Designation: D7224 - 08 <u>D7224 - 12</u>

An American National Standard

Standard Test Method for Determining Water Separation Characteristics of Kerosine-Type Aviation Turbine Fuels Containing Additives by Portable Separometer¹

This standard is issued under the fixed designation D7224; 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.

INTRODUCTION

This test method was developed to satisfy three objectives: (1) Develop a test method that would respond in the same manner as Test Method D3948 to strong surfactants, but not give low micro-separometer (MSEP) ratings to fuels containing weak surfactants (additives) that do not degrade the performance of commercial filter separator elements; (2) Use filter media in the coalescer test that would be representative of the filtration media in commercial filter separator elements; and (3) Improve the precision of the test method compared to Test Method D3948.

This test method was developed using material that is representative of coalescing materials currently used in commercial filter separator elements. The fiberglass coalescing material used in Test Method D3948 was suitable for coalescing filters in use when that test method was developed, but developments in coalescing elements in the intervening years have resulted in improved materials that are not affected by weak surfactants. Test Method D3948 yields low results on some additized fuels that do not affect the performance of filter separators (coalescing filters) in actual service. Since this test method was developed with material that is representative of the media used in current filter separators, the results by this test method are more relevant to performance in current filter separators.

1. Scope*

- 1.1 This test method covers a rapid portable means for field and laboratory use to rate the ability of kerosine-type aviation turbine fuels, both neat and those containing additives, to release entrained or emulsified water when passed through fiberglass coalescing material.
- 1.1.1 This test method is applicable to kerosine-type aviation turbine fuels including: Jet A and Jet A-1 (as described in Specification D1655); JP-5, JP-7, JP-8, and JP-8+100. (See Section 6.)
 - 1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.
- 1.3 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 warning statements, see 8.2-8.5.

2. Referenced Documents

2.1 ASTM Standards:²

D1655 Specification for Aviation Turbine Fuels

D2550 Method of Test for Water Separation Characteristics of Aviation Turbine Fuels (Withdrawn 1989)³

D3602 Test Method for Water Separation Characteristics of Aviation Turbine Fuels (Withdrawn 1994)³

D3948 Test Method for Determining Water Separation Characteristics of Aviation Turbine Fuels by Portable Separameter

¹ 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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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 The last approved version of this historical standard is referenced on www.astm.org.



D4306 Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination

2.2 Military Standards:⁴

MIL-DTL-5624 Turbine Fuel, Aviation Grades JP-4, JP-5, and JP-5/JP- 8 ST

MIL-DTL-25524 Turbine Fuel, Aviation, Thermally Stable

MIL-DTL-38219 Turbine Fuels, Low Volatility, JP-7

MIL-DTL-83133 Turbine Fuel, Aviation, Kerosene Types, NATO F-34 (JP-8), NATO F-35, and JP-8+100

3. Terminology

3.1 Definitions:

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

3.1.1.1 Discussion—

This test method uses the same instrument, Micro-Separometer, that is used in Test Method D3948. As in Test Method D3948, the MSEP 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 MSEP rating is expected to show good water-separating properties when passed through a filter-separator (coalescing-type filter) in actual service.

- 3.1 Definitions of Terms Specific to This Standard: Definitions:
- 3.1.1 <u>MCellMicro-Separometer⁵ Coalescer, rating (MSEP⁵ rating), n—in the aviation fuel industry, a registered trademark of EMCEE Electronics, Inc.numerical value indicating the ease of separating emulsified water referring to a particular coalescing filter element specifically designed for this test method.from aviation (jet) fuel by coalescence as affected by the presence of surface active materials (also known as surface active agents or surfactants).</u>

⁵ A trademark of EMCEE Electronics, Inc., 520 Cypress Ave., Venice, FL 34285, www.emcee-electronics.com.

3.1.1.1 Discussion—

MSEP 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.

- 3.2.2 reference fluid bases, n—fuels that have been carefully cleaned in a prescribed manner to remove all surface-active contaminants, and having a minimum MSEP rating of 97.
- 3.1.2 reference fluids, fluid, n—in MSEP and DSEP⁵ [diesel separability] water separability tests, a reference fluid bases base to which a prescribed quantities quantity of a known surface active agent have has been added.

3.2.3.1 Discussion—

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

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 reference fluid base, n—in aviation MSEP water separability tests, jet fuel that has been cleaned in a prescribed manner to remove all surface-active contaminants (agents), and having a minimum MSEP rating of 97.
- 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—

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



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

- 3.1.5 <u>surfactants</u>, <u>strong surfactant</u>, n—<u>in petroleum fuels</u>, surface active <u>materials that could disarm (deactivate) filter separator (coalescing) elements so that free water is not removed from the fuel in actual servic. <u>material that disarms filter separator elements</u>, allowing water to pass.</u>
- 3.2.4.1 strong surfactants—surface active materials that disarm filter separator elements. Strong surfactants can be refinery process chemicals left in the fuel or contaminants introduced during transportation of the fuel.

3.1.5.1 Discussion—

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

3.2.4.2 weak surfactants—surface active materials that are typically certain types of additives that do not adversely affect the performance of filter separator elements in actual service.

- 3.1.6 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 MCell⁶ Coalescer, n—referring to a particular coalescing filter element specifically designed for this test method.
 - 3.3 Abbreviations:
 - 3.3.1 *AOT*—aerosol OT (see 8.1).
 - 3.3.2 *MSEP*—micro-separometer. Micro-Separometer.
 - 3.3.3 SDA—static dissipator additive.

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 specific fiberglass coalescer, the MCell Coalescer,⁶ and the effluent is analyzed for uncoalesced water (that is, dispersed water droplets) by a light transmission measurement. The Micro-Separometer instrument has an effective range of 50-to-100 scaled to the nearest whole number. A test can be performed in 5 to 10 minutes.

5. Significance and Use

- 5.1 This test method provides a measurement of the presence of surfactants in aviation turbine fuels. Like previous obsolete Test Methods D2550 and D3602 and current Test Method D3948, this test method can detect trace amounts of refinery treating chemicals in fuel. The test methods can also detect surface active substances added to fuel in the form of additives or picked up by the fuel during handling from point of production to point of use. Some of these substances degrade the ability of filter separators to separate free water from the fuel.
- 5.2 This test method yields approximately the same (low) MSEP ratings as Test Method D3948 for fuels that contain strong surfactants.
- 5.2.1 This test method will give approximately the same MSEP ratings for Jet A, Jet A-1, JP-5, JP-7, and JP-8 fuels as Test Method D3948 when testing reference fluids.
- 5.3 The MSEP ratings obtained by this test method are less affected by weak surfactants than Test Method D3948. Somewhat higher MSEP ratings for Jet A, Jet A-1, JP-5, JP-7, and JP-8 fuels are obtained by this test method than those obtained by Test Method D3948 when additives such as static dissipater additives (SDA) and corrosion inhibitors are present in the fuel. This correlates with the satisfactory performance of filter separators for such fuels, when wet. However, these same additives adversely affect the MSEP ratings obtained by Test Method D3948 by erroneously indicating that such additized fuels would significantly degrade the ability of filter separators to separate free water from the fuel in actual service.
- 5.4 The Micro-Separometer <u>instrument</u> has an effective measurement range from 50 to 100. Values obtained outside of those limits are undefined and invalid.

Note 1—In the event a value greater than 100 is obtained, there is a good probability that light transmittance was reduced by material, typically water, contained in the fuel that was used to set the 100 reference level. During the coalescing portion of the test, the contaminating material as well as the 50 \pm 1 μ L of distilled water was subsequently removed during this 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 solid 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 A registered trademark of EMCEE Electronics, Inc., 520 Cypress Ave., Venice, FL 34285.34285, www.emcee-electronics.com.



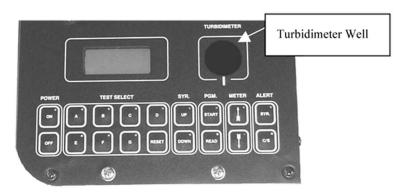


FIG. 1 Micro-Separometer Mark V Deluxe and Associated Control Panel

7. Apparatus

7.1 *Micro-Separometer Instrument*⁷ is used to perform the test. The unit is completely portable and self-contained, capable of operating on an (optional) 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 effect battery recharge. The power cords, test accessories and operators manual can be packed in the cover of the lockable case.

Note 2—An extensive study was performed to verify that the Mark X Micro-Separometer <u>instrument gives</u> equivalent results to the Mark V Deluxe Micro-Separometer. Micro-Separometer instrument. See Research Report RR:D02-1647.⁸

Note 3—The Mark X has a universal power supply and requires only one power cord as compared to the Mark V Deluxe that requires individual power cords for different voltages.

- 7.1.1 Review the Operating Manual of the Micro-Separometer instrument that is furnished with each unit (and is also available from the manufacturer's website) for operating instructions. The instrument is not field repairable. Also note that this instrument is designed to perform a number of different functions in addition to this specific test method.
- 7.1.2 The Micro-Separometer Mark V Deluxe and Mark X <u>instruments</u> and associated control <u>panel panels</u> are shown in Fig. 1 and Fig. 2, respectively. 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 1 lists the manual and audio operating characteristics of the instrument.
- 7.1.3 All of the controls are located in a push-button array on the control panel. The push-buttons illuminate when depressed thus indicating operational status. A circuit breaker located on the control panel provides protection for the ac power circuit.
- 7.1.3.1 The Mark X has an LCD display on the control panel that provides information to the operator during the test. The information includes test status and an error code that defines a malfunction in the <u>Micro-Separometer.Micro-Separometer</u> instrument.
- 7.1.4 The turbidimeter is located under the main control panel and consists of a well in which the sample vial is placed (in a specified orientation), a light source and a photocell.
- 7.1.5 By depressing the ON push-button, the electronic circuits are energized. The ON push-button pulses on and off when the instruments are being operated by an ac source and remains constantly on when the battery (dc) pack is used. The lettered push-buttons will sequentially illuminate indicating READY operational status.

Note 4—Of the lettered (A-G) pushbuttons on the control panel of the Mark V Deluxe, only the A pushbutton is applicable to this test method. Of the lettered (Jet A – Diesel) pushbuttons on the control panel of the Mark X, only the Jet A pushbutton is applicable to this test method.

- 7.1.6 The RESET push-button can be depressed at any time to cancel the test in progress and restore the program to the initial start mode. The lettered push-buttons commence to sequentially illuminate, thus indicating a READY operational status enabling test mode selection.
 - 7.2 Mark V Operation:
 - 7.2.1 Depress the A push-button to select test Mode A. The depressed push-button and the START push-button will illuminate.
- 7.2.2 The START push-button, 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.
- 7.2.3 The START push-button, when depressed after the second CLEAN cycle initiates the automatic program sequence causing the read indicator and the two ARROWED push-buttons to illuminate, indicating that a full-scale adjustment period is in effect. A numerical value also appears on the display.

⁷ The sole source of supply of the apparatus, the Model 1140 Micro-Separometer Mark V Deluxe and Mark X, X instruments, 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, which you may attend.

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

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FIG. 2 Micro-Separometer Mark X Instrument and Associated Control Panel

TABLE 1 Manual and Audio Operating Characteristics of the Model 1140 Micro-Separometer Instrument

Available Test Mode(s) Function	Mark V Deluxe	Mark X	
Test Mode—Select Mode A:			
Depress	A push-button	Jet A Pushbutton	
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	
Cancel Automatic Sequence:			
Depress	RESET push-button	RESET push-button	
1st Meter Read			
1st Meter Adjust	Depress ARROWED push-buttons	No action required	
2nd Meter Read			
2nd Meter Adjust	Depress ARROWED push-buttons	No action required	
Collect Sample	Short Tone and C/S	Short Tone and C/S	
	Annunciator Lamp Illuminates	Annunciator Lamp Illuminates	
3rd Meter Read			
Record Measurement	Pulsed Tone Sounds 5 s into 3rd Meter Reading	Steady tone	

- 7.2.4 By depressing the appropriate ARROWED push-button, the displayed value on the meter can be increased or decreased, as required, to attain the 100 reference level for the vial of fuel sample in the turbidimeter.
 - 7.3 Mark X Operation:
- 7.3.1 Selection of Test Mode A program is accomplished by depressing the Jet A lettered pushbutton. The depressed pushbutton illuminates and the sequential illumination of the other lettered pushbuttons ceases. The CLEAN 1 pushbutton also illuminates.
- 7.3.2 The first and second clean cycles are initiated by depressing the CLEAN 1 and CLEAN 2 pushbuttons. The RUN pushbutton will illuminate at the end of the second clean cycle.
 - 7.3.3 The automatic portion of the test sequence is initiated by depressing the RUN pushbutton.
- 7.3.4 The 100 reference level for the vial of fuel in the turbidimeter is set automatically and does not require any adjustment. If the turbidimeter could not auto adjust to 100, the error alert indicator illuminates and an ERR-04 is displayed.
 - 7.4 Accessory equipment and expendable materials needed to perform the test are shown in Fig. 3 and consist of the following:
- 7.4.1 *Connector* (A)—A plastic connector used to affix the MCell Coalescer⁶ to the aluminum syringe barrel. The connector is not required with the plastic syringe.
 - 7.4.2 Syringe Plug (B)—A plastic plug used to stopper the syringe during the CLEAN and EMULSION cycles.
 - 7.4.3 Syringe, (Barrel (C) and Plunger (D)), either:
 - 7.4.3.1 A single use plastic syringe and plunger, which is furnished in each six-pack (7.5), or
- 7.4.3.2 A reusable aluminum syringe and plunger. The barrel of the aluminum syringe has an internal, circumferential scribe mark that indicates the 50-mL fill level (Fig. 4). The plunger has an external, circumferential scribe mark that indicates the point of insertion in the syringe barrel to where the plunger tip comes into contact with the fuel.
- 7.4.3.3 Use of syringes other than those demonstrated to be free of surfactant contamination in a precision program such as described in Section 15 will render test results invalid.
 - 7.4.4 Vials, (E)—A 25-mm outside diameter vial premarked for proper alignment in the turbidimeter well.
- 7.4.5 *MCell Coalescer*, ⁶ (*F*)—An expendable coalescer cell with a tapered end in which the plastic connector is inserted. The other end of the plastic connector is inserted in the tapered end of the syringe barrel. Coalescer is labeled in red background with black lettering:

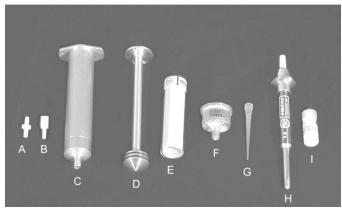


FIG. 3 Test Items and Expendables



FIG. 4 Syringe Barrel with Scribe Mark

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FIG. 5 Six-Pack and Test Accessories

MCell, JET FUEL. D7224

- 7.4.6 Plastic Tip and Pipet, (G) with (H)—A disposable plastic tip and an automatic 50 μ L hand pipet. Plastic tips are supplied with each six-pack and a pipet is supplied with each Micro-Separometer.
- 7.4.7 *Distilled Water (I)*—A clean container of double-distilled water. A container of double-distilled water is supplied with each six-pack. A holder for the water container is affixed to the control panel (Fig. 11).
- 7.4.8 *Beaker, Catch Pan, or Plastic Container*—(supplied with each Micro-Separometer) used to receive the waste fuel during the coalescing period of the test (not shown).

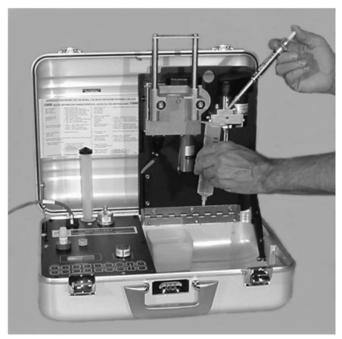


FIG. 6 Water Addition



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FIG. 7 Emulsification

7.5 A plastic connector, syringe plug, test sample vial, MCell Coalescer,⁶ plastic syringe with plunger, pipet tip and distilled water are used in each test. These expendable materials are packaged so that each package has sufficient expendables to perform one test. Six of these packages, including a container of double-distilled water, are included in a kit containing supplies for six tests. This kit is termed the Micro-Separometer Six-Pack (Fig. 5).⁹

8. Reagents and Materials

- 8.1 Aerosol OT (AOT), solid (100 % dry) bis-2-ethylhexyl sodium sulfosuccinate.
- 8.2 Toluene, ACS reagent grade. (Warning—Flammable. Vapor harmful.).

⁹ A kit containing six each of these test expendables is available from EMCEE Electronics, Inc., 520 Cypress Ave., Venice, FL 34285.

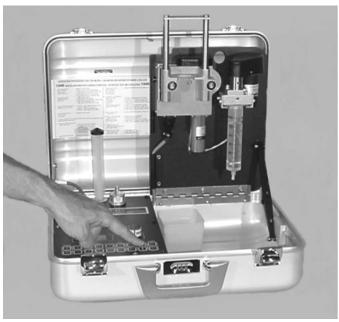


FIG. 8 Meter Adjustment



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FIG. 9 Insert Plunger

- 8.3 Dispersing Agent, Toluene solution (Warning—Flammable. Vapor harmful.) containing 1 mg of AOT per millilitre of toluene.
- 8.4 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.)
- 8.5 Reference Fluid—(Warning—Flammable. Vapor harmful.) A fluid used for checking the operational performance of the Micro-Separometer instrumentation, consisting of increasing concentrations (0.0 to 0.8 mL/L) of dispersing agent added to the reference fluid base. The MSEP ratings for this range of concentration appear in Table 2 for Jet A, Jet A-1, JP-5, JP-7, and JP-8 fuels using Mode A. The reference fluids are tested as described in Section 13. If the results do not fall within the range of limits



FIG. 10 Syringe Assembly

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shown in Table 2, the reference fluid shall be discarded and a fresh quantity of reference fluid prepared and the validation repeated. Repeated out-of-tolerance test results are cause for returning the instrument to the factory for adjustment and calibration. (Refer to the Operators Manual. 10)

Note 5—The reference fluid base without any dispersing agent should have a minimum MSEP rating of 97; otherwise, the results may not be indicative of the accuracy of the instrument.

- 8.5.1 Reference fluids shall be prepared by adding dispersing agent (8.3) to a suitable quantity of reference fluid base contained in a properly equilibrated container or graduate.
- 8.5.1.1 If a new or non-equilibrated container is used, the additive can adsorb on the walls and the MSEP ratings can erroneously improve significantly. To equilibrate the container surface, an additive blend should be held for a minimum of 24 h in the container, discarded, and replaced with a fresh blend.
 - 8.6 Water, clean, double-distilled and surfactant-free (furnished with each six-pack).
 - 8.6.1 Use of water other than double-distilled water (such as tap or deionized 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 A Micro-Separometer Instrument Operation Manual is furnished with each instrument. It may also be downloaded from www.emcee-electronics.com.



FIG. 11 Coalescence

TABLE 2 Expected Performance with Jet A, Jet A-1, JP-5, JP-7, or JP-8 Reference Fluid Containing a Dispersing Agent Using Mode A Operation

	Concentration of Dispersing	Standard Rating -	Limits for Acceptable Performance ^A		
Agent, mL/L	natility —	min	max		
	0.0	99	97	100	'
	0.2	89	0/2/82/1/	94	
	to 10 c/ 0.4 double	80 7267	60 769 7 /11	e-8aa(88)2203	
	0.6	5/515172 / 50 /	59	6-0aa(83)2205	
	0.8	65	51	77	

^A Expected range of values obtained by using increasing amounts of dispersing agent used to verify instrument calibration.

10. Sampling and Sample Preparation

- 10.1 Special precautions concerning sampling technique and sample containers are described 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 specimen from the container, wipe the container outlet thoroughly with a clean, lintless wiper; pour the test specimen into a clean beaker or directly into the barrel of the test syringe.
- 10.1.1 Test method results are known to be sensitive to trace contamination from sampling containers. Refer to Practice D4306 for recommended sampling containers.
- 10.2 Under no circumstances shall a sample be pre-filtered, because filter media can remove the very materials, surfactants, that the test method is designed to detect.
 - 10.2.1 Haze in a sample can be indicative of free water, which can result in MSEP ratings in excess of 100.
- 10.2.2 If the sample is contaminated with particulate matter or haze, allow such materials to settle out of the sample before testing.
- 10.2.3 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.
- 10.3 If the sample is not within the test temperature limits, 18 to 29°C (65 to 85°F), 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 (80°F).