**Designation: ~~D4860 – 21~~ D4860 – 22**

# Standard Test Method for Free Water and Particulate Contamination in Middle Distillate Fuels (Clear and Bright Numerical Rating)[1]

This standard is issued under the fixed designation D4860; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope*

1.1 This test method covers a rapid, portable means for field and laboratory use to inspect visually for particulate matter and numerically rate free water in aviation turbine and distillate fuels.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific warning statements, see 11.2.3 and Annex A1.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)
D2276 Test Method for Particulate Contaminant in Aviation Fuel by Line Sampling
D2709 Test Method for Water and Sediment in Middle Distillate Fuels by Centrifuge
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
D4176 Test Method for Free Water and Particulate Contamination in Distillate Fuels (Visual Inspection Procedures)
D6304 Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration

## 3. Terminology

3.1 *Definitions:*

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

3.1.2 *clear-and-bright* ~~(also termed~~(*clean-and-bright*)~~,*n*—~~*adj*—a condition in which the ~~fuel~~ liquid sample (fuel) contains no visible water drops or particulates and is free of haze or cloudiness.

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

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

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

3.1.3 *free water, n*—water in excess of that soluble in the liquid sample (fuel) at the temperature of the test and usually appearing in the liquid sample (fuel) as a haze (cloudiness), droplets, or water layer.

3.1.4 *solid particulate, n—in liquid fuels*, small solid or semi-solid particles, sometimes referred to as silt or sediment, present in fuel.

3.1.4.1 *Discussion—*

Some examples of solid particulates are air-blown dust, corrosion by-products, internal protective-coating deterioration, and products of fuel degradation and microbial growth.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *Micro-Separometer*[3]*clear-and-bright (MSEP–C&B*[3]*),n*—a numerical rating indicating the presence and ease of removal of free water and particulate contamination by filtration.

## 4. Summary of Test Method

4.1 Visual inspection of the fuel sample for free water and particulate matter is performed immediately when the sample is taken. A glass container is used to view for water haze, and the fuel sample is swirled to create a vortex to detect the presence of particulate matter.

4.2 A numerical rating for free water is obtained by filtering a portion of the fuel sample at a programmed rate (50 mL/45 s) through a standard fiberglass coalescer/filter. A portion of the effluent is used to establish a reference (100) level by a light transmittance measurement. Another portion of the unprocessed (unfiltered) fuel sample is then compared to the 100 reference level. The results are reported on a 50 to 100 scale to the nearest whole number. A test can be performed in 5 min to 10 min.

NOTE 1—The standard fiberglass coalescer/filter consists of a precision-machined aluminum housing containing fiber-glass material that has been selected to specific air flow characteristics. These criteria have a direct bearing on the test results.

## 5. Significance and Use

5.1 The test provides a field test to evaluate visually a fuel sample for particulate matter and free water similar to Test Method D4176 plus a numerical rating for free water. High numerical ratings indicate that the fuel is relatively free of free water. The degree of water and particulate contamination can be measured using other methods such as Test Methods D2276, D2709, and D6304.

NOTE 2—Clean and bright is sometimes used in place of clear and bright. The meaning is identical.

5.2 The color of the sample does not affect the measurement. Limited laboratory evaluations of samples have determined the degree of free water can be rated in fuels with dark opaque color having a darker rating than five in Test Method D1500.

## 6. Interferences

6.1 When a fuel is visually inspected at or below the cloud point temperature of the fuel, small amounts of solid wax particles can be confused with a water-induced haze or cloudiness.

6.2 The presence of free water or particulate can be obscured and missed during visual inspection of the fuel, if the ASTM color rating is greater than five.

## 7. Apparatus

7.1 *Micro-Separometer Instrument, Mark V Deluxe or Mark X:*[4]

[3] 'MSEP' and 'Micro-Separometer' are trademarks of EMCEE Electronics, Inc., 520 Cypress Ave., Venice, FL 34285.

[4] The sole source of supply of the apparatus (Model 1140 Micro-Separometer, Mark V Deluxe and Mark X) known to the committee at this time is EMCEE Electronics, Inc., 520 Cypress Ave., Venice, FL 34285 www.emcee-electronics.com. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

7.1.1 The Micro-Separometer is a completely portable and self contained unit capable of operating on an internal rechargeable battery pack (optional) or being connected to an ac power source using power cords that are furnished for various voltages. Connection to an ac power source provides power to the unit and effects battery recharge. The accessories as well as the expendable materials for six tests can be packed in the cover of the lockable case.

7.1.1.1 The Micro-Separometer Mark V Deluxe model and associated control panel are shown in Fig. 1.

7.1.1.2 The Micro-Separometer Mark X model and associated control panel are shown in Fig. 2.

NOTE 3—An extensive study was performed to verify that the Mark X Micro-Separometer gives equivalent results to the Mark V Deluxe Micro-Separometer. See Research Report RR:D02-1647.[5]




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

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


**FIG. 2 Mark X Micro-Separometer and Control Panel**

7.1.2 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.

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.

7.1.4 By depressing the *ON* push button, the electronic circuits are energized. The *ON* push button light pulses on and off when the instrument is being operated by an ac source and constantly remains on when the battery (dc) pack is used. The push buttons sequentially illuminate on and off indicating *ready* operational status.

NOTE 4—For the Mark V Deluxe—Of the lettered (*A*-*G*) push buttons, only the *C* push button is applicable to this test method.
For the Mark X—Only test mode Clear/Bright is applicable to this test method.



7.1.5 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 illuminate sequentially, thus indicating a *ready* operational status enabling test mode selection.

7.1.6 Test mode selection is accomplished by depressing the applicable push button—letter (*C*) on the Mark V and CLEAR/BRIGHT on the Mark X. The depressed push button illuminates and the sequential illumination of the other lettered push buttons ceases. The *START* push button on the Mark V and the *RUN* push button on the Mark X also illuminate and the syringe drive mechanism moves to the *UP* position.

7.1.7 The automatic program for the Clear/Bright test is initiated by depressing the *START* push button on the Mark V and the *RUN* push button on the Mark X.

7.1.8 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. A mark on the panel in front of the turbidimeter well is used to align the sample vial.

7.1.9 By depressing the appropriate arrow push button on the Mark V, the displayed value on the meter can be increased/decreased, as required, to establish the 100 reference level for the vial of filtered fuel sample in the turbidimeter. The 100 reference level is automatically set on the Mark X.

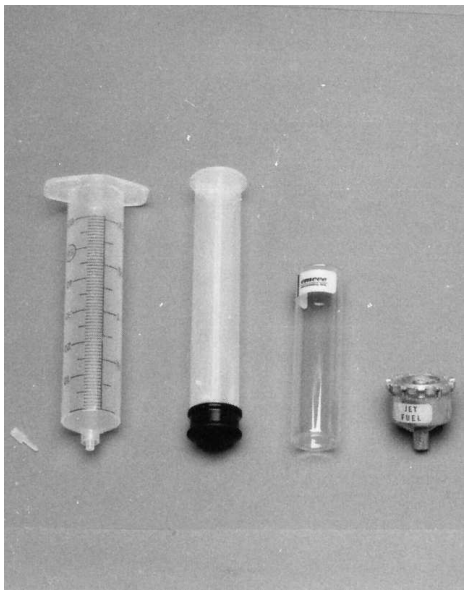
7.2 Accessory equipment and expendable materials needed to perform the test are shown in Fig. 3 and consist of the following:

7.2.1 *Syringe Plug (A)*—A plastic plug used to stopper the syringe.

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

7.2.3 *Vials (D)*—25 mm outside diameter vial premarked for proper alignment in the turbidimeter.

7.2.4 *Clear and Bright Coalescer/Filter (E)*[4]—An expendable, precalibrated aluminum coalescer/filter cell with a tapered end to fit the syringe. It is labeled in blue background with black lettering:



A B C D E

**FIG. 3 Test Supplies**

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7.2.5 *Beaker,* catch pan or the plastic container supplied with each Micro-Separometer can be used to receive the waste fuel during the coalescence period of the test (not shown).

7.3 *New Syringe, Syringe Plug, Test Sample Vial, and Clear and Bright Coalescer/Filter*[4] are used in each test. These expendable materials are available in a kit containing supplies for six tests. This kit is termed Micro-Separometer Clear and Bright Six Pack.[4]

7.4 *Sample Container,* cylindrical, wide-mouth, clear-glass, container capable of holding at least 900 mL of fuel. The minimum dimensions of the container shall be 100 mm in diameter with a height of 120 mm. The container shall have a lid to seal the contents.



7.5 *Temperature Measuring Device,* accurate to ±2 °C, to measure fuel temperature.

## 8. Sampling

8.1 Sampling shall be consistent with the procedures of Practice D4057. When practical, take the sample directly into the sample container; however, in some instances the sample may be transferred from the apparatus used to secure the sample to the sample container used in the test.

NOTE 5—Exercise care when transferring a sample from one container to another to ensure the test sample is representative of the fuel source.

8.2 Use the following procedure when the sample is drawn directly into the sample container from a sampling valve:

8.2.1 Be sure the sampling valve is free of loose solid contaminants. If rust or other loose encrustations are present, remove with a cloth; then flush the sampling valve before taking the actual sample.

8.2.2 Rinse a clean sample container thoroughly with the fuel being sampled.

8.2.3 Draw approximately 700 mL of fuel into the sample container (at least ¾ full) as rapidly as possible. Use a full flush rather than permitting the fuel sample to trickle out.

8.2.4 A lid shall be placed on the container to prevent water absorption or loss from the sample to the ambient environment, especially if the test is performed under different environmental conditions than those of the sample site or at a later time.

## 9. Preparation of Apparatus

9.1 Locate the Micro-Separometer on a clean workbench in an area in which the temperature is within the operating limits of the instrument, 0 °C to 50 °C.

9.2 Open the case and remove the six-pack box from the lid. Raise the right panel until completely vertical and locked in place. When ac power is available, connect the power cord and turn the instrument on; otherwise operate using battery power.

9.3 Depress the switch (push button) marked *ON*.

NOTE 6—Flickering of the power indicator light during any portion of a test sequence being performed when using battery power indicates that recharging is necessary.

9.4 Have ready a supply of syringes, syringe plugs, vials, and Clear and Bright coalescer/filters. In addition, have the catch pan positioned under the syringe drive mechanism to accept the spent fuel.

## 10. Conditioning

10.1 Under no circumstances is the test fuel to be prefiltered as filter media can remove the very materials, water and particulate matter, that the test is designed to detect.

10.2 The sample temperature shall not be lower than the temperature at which the fuel will be stored and used. Too low a storage temperature can cause a haze to form from water previously in solution. When possible, perform the test with the fuel sample at a temperature representing a real-use situation.



## 11. Procedure

11.1 Visual inspection for water or particulate contamination.

11.1.1 Immediately upon drawing a sample for field testing, check visually for evidence of water or particulate contamination. Hold the sample container up to the ambient light source and view the fuel through the walls of the container, visually examining for haze or lack of clarity. Check the same sample by swirling the fuel in the sample container to produce a vortex. The bottom of the vortex is visually examined for particulate matter. Record the visual clarity as *clear-and-bright* or *not clear-and-bright*. Record if particulate matter or water was or was not viewed at the bottom of the vortex. Measure the temperature of the fuel to ±2 °C and record. Cap the container.

11.2 *Numerical Rating of Free Water Using Micro-Separometer:*

11.2.1 Select the *clear-and-bright* test mode. The syringe drive mechanism travels to the *UP* position and the proper syringe drive speed is automatically selected.

11.2.2 *Prepare Fuel Sample:*

11.2.2.1 Remove the plunger (Fig. 3-C) 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 (Fig. 3-A) into the tapered bottom of the syringe barrel (Fig. 3-B). Add 50 mL ± 1 mL of fuel sample into the barrel of the syringe.

11.2.2.2 Partially insert the plunger to seal the open end of the syringe. Invert the syringe (exit hole up), remove the plug, and expel the entrapped air in the syringe barrel without significant fuel loss by carefully inserting the plunger to the 50 mL mark. (Use a clean wipe over the exit hole to capture the small amount of fuel which may be extruded as foam.) Affix a new Clear and Bright coalescer (Fig. 3-E) to the end of the syringe barrel.

11.2.3 *Prepare for Coalescing/Filter Process*—Place the entire syringe assembly into the syringe drive mechanism. Electrically bond the coalescer/filter to the instrument by using the ground lead provided. Insert the end with the banana plug into the receptacle located left of the syringe drive mechanism and attach the alligator clip to one of the coalescer/filter fins. Position a waste container beneath the coalescer/filter (see 7.2.4) to collect the unwanted portion of the processed fuel sample during the coalescing/filtering

period of the test (Fig. 4). (**Warning—**Clear and Bright coalescer/filters should be electrically bonded to the Micro-Separometer to prevent buildup of an electrostatic charge that could result in ignition of flammable test fluids (Fig. 4).)

11.2.4 *Initiate the Coalescing/Filtering Process:*

11.2.4.1 Depress the START push button which initiates the automatic program (see Table 1). The automatic program is a series of timed events controlled by the instrument that begins with the syringe drive mechanism forcing the fuel through the coalescer/filter.

11.2.4.2 Discard the first 35 mL of expended fuel and, using a new vial, collect the last 15 mL of fuel sample being processed from the coalescer/filter (Fig. 5). 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.

11.2.4.3 Wipe the outside of the vial with a clean, lintless wiper to remove fingerprints and fuel. Insert the vial into the turbidimeter well aligning the black mark on the vial at 90° from the white line on the front of the turbidimeter well. Rotate the vial until the black mark on the vial aligns with the white line on the front of the turbidimeter well.

11.2.5 Adjust the meter to the 100 reference level.

11.2.5.1 A short 4 s tone sounds followed by the meter activating for 10 s. During this period, the meter is adjusted to the 100 reference level by depressing the appropriate arrow push buttons. At the end of the 10 s period, the meter will deactivate for 30 s. Once the meter deactivates, further adjustment of the meter from this point on through the end of the test is not possible.

11.2.6 *Perform Measurement:*

11.2.6.1 Remove the vial from the turbidimeter well and discard the sample. Half fill the vial with fresh fuel sample that has not been processed (filtered).



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

11.2.7 *Read the MSEP–C&B Rating*—Following the 30 s *METER OFF* period that occurs after the coalescing/filtering process, a 4 s tone sounds and the *METER* automatically activates for a period of 10 s. Mark V Deluxe—During this period, read and record the value displayed on the meter. The Mark X instrument automatically measures the C & B Rating.



## 12. Report

12.1 Report the results obtained in 11.1.1 regarding the appearance and presence of particulate matter, the sample temperature, the value obtained in 11.2.7 (the MSEP–C&B rating), and reference this test method.

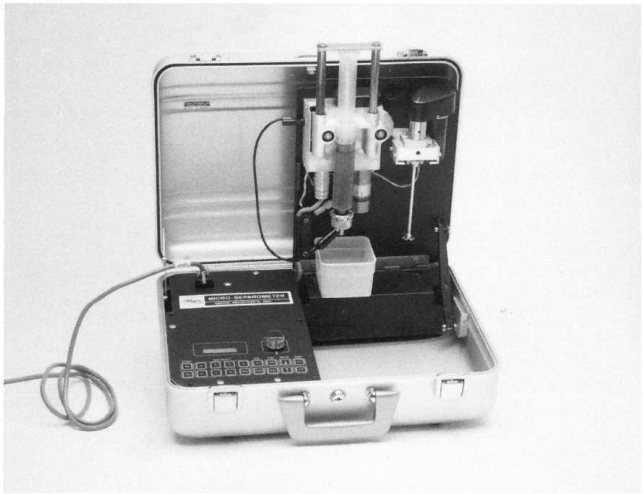

**FIG. 4 Syringe Assembly in Syringe Drive**