET B}) = 0.60 \text{ WSIM} + 40 \quad (X3.2)$$

### X3.5 Correlation of MSEP-A Ratings with Field Experience

X3.5.1 Fuel blends that simulate field samples reveal that fuels containing corrosion inhibitors exhibit higher MSEP-A Ratings than blends with other additives including standard dispersing agents.

X3.5.2 In Note 9 of Test Method D 3602 reference is made to RR: D02-1050 which contains information relating the MSS Ratings of fuel to actual field performance of ground handling equipment. Since MSEP-A ratings are essentially equivalent to MSS ratings, this report on file at ASTM Headquarters would also correlate MSEP-A ratings with field experience.

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