Designation: D 4860 – 91 (Reapproved 2000)ε1

An American National Standard

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

This standard is issued under the fixed designation D 4860; 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.

ε[1] NOTE—Warning notes were placed in the text editorially in November 2000.

1. Scope

1.1 This test method provides 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 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 hazard statements, see 11.2.3.1 and Annex A1.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)[2]

D 1744 Test Method for Water in Liquid Petroleum Products by Karl Fischer Reagent[2]

D 2276 Test Method for Particulate Contaminant in Aviation Fuel by Line Sampling[2]

D 2709 Test Method for Water and Sediment in Middle Distillate Fuels by Centrifuge[3]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[3]

D 4176 Test Method for Free Water and Particulate Contamination in Distillate Fuels (Visual Inspection Procedures)[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *free water*—water in excess of that soluble in the fuel at the temperature of the test and appearing in the fuel as a haze or cloudiness, or as droplets.

3.1.2 *solid particulates*—small solid or semi-solid particles, sometimes referred to as silt or sediment, present in fuel as a result of contamination by air-blown dusts, corrosion by-products, fuel instability, or protective-coating deterioration.

3.1.3 *clear-and-bright* (also termed *clean-and-bright*)— a condition in which the fuel contains no visible water drops or particulates and is free of haze or cloudiness.

3.1.4 *Micro-Separometer clear-and-bright (MSEP-C&B)*—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 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 D 4176 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

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

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[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 05.02.

D 1744, D 2276, and D 2709.

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 5 in Test Method D 1500.

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, Mark V Deluxe*[4]

7.1.1 The Micro-Separometer is a completely portable and self contained unit capable of operating on an internal rechargeable battery pack or being connected to an a-c power source using power cords that are furnished for various voltages. Connection to an a-c 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.

7.1.2 The Micro-Separometer Mark V Deluxe model and the associated control panel is shown in Fig. 1. The emulsifier is on the right side of the raised panel and the syringe drive

[4] The Micro-Separometer is available from EMCEE Electronics, Inc., 520 Cypress Ave., Venice, FL 34292.

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

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 a-c 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 a-c source and constantly remains on when the battery (d-c) pack is used. The A-G lettered push buttons sequentially illuminate on and off indicating *ready* operational status.

NOTE 2—Of the lettered (*A-G*) push buttons, only the *C* push button 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 lettered (*C*) push button. The depressed push button illuminates and the sequential illumination of the other lettered push buttons ceases. The *START* push button also illuminates and the syringe drive mechanism moves to the *UP* position.

7.1.7 The *START* push button, when depressed sequentially after depressing the *C* lettered push button, initiates the automatic program for the clear-and-bright test.

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 *ARROWED* push button, 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.

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

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

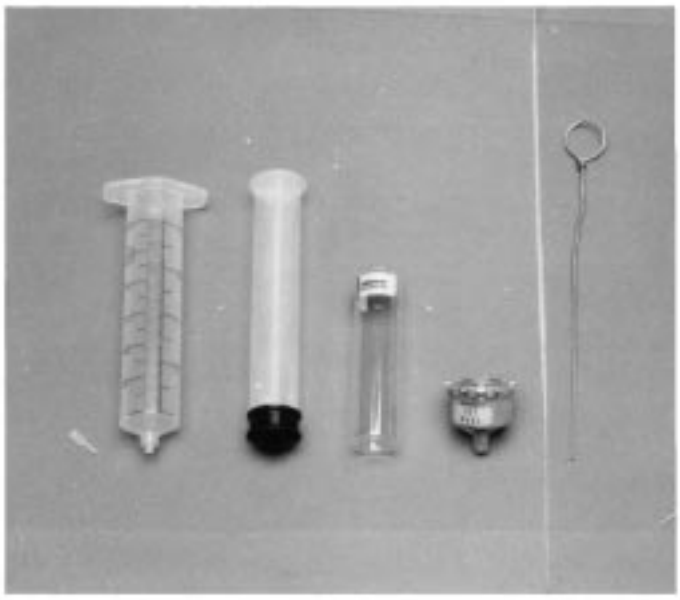
7.2.2 *Syringe, (Barrel, B* and *Plunger, C*), a disposable plastic syringe.

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

7.2.4 *Alumicel*[5] *Coalescer/Filter (E),* labeled *Clear and Bright*—an expendable, precalibrated aluminum coalescer/filter cell with a tapered end to fit the syringe.

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

7.2.6 *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).



A B C D E F

FIG. 2 Test Supplies and Accessory

7.3 *New Syringe, Syringe Plug, Test Sample Vial, and Alumicel*[5] *Coalescer/Filter* are used in each test. These expendable materials are available in a kit containing supplies for six tests. This kit, termed Micro-Separometer Clear and Bright Six Pack,[6] is designed to fit inside the top lid of the Micro-Separometer.

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.

8. Sampling

8.1 Sampling shall be consistent with the procedures of Practice D 4057. When practical, take the sample directly into the sample container; however, in some instances the sample can be transferred from the apparatus used to secure the sample to the sample container used in the test.

NOTE 3—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.

[5] A registered trademark of EMCEE Electronics, Inc.

[6] The Micro-Separometer Clear & Bright Six Pack is available from EMCEE Electronics, Inc., 520 Cypress Ave., Venice, FL 34292.

8.2.4 A lid must 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 to 50° (32 to 122°F).

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 a-c 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 4—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 Alumicel coalescer/filters. In addition, have the wire aid readily available and 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 temperature may 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. Cap the container.

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

11.2.1 Select the *clear-and-bright* test mode by depressing the push button marked *C* which illuminates. 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 a plunger from a new 50-mL syringe, insert a plug into the tapered bottom of the syringe, and add 50 ± 1 mL of fuel sample into the barrel of syringe.

11.2.2.2 Insert the plunger using the wire aid to release the entrapped air. Insert the wire aid in the syringe barrel with the loop end out. (To ensure that only the entrapped air is expelled without fuel loss, the syringe barrel should be cocked at a slight angle with the wire aid held against the upper portion of the barrel—Fig. 3.) Insert the plunger to the 50-mL mark on the syringe barrel. Remove the wire aid from the syringe. Remove the plug from the syringe and replace it with an Alumicel coalescer.

11.2.3 *Prepare for Coalescing/Filter Process*:

11.2.3.1 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 recepticle 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 Alumicel coalescer/filter to collect the unwanted portion of the processed fuel sample during the coalescing/filtering period of the test (Fig. 4).(**Warning**—Alumicel 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.))

NOTE 5—**Caution:**

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 Alumicel coalescer/filter.

11.2.4.2 Disregard the first 5 mL of expended fuel and, using a new vial, collect the next 15 mL of fuel sample being processed from the Alumicel 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. 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.

11.2.5 Adjust the meter to the 100 reference level.

FIG. 3 Plunger Insertion

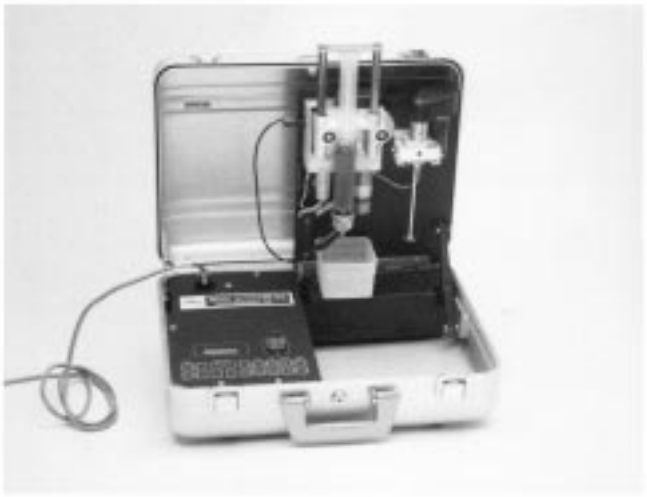


FIG. 4 Syringe Assembly in Syringe Drive

TABLE 1 Automatic Program

Operator	*Micro-Separometer*
Depress *START* push button	Syringe drive starts down (total start/stop time 45 ± 2 s)
No activity	5 mL of filtered fuel expended
Collect next 15 mL of fuel	Syringe drive continues down
Place vial in turbidimeter	Syringe drive stops
No activity	Pulsed tone for 4 s
Adjust meter to 100	Meter activates for 10 s
No activity	Meter deactivates
Discard fuel and retain vial	No activity for 30 s
Half fill vial with unfiltered fuel	No activity
Place vial in turbidimeter	No activity
No activity	Steady tone for 4 s
Read and record MSEP C&B	Meter activates for 10 s
No activity	Meter deactivates
Discard vial with fuel	End of test sequence

FIG. 5 Taking Sample

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 *ARROWED* 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*:

11.2.7.1 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. During this period, read and record the value displayed on the meter.

12. Report

12.1 Report the results obtained in 11.1.1 regarding the appearance and presence of particulate matter and the value obtained in 11.2.7.1 as the *MSEP-C&B* rating.

13. Precision and Bias [7]

13.1 *Visual Examination for Free Water and Particulate Matter*—It is not practical to specify the precision, and no justifiable statement can be made on the bias of the procedure in this test method since this is a pass/fail test based on a subjective visual examination.

13.2 *Precision*—Numerical rating of free water, as determined by statistical analysis of the test results obtained by twelve operator/instrument pairs on identical fuel samples at a common site is as follows:

13.2.1 *Repeatability*— The difference between successive measured MSEP–C&B ratings obtained by the same operator with the same Micro-Separometer under constant operating conditions on identical material would, in the long run and in the normal and correct operation of the test method, exceed six for only one case in twenty (Note 6).

13.2.2 *Reproducibility*—The difference between two single and independent measurements of MSEP–C&B ratings by different operator/instrument pairs at the same location on identical material would, in the long run and in the normal and correct operation of the test method, exceed seven for only one case in twenty (Note 6).

NOTE 6—These results are based on a March 1989 twelve-laboratory cooperative test program using samples specifically prepared for the program. Samples were specifically prepared, since representative samples covering a wide range of level of contaminates were unobtainable from field sources. The repeatability and reproducibility values were estimated from results obtained at the same location and on two consecutive days, by different operator/instument 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 as a result of sampling, environmental factors, and instability of the contaminates present.

13.2.2.1 In practice, two results obtained at different laboratories (locations) would be acceptable, if their difference did

[7] Supporting data may be obtained from ASTM Headquarters. Request RR D02-1268.

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. As a practical matter, testing for free water and particulate matter is a real time test, since both contaminates can vary according to changes in the environment, particularly temperature, and time (settling effects).

13.3 *Bias*—The procedure in Test Method D 4860 has no bias, because the value of MSEP-C&B ratings is defined only in terms of this test method.

14. Keywords

14.1 clear and bright numerical rating; free water; Micro-Separometer; mid-dillate fuels; MSEP-C&B rating; particulate contamination

ANNEX

(Mandatory Information)

A1. PRECAUTIONARY STATEMENT

A1.1 Flammable Liquid (general)

Keep away from heat, sparks, and open flame.
Keep container closed.
Use only with adequate ventilation.
Avoid prolonged breathing of vapor or spray mist.
Avoid prolonged or repeated contact with skin.
Spillage and fire instructions will depend on nature of liquid.