Standard Test Method for Determining Water Separation Characteristics of Kerosine-Type Aviation Turbine Fuels Containing Additives by Portable Separometer[1]

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 Separometer

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

D7261 Test Method for Determining Water Separation Characteristics of Diesel Fuels by Portable Separometer

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[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.J0.05 on Fuel Cleanliness.

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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.

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

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 For definitions of the terms used in this test method that are not shown below, refer to Test Methods D3948 and D7261.

3.2 *Definitions:*

3.2.1 *Micro-Separometer*[5] rating (MSEP[5] rating), *n—in the aviation fuel industry*, a numerical value indicating the ease of separating emulsified water from aviation (jet) fuel by coalescence as affected by the presence of surface active materials (also known as surface active agents or surfactants).

3.2.1.1 *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, n—in MSEP and DSEP*[5]*, [diesel separability] water separability tests*, a reference fluid base to which a prescribed quantity of a known surface active agent has been added.

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

3.2.3 *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.

3.2.4 *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.

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

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

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

3.2.6 *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.

3.3 *Definitions of Terms Specific to This Standard:*

3.3.1 *MCell*[6] Coalescer, *n*—referring to a particular coalescing filter element specifically designed for this test method.

3.4 *Abbreviations:*

3.4.1 *AOT*—aerosol OT (see 8.1).

3.4.2 *DSEP*—diesel separability.

3.4.3 *MSEP*—micro-separometer.

3.4.4 *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 min to 10 min.

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 instrument 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

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

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

[6] A registered trademark of EMCEE Electronics, Inc., 520 Cypress Ave., Venice, FL 34285, www.emcee-electronics.com.

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

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 µL ± 1 µL 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.

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 instrument gives equivalent results to the Mark V Deluxe 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 instruments and associated control panels 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 Micro-Separometer 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.

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[7] The sole source of supply of the apparatus, the Model 1140 Micro-Separometer Mark V Deluxe and Mark 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,[1] which you may attend.

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

FIG. 2 Micro-Separometer Mark X 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 Annunciator Lamp Illuminates	Short Tone and C/S Annunciator Lamp Illuminates
3rd Meter Read		
Record Measurement	Pulsed Tone Sounds 5 s into 3rd Meter Reading	Steady tone

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.

7.2.4 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 push-button 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 Coalescer[6] 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.

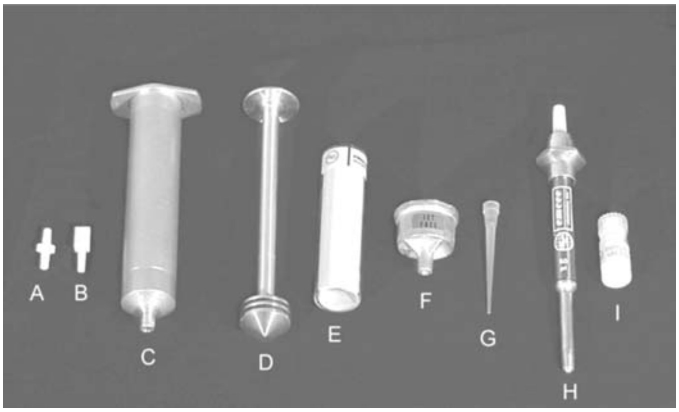

FIG. 3 Test Items and Expendables

FIG. 4 Syringe Barrel with Scribe Mark

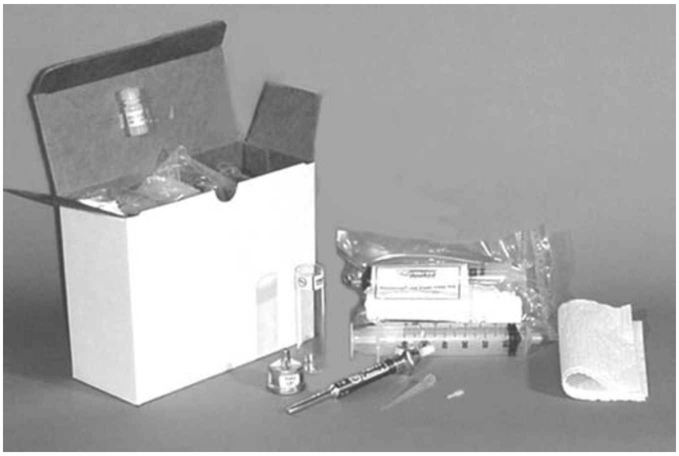

FIG. 5 Six-Pack and Test Accessories

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:

MCell, JET FUEL. D7224

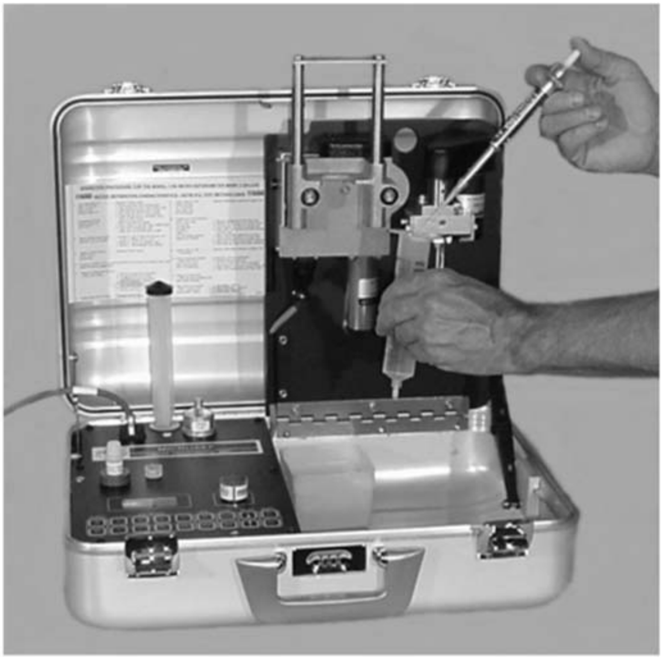

FIG. 6 Water Addition

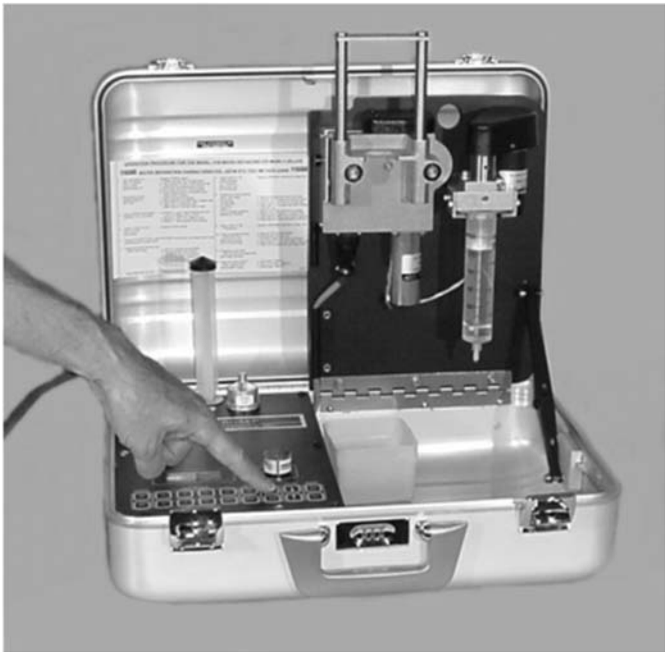

FIG. 7 Emulsification

7.4.6 *Plastic Tip and Pipet, (G) with (H)*—A disposable plastic tip and an automatic 50 µL 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).

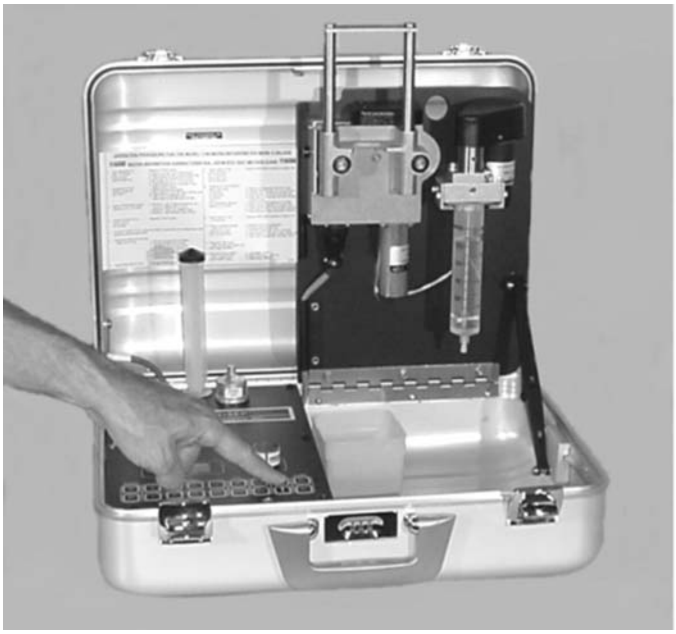

FIG. 8 Meter Adjustment

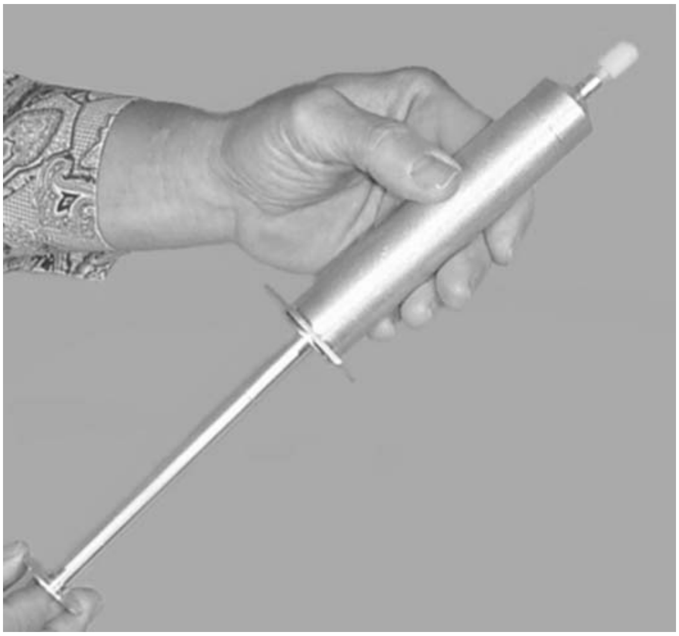

FIG. 9 Insert Plunger

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]

FIG. 10 Syringe Assembly

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.).

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 mL/L 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 shown in Table 2, the reference fluid

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

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 Agent, mL/L	Standard Rating	Limits for Acceptable Performance[A]	
		min	max
0.0	99	97	100
0.2	89	82	94
0.4	80	69	88
0.6	72	59	83
0.8	65	51	77

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

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. 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 °C to 29 °C (65 °F 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).

11. Preparation of Apparatus

11.1 Locate the instrument on a clean workbench in an area where the temperature is between 18 °C and 29 °C (65 °F and 85 °F) and does not vary more than ±3 °C (5 °F).

11.2 Open the case and remove the power cord and test accessories from the lid. Raise the right panel until completely vertical and locked in place. If ac power is available, connect the power cord, and turn the instrument on. If the internal battery power is used, ensure 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. To recharge the battery, connect the instrument to an ac power

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[10] A Micro-Separometer Instrument Operation Manual is furnished with each instrument. It may also be downloaded from www.emcee-electronics.com.

source for at least 16 h (full charge) prior to use. Depending on the age of the battery, approximately 25 tests can then be performed.

NOTE 6—If the battery in the Mark X is not charged sufficiently to run a test, an ERR-06 will be displayed indicating that the battery must be recharged.

11.2.1 Turn the Mark V Deluxe and Mark X instruments on by depressing the switch (push-button) marked ON. 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. Flickering of the power indicator light, during any portion of a test sequence being performed using battery power, indicates that recharging is necessary.

11.3 Have ready a supply of vials, MCell Coalescers,[6] plastic syringes with plungers, syringe plugs, pipet tips, as well as a clean container of double-distilled water. All of these expendable items are furnished in each six-pack. Also have the pipet that is furnished with each Micro-Separometer instrument readily available. If the aluminum syringe and plunger are being used instead of the plastic syringe, have a plastic connector available as well.

11.3.1 Under most circumstances, either the plastic syringe or the aluminum syringe may be used. Research and round robin studies during the development of this test method demonstrated that aluminum and plastic syringes had equivalent precision. Under very dry ambient conditions (when electrostatic charges can readily develop in the plastic syringe, giving erroneously high results), or in case of dispute, use the aluminum syringe.

11.4 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.

11.4.1 The acceptable drive travel time range for Mode A operation is 45 s ± 2 s.

NOTE 7—Slow syringe drive travel times exceeding the upper limit (47 s) will cause the final results to measure high; conversely, fast travel times below the lower limit (43 s) will cause the final results to measure low.

11.4.2 Both the Mark V Deluxe and Mark X 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.

11.4.2.1 An occasional out-of-tolerance alert may be experienced due to some intermittent condition that probably will not be indicative of instrument failure. To verify that an alert is only an intermittent condition, time the syringe travel from when the syringe drive begins down to when it stops. This must be performed during an actual test so that proper back-pressure is present. Repeated alerts are cause for returning the instrument to the factory for adjustment.

11.4.2.2 *Mark X*—During a test, the error alert indicator will illuminate and ERR-03 will be displayed indicating an out of tolerance syringe travel time. Error alerts ERR-01 and ERR-02 will be displayed if the syringe stalls while traveling up or down, respectively.

12. Calibration and Verification

12.1 The instrument is calibrated at the factory by using in-house test fixtures.

12.2 Instrument performance, especially for field use, may be verified by performing MSEP tests using a dilution of the dispersing agent (as prepared in 8.3), a reference fluid base (as prepared in Appendix X1), and double-distilled water. A 10:1 dilution is prepared by diluting 10 mL of dispersing agent with 90 mL of toluene (8.2). Since 1 mL of dilution is equal to 0.1 mL of dispersing agent, 50 mL 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. This facilitates using the 50 mL pipet (7.4.6) to add increments of 0.1 mL ⁄L of dispersing agent, as well as the double-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.

12.3 If the results do not fall within the range of limits shown in Table 2, the reference fluid shall be discarded, 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.

13. Procedure

13.1 *Select Mode A Operation:*

13.1.1 Depress push-button A (Mark V) or Jet A (Mark X) for Mode A operation. Sequential illumination of the lettered push-buttons will cease and the depressed push-button will stay lit. The correct syringe drive speed is set automatically.

13.2 Depending on which syringe is used, the following procedure shall be followed:

13.2.1 *Plastic Syringe*—Remove the plunger from a new 50 mL plastic 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 *Aluminum Syringe*—Flush the barrel of the 50 mL aluminum syringe, and wipe the plunger tip with toluene or clean jet fuel using a clean, lintless wipe to remove any residue deposited from the previous test. Insert a plug into the exit hole of the syringe barrel, add 50 mL ± 1 mL of fuel to the scribe mark in the syringe barrel (see Fig. 5), and place the syringe barrel on the emulsifier mount, turning to lock in place.

13.2.3 Ensure that the syringe barrel is properly aligned concentrically with the mixer shaft and not touching the propeller. Proper alignment can be verified by grasping the syringe barrel and moving it until the propeller on the end of the mixer shaft is free and not touching. Misalignment can cause damage to the propeller by the barrel of the aluminum syringe, slow the mixer causing a poor emulsion, and create shavings from the plastic syringe to form and collect on the coalescer filter material, or both, resulting in erroneous test results. This alignment requirement applies to all instruments manufactured prior to July 1988 which 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.

13.3 Initiate the first CLEAN cycle by depressing the START (Mark V) or CLEAN 1 (Mark X) push-button 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.)

NOTE 8—The Mark X will illuminate ERROR ALERT and display an ERR-05 when the emulsifier speed is outside of acceptable limits.

13.4 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. 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.

13.5 *Second Clean Cycle:*

13.5.1 *Mark V Deluxe*—Initiate the second CLEAN cycle by sequentially pressing the RESET, A, and the START pushbuttons, as designated by the annunciator lights.

13.5.2 *Mark X*—Initiate the second CLEAN cycle by pressing the CLEAN 2 pushbutton.

13.6 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 turbidometer well. Rotate the vial until the black mark on the vial aligns with the white line on the front of the turbidometer well (Fig. 1).

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

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

13.8 Using a fresh plastic tip on the hand pipet, add 50 µL 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. 6) 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.

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

13.10 The Mark V automatic portion of the applicable test mode program listed in Table 3 is initiated, with the syringe in place, by depressing the START push button.

TABLE 3 Test Sequence (Mode A Operation)

Micro-Separometer Action	Operator Activity	Time, min:s	
		Test Sequence (Time)	Elapsed Time
Start sequence	depress start switch	0:00	0:00
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

13.10.1 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. 8). If the adjustment cannot be completed at this time, final adjustment may be accomplished during the second meter adjust period occurring later in the test sequence.

13.11 The Mark X automatic portion of the applicable test mode program listed inTable 3 is initiated, with the syringe in place, by depressing the RUN pushbutton.

13.11.1 The automatic program starts with a read meter indication (four short tones) followed by a 2 s to 30 s adjustment period. During this period, the turbidimeter will automatically adjust to 100. If the adjustment cannot be completed at this time, the ERROR ALERT will illuminate and an ERR-04 will be displayed.

13.12 If for any reason it is desired to interrupt the sequence and start over, pushing the RESET push-button on the Mark V or the Mark X instrument will cancel the test in progress and reset the program to the beginning of the CLEAN segment of the test cycle.

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

NOTE 9—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.

13.14 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. 9). 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 the plunger to the scribe mark on the plunger (the 50 mL mark). Use a clean wiper over the exit hole to capture the small amounts of fuel which may be extruded as foam. Affix the plastic connector to the barrel of the aluminum syringe and attach a new MCell Coalescer[6] to the other end of the connector (Fig. 10). The plastic connector is not required with a plastic syringe.

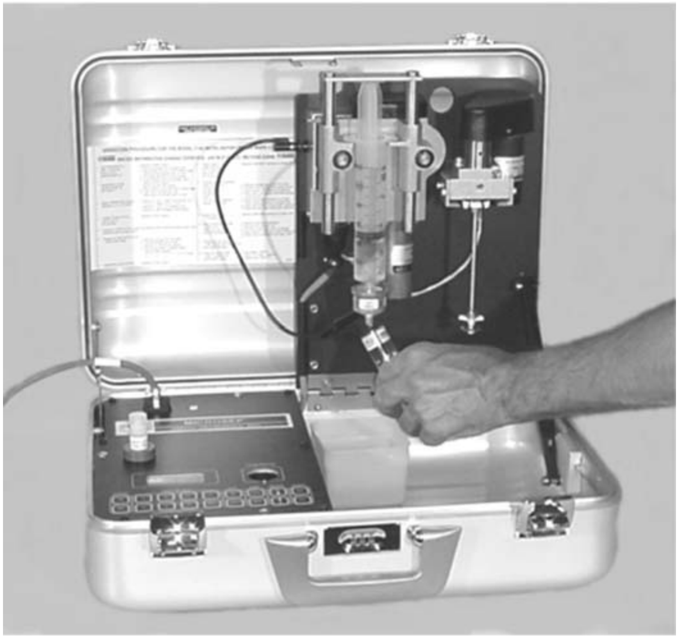

FIG. 12 Taking Sample

13.14.1 To facilitate ease of insertion of the plunger into the syringe barrel, wet the tip of the plunger with some of the sample fuel.

13.14.2 Place the entire syringe assembly into the syringe drive mechanism (Fig. 11). 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 the push bar of the syringe drive mechanism. Position a waste container beneath the MCell Coalescer[6] to collect the unwanted portion of the processed fuel sample during the coalescing period.

13.14.3 Electrically bond MCell Coalescers[6] to the Micro-Separometer instrument to prevent buildup of an electrostatic charge that could result in ignition of flammable test fluids. Each Mark V and Mark X 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 MCell Coalescer[6] and insert the plug into the chassis ground jack (Fig. 11).

13.15 Four short tones will indicate the second meter adjustment period. If required (Mark V), adjust the meter reading to 100. The syringe drive mechanism will start down at the end of the meter adjust period forcing the water/fuel emulsion through the MCell Coalescer[6]. During this operation remove the vial from the turbidimeter well, and discard the fuel.

13.16 Collect the last 15 mL of fuel sample being processed from the MCell Coalescer[6] (Fig. 12) 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 MCell Coalescer.[6]

13.17 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. (Fig. 1) 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.

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

13.18.1 Read the MSEP rating at the midpoint of the 10 s meter read cycle indicated by a short 1 s tone.

NOTE 10—The Mark X stores the reading until another test is performed.

NOTE 11—Numerical values obtained outside the measurement range from 50 to 100 are undefined and invalid (see 5.4 and Note 1)

14. Report

14.1 Report the results obtained in 13.18.1 as the MSEP rating, and reference this test method.

15. Precision and Bias

15.1 MSEP ratings obtained by this test method have precision as determined by round robin programs conducted at a common site, as follows:

NOTE 12—These results are based on an eight operator cooperative test program conducted in March 2002, using a combination of ten samples consisting of a base fuel, two reference fuels, and seven 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.[11] 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.

15.1.1 *Repeatability*—The difference between successive measured MSEP 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 13).

15.1.1.1 *Reference Fuels*—The repeatability of MSEP ratings for Jet A, Jet A-1, JP-5, JP-7, and JP-8 reference fuels is shown in Fig. 13 (Note 13).

NOTE 13—This repeatability is based on a 1983 eight laboratory cooperative test program using reference fluids prepared in accordance with Appendix X1.[12] This data was used because the MCell Coalescer[6] with the aluminum syringe has repeatedly demonstrated that the MSEP results for reference fluids are the same as those obtained by performing MSEP tests according to Test Method D3948 using the Alumicel Coalescer (TM) and plastic syringe. This was substantiated from the data obtained in the March 2002 program.[12]

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[11] Supporting data (report of the data and conclusions for the March 2002 eight laboratory, ten sample test program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1586.

[12] Supporting data (report of the data and conclusions for the 1983 eight laboratory cooperative program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1274.

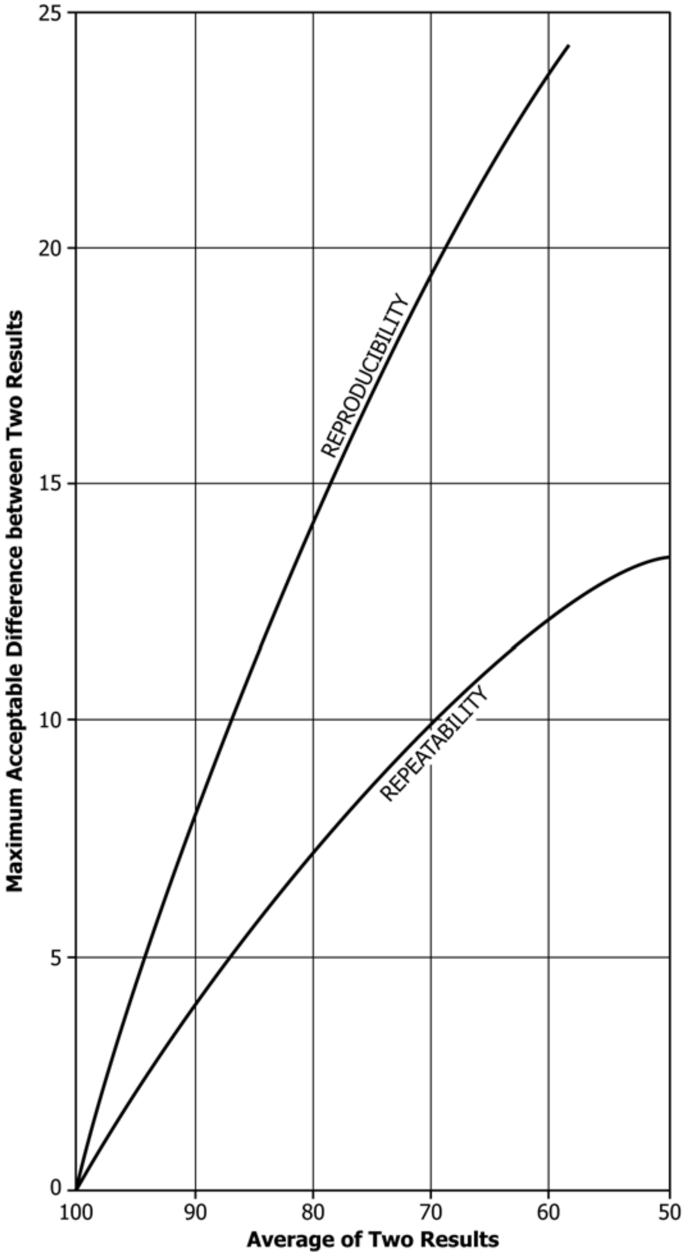


FIG. 13 Reference Fuels—Variation of Repeatability and Reproducibility of MSEP Ratings Obtained for Reference Fuels (Jet A, Jet A-1, JP-5, JP-7, and JP-8) Containing a Dispersing Agent

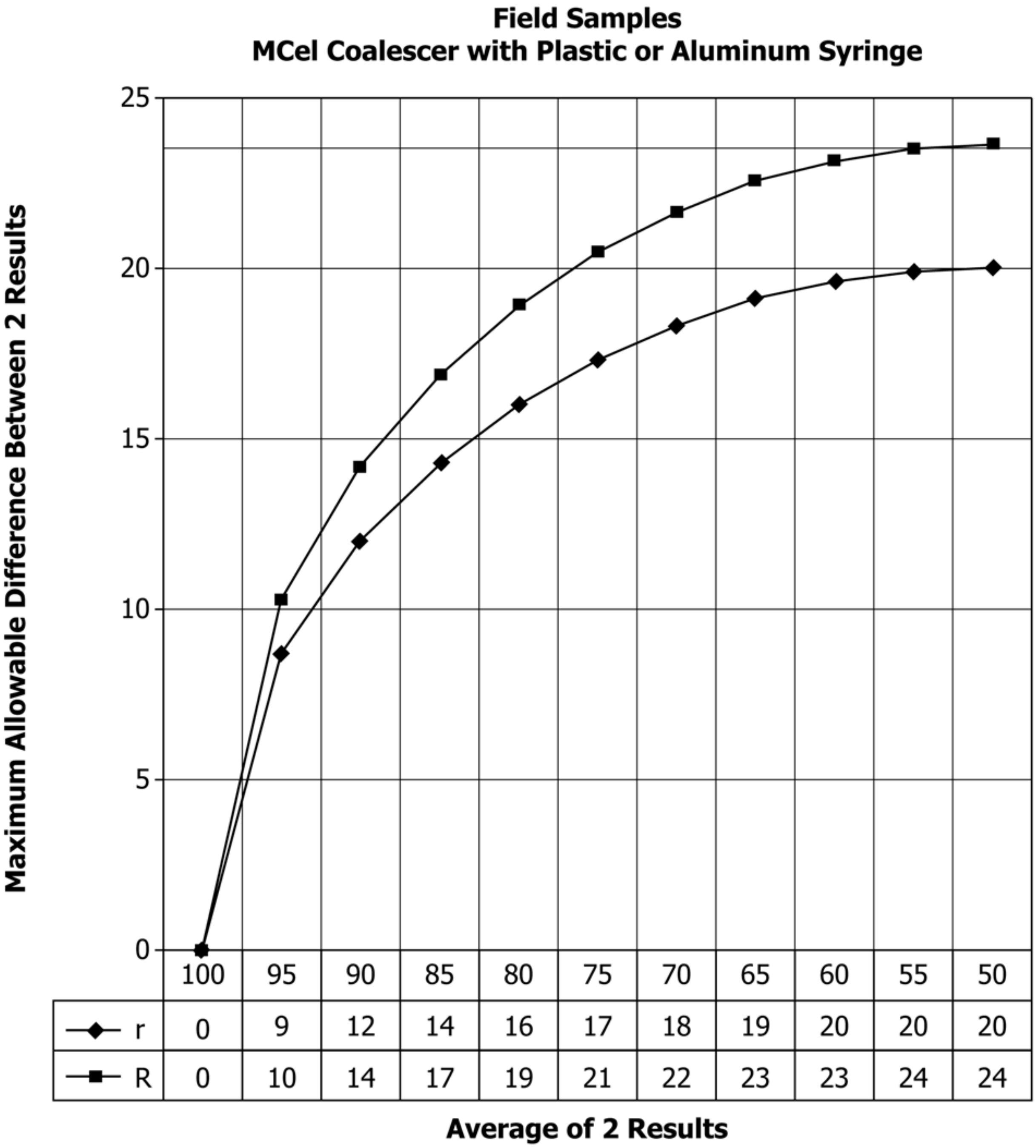
15.1.1.2 *Field Samples*—The repeatability of MSEP ratings for Jet A, Jet A-1, JP-5, JP-7, and JP-8 field samples, obtained by using test Mode A of the Mark V Deluxe instruments, is shown in Fig. 14 (Note 14).

NOTE 14—These results are based on a March 2002, eight laboratory cooperative test program[10] using ten samples, seven of which were field samples containing various concentrations and combinations of typical additives. 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.

15.1.2 *Reproducibility*—The difference between two single and independent measurements of MSEP 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 12).

15.1.2.1 *Reference Fuels*—The reproducibility of MSEP ratings for Jet A, Jet A-1, JP-5, JP- 7, and JP-8 reference fuels is shown in Fig. 13 (Note 13).

15.1.2.2 *Field Samples*—The reproducibility of MSEP ratings for Jet A, Jet A-1, JP-5, JP-7, and JP-8 field samples using this test method is shown in Fig. 14 (Note 14).



	100	95	90	85	80	75	70	65	60	55	50
◆ r	0	9	12	14	16	17	18	19	20	20	20
■ R	0	10	14	17	19	21	22	23	23	24	24

This precision is valid with both plastic and aluminum syringes when using the MCell Coalescer (see 3.3.1).

FIG. 14 Field Samples—Variation of Repeatability and Reproducibility of MSEP Ratings Obtained for Field Samples (Jet A, Jet A-1, JP-5, JP-7, and JP-8)

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

15.3 *Relative Bias*—There is no statistically significant relative bias between this test method and Test Method D3948 for clean fuels (with high MSEP results) and for fuels containing strong surfactants such as reference fuels (with low MSEP results). For fuels containing weak surfactants, such as approved additives like static dissipator additive and corrosion inhibitor, Test Method D3948 typically gives lower MSEP results relative to this test method.

16. Keywords

16.1 additives; aviation turbine fuel; coalescence; jet fuel; micro-separometer; 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 Specification D1655 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 JP-4, JP-5, JP-7, and 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 (see Table 2), 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 mm 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 mm to 10 mm with the legs 100 mm to 150 mm apart. The suction leg shall be 380 mm to 400 mm long to reach the bottom of the feed container. The other leg shall be 50 mm to 100 mm longer.

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 mm to 20 mm outlet.

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

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

X1.3.8 *Beaker,* 2 L capacity.

X1.4 Materials

X1.4.1 *Attapulgus Clay,* 30/60 mesh, LVM (calcined) grade or equal. Store the clay protected from atmospheric moisture and avoid handling that will cause particle size segregation.

X1.4.2 *Fine Glass Wool.*

X1.4.3 *Isopropyl Alcohol,* 90 %.

X1.4.4 *Toluene,* in a squeeze bottle. (**Warning—** Flammable. Vapor harmful.)

X1.4.5 *Water,* preferably distilled.

X1.4.6 *Salt (sodium chloride),* 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 of 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 mm to 15.0 mm (0.5 in. 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 X1.1—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 mL/min to 60 mL/min. Check by measuring with the small graduated cylinder for 1 min 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 X1.2—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 the 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 X1.3—At the specified flow rate, the 20 L (5 gal) percolation will be complete in 6 h to 6.5 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. 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*—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*—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 1 L (1 qt) of water (X1.4.5) thoroughly with 19 L (5 gal) of base fuel by agitating with 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 – X1.9.3, as required, to ensure 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 ensure testing of a representative sample. The round-robin precision study for this test method 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 appears in X2.2; 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 that 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 mm membrane filter) and allowed to stand for at least 24 h. The fuel should then be disposed of and the sample container flushed with the fuel to be tested prior to 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 mm to 12.7 mm (¼ in. to ½ 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 1 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.

SUMMARY OF CHANGES

Subcommittee D02.J0.50 has identified the location of selected changes to this standard since the last issue (D7224 – 13) that may impact the use of this standard. (Approved Dec. 1, 20143.)

(1) Added new subsection 13.2.1.1 and revised subsection 13.6.

Subcommittee D02.J0.50 has identified the location of selected changes to this standard since the last issue (D7224 – 12) that may impact the use of this standard. (Approved June 15, 2013.)

(1) Added Test Method D7261 to Referenced Documents.
(2) Added new subsection 3.1.
(3) Added DSEP to subsection 3.4.

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