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Coarse Droplet Water/Fuel Separation Test Procedure

RATIONALE

SAE J1839 was revised to meet the five-year review requirements and to include improvements to the test method as defined by the SAE Filter Test Method Committee (FTMC).

FOREWORD

Water in fuels is one of the major causes of diesel engine maintenance problems. The effects of water in fuel are characterized by corrosion of fuel system parts, plugging of filters and orifices and, in some cases, failure of fuel injection equipment. Water in fuel often dissolves sulfur compounds, becomes acidic, and enhances corrosion in fuel injection systems as well as in the engine itself. The presence of water also encourages microbiological growth, which generates orifice and filter restricting sludge. Further, due to displacement of fuel lubrication in close tolerance injector parts, and rapid expansion of heated water at the fuel injector tip, galling, and more serious failure may also occur.

During transportation, transfer, and storage of fuel, water may become entrained in a variety of ways. The mode and timing of water entry in the handling sequence before use, as well as the chemistry of the fuel itself (additives and surfactants), will determine what form the contaminant takes. In systems where the water and fuel pass through high shear pumps, fuel water interfacial tension is relatively low and settling time is minimized, fine emulsions may predominate. In systems where water enters before or after low shear pumps, or where there is a prolonged settling time in high interfacial tension fuel, larger water droplets may predominate. In some systems, both fine emulsions and large droplets may be present simultaneously. Generally, fine emulsions are more likely to predominate on the pressure side of high shear pumps, whereas larger water droplets are more likely to predominate on the suction side of pumps. (A water removal test procedure designed for applications where finely dispersed droplets predominate is also recommended. This procedure is given in SAE J1488.)

The following test procedure is relevant to coarsely dispersed water separation devices whether applied on the suction or discharge side of engine fuel transfer pumps. (This procedure recommends pressure side location of the test unit for ease and convenience of testing only. Modification of the procedure to place the test unit on the suction side of the pump should not alter test results as long as the water droplet size distribution remains unaltered.) The procedure is well suited to lower flow rates, although it may be applied with due caution to flow rates up to 100 Lpm. It has been designed to approximate field conditions in a practical manner. A water dispersing technique simulating the water droplet sizes experienced drawing fuel/water mixtures through fuel lines and fittings (180 to 260 mm mean droplet size), is used to reproduce field conditions where coarse droplets predominate. The test fuel may be an actual fuel sample (with additives) that is to be used in the field, or it may be No. 2 fuel oil that has been clay treated (conditioned) so as to enable equal and reproducible laboratory comparisons of various test devices. Additionally a fluid simulating a representative biodiesel is recommended as biodiesel has been established as being particularly problematical for controlled water separation and water separation testing. Test fuel conditioning is recommended for laboratory comparisons only, as this treatment may yield water removal efficiency results, which are significantly different from those obtained using water separating devices in untreated or biodiesel fuel. Furthermore, testing unused "clean" water separators may provide water removal efficiencies that are far superior to those obtained from the same water separators after very short exposure to natural fuel and natural fuel contaminants.

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1. SCOPE

To determine the undissolved water removal performance of a fuel/water separator under controlled laboratory conditions, using water droplets. This test method is applicable for diesel and biodiesel fuel.

2. REFERENCES

2.1 Applicable Documents

The following publications form a part of this specification to the extent specified herein. Unless otherwise indicated, the latest issue of SAE publications shall apply.

2.1.1 SAE Publication

Available from SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001, Tel: 877-606-7323 (inside USA and Canada) or 724-776-4970 (outside USA), <u>www.sae.org</u>.

SAE J1488 Emulsified Water/Fuel Separation Test Procedure

2.1.2 ASTM Publications

Available from ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959, Tel: 610-832-9585, <u>www.astm.org</u>.

- ASTM D 971 Test Method for Interfacial Tension of Oil Against Water by the Ring Method
- ASTM D 7261-08 Standard Test Method for Determining Water Separation Characteristics of Diesel Fuels by Portable Separometer
- ASTM D 4176 Test Method for Free Water and Particulate Contamination in Distillate Fuels (Visual Inspection Procedure)
- ASTM D 6304-00 Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration
- 2.1.3 ISO Publication

Available from American National Standards Institute, 25 West 43rd Street, New York, NY 10036-8002, Tel: 212-642-4900, <u>www.ansi.org</u>.

ISO R 760 Determination of water—Karl Fischer method (general method)

3. TEST APPARATUS

A test system, as illustrated in Figure 1, is to include:

- 3.1 Operating sump with a flat bottom. Material to be compatible with diesel fuel (polyethylene, etc.). Sump size to be adequate to hold test fluid volume under test conditions. Outlet to be 2.5 cm from bottom of tank or higher.
- 3.2 A pump, capable of providing test flow rate, under test conditions.
- 3.3 A water dispersing device, as described in Appendix A.
- 3.4 Thermometer or temperature readout accurate to ±2 °C under test conditions.
- 3.5 The fuel/water separator under test.

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- 3.6 Differential pressure gauge or manometer with 1.0 mm Hg or 0.1 kPa subdivisions, or as required.
- 3.7 In-line static mixer with at least three internal mixing units, or similar, to provide a representative sample at the sample port.
- 3.8 Sample port. Provision must be made to allow adequate flushing, immediately prior to sampling.
- 3.9 Test fuel flow meter, flow range appropriate for the unit under test, accuracy ±5%, repeatability ±2%.
- 3.10 A final fuel/water separator assembly, such that not more than 30 ppm undissolved water is recycled on an average basis under test conditions.
- 3.11 Suitable heat exchange and controls for maintaining constant test temperatures as specified.
- 3.12 Suitable valve or other control for adjusting and controlling test flow.
- 3.13 Diffuser, to promote uniform mixing in test reservoir.
- 3.14 Water flow meter with range to suit application capable of measuring flows of 0.25% of fuel flow rate, ±5% accuracy.



- 3.1 Operating Sump
- 3.2 Pump
- 3.3 Water Dispensing Device, Appendix A
- 3.4 Thermometer
- 3.5 Test Fuel Filter/Water Separator
- 3.6 Differential Pressure Gauge
- 3.7 Inline Mixer
- 3.8 Sample Port
- 3.9 Fuel Flow Meter
- 3.10 Final Fuel Filter/
- Water Separator
- 3.11 Heat Exchanger
 - **FIGURE 1 TEST SYSTEM**

- 3.12 Flow Control Valve
- 3.13 Diffuser
- 3.14 Water Flow Meter
- 3.15 Water Flow Control Valve
- 3.16 Water Filter/Deionizer
- 3.17 High Pressure Water Supply

- 3.15 Suitable valve for adjusting and controlling water injection flow.
- 3.16 A supply of clean deionized or distilled water with a surface tension greater than 70 mN/m ± 2 mN/m at 20 °C ± 1.5 °C. Adequate pressure must be available to inject water on the high pressure side of the pump. Use of a deionizing filter will permit use of local water supply and pressure.
- 3.17 Automatic Karl Fischer Titration Apparatus for water content analysis.
- 3.18 All interconnecting piping should be selected, sized, and oriented to prevent the separation of water and solid contaminants from the fuel or provide traps for these contaminants (13 mm PVC is recommended for 0 to 23L/m flow rates).

4. TEST MATERIALS

4.1 Test Fluid

Since fuel oil contains various constituents, the test oil type should be categorized and recorded as one of the following:

- a. A sample of the fluid used in the application. As defined by user, to include biodiesel fuels
- b. Specially treated fluid, per Appendix B

In all these cases, it should be understood that the results are relevant to this fuel and that some amount of variance in performance can be expected with different fuels, depending on the particular design of the test fuel/water separator and the chemical nature of the particular lot of fuel.

The IFT of the test fluid must be between 15 and 19 mN/m.

NOTE: Microseparometer (ASTM D 7261-08) readings lower than 75 may indicate a fuel's poor water separation ability.

- 4.2 Distilled or deionized water with a surface tension of 70 mN/m \pm 2 mN/m at 20 °C \pm 1.5 °C.
- 5. TEST CONDITIONS
- 5.1 Volume of Fuel in the Test System

Five times the flow rate, per minute, with a minimum of 38 L (including filters, piping, etc.)

5.2 Temperature

26.6 °C ± 2.5 °C measured at the test separator inlet, or at a temperature agreed upon by the supplier and user.

5.3 Pressure

Pressure or vacuum operating parameters are set as required by the manufacturer, user, or application, otherwise they are controlled by the fuel flow rate.

5.4 Flow Rate of Fuel

Rated flow of unit to be tested or as specified.

5.5 Water Flow Rate

0.25% of fuel flow rate.

6. TEST PROCEDURES

- 6.1 Clay-treated fuel (Appendix B) and non clay-treated fuel may be used more than once for consecutive tests with or without re-treating, as long as the fuel meets the requirements of 4.1.
- 6.2 Determine the water saturation level for each batch of test fluid:
- a. According to Appendix C
- b. By using tables or charts where available; see Appendix D
- 6.3 Prior to filling the fuel tank, determine whether the test fluid has a reasonable contamination level and water concentration (indicated by a "pass" or "clear and bright" result in procedure 1 of ASTM D 4176, visual inspection procedure).
- 6.4 Install test fuel/water separator or filter on the discharge side of the pump (see Figure 1) before circulating the appropriate test fluid. Adjust the fuel flow rate by throttling the discharge side valve. System should circulate for at least 2 turnovers to adequately coat test filter media. Take initial (fuel only) pressure drop reading at the rated flow. To determine 1 turnover:

$$\frac{SystemVolume(L)}{FlowRate(L/\min)} = 1turnover(\min)$$
(Eq. 1)

- 6.5 Open the water valve and adjust water flow rate to 0.25% of fuel flow. Start the clock at the same time water begins to flow. Establish proper flow rate within 1 min.
- 6.6 Without interrupting test flows periodically drain the water from the water collection sump of the unit under test and wastewater sumps into a volumetric container. It may be necessary to bring the system under positive pressure to drain under suction applications. The amounts of water collected from the test can be used to obtain a water balance verification efficiency (see Appendix E). Manually draining may not be necessary if an automatic water-sensing switch/drain is available. Do not let water build up beyond the maximum recommended level of the water sump. DO NOT TAKE ANY SAMPLES WHEN ASSEMBLY IS BEING DRAINED.
- 6.7 Record test time for each drain.
- 6.8 At approximately 10 min, carefully withdraw a sample, being sure to flush the sample port thoroughly. Record the time of the sample. Be sure that the sample syringe or container is thoroughly dry. Analyze the sample immediately using the appropriate method (see ISO R 760 or ASTM D 6304). Record each reading. Additional sampling is permitted. Repeat this sampling procedure every 20 min thereafter until termination of the test.
- NOTE: For low-efficiency filters, it may be necessary to obtain larger effluent samples and disperse coarse water droplets by using an ultra-sonicating bath.
- 6.9 Record the differential pressure across the test fuel/water separator at each effluent sample interval.
- 6.10 Terminate the test if one or more of the following conditions is met:
- a. Water concentration in effluent fuel is above an acceptable level, to be specified by manufacturer or user, or
- b. An equilibrium pressure drop has been reached and a minimum of 30 min or more of test time has elapsed and an equilibrium pressure drop is attained. Equilibrium pressure drop has been reached when an increase of no more than 2.5 mm Hg occurs during a 20 min interval
- c. Differential pressure exceeds an upper limit specified by the manufacturer or user for the element or application.

6.11 Upon completion of test, determine the IFT and DSEP values and record.

6.12 Take a final effluent sample for analysis at test termination

7. PRESENTATION OF DATA

- 7.1 Plot concentration of undissolved water in effluent versus time (minutes) on linear graph paper. Undissolved water = total water minus dissolved water (see 6.2). Indicate drain times on graph.
 - NOTE: Many Karl Fischer titration devices will determine the water concentration in terms of micrograms, or ppm by weight. To convert micrograms or ppmw to parts per million (ppm) by volume, use the following equation:

 $Titration Reading(in ppm (volume fraction)) = \frac{Titration Reading (in ppm (mass fraction)) * Fuel Density (g/l)}{Water Density (g/l)}$ (Eq. 2)

- 7.2 Plot of pressure drop (mm Hg or kPa) versus time (minutes) is optional. If plot is not presented, total pressure drop should be reported.
- 7.3 Record the following:
- 7.3.1 IFT and DSEP
- 7.3.2 Fuel flow rate
- 7.3.3 Actual test temperature.
- 7.3.4 Total test time.
- 7.3.5 Equilibrium pressure drop.
- 7.3.6 Dissolved water saturation level, method used to determine level.
- 7.4 Calculate and report the average free water content of effluent:

$$Cav = \sum_{i=1}^{n} ci (ti - t(i-1)/ttotal)$$
(Eq. 2)

where:

 c_i is the effluent water concentration, ppm, at time ti, min i is an integer from 1 to n, where n equals the number of effluent samples taken t_{total} is the total test time, min

7.5 Calculate and report average undissolved water separation efficiency by using Equation 3:

Average Efficiency =
$$\left(1 - \frac{Cav}{2500}\right) * 100\%$$
 (Eq. 3)

where:

c av = Average undissolved water content of effluent

8. NOTES

8.1 Marginal Indicia

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PREPARED BY THE SAE FILTER TEST METHODS STANDARDS COMMITTEE





(1) Fuel velocity past nozzle tip should be between 0.75 and 1.50 m/s.

Water velocity through nozzle should be between 4.7. and 7.0 m/s.

where L/M = Test Flow Rate

- D = Maximum diameter (mm) = $20\sqrt{LPM \times .07074}$ for 0.75 m/s velocity
- D = Minimum diameter (mm) = $20\sqrt{LPM} \times .03536$ for 1.50 m/s velocity
- d = Maximum diameter (mm) = $20\sqrt{LPM} \times .0000282$ for 4.7 m/s velocity
- d = Minimum diameter (mm) = $20\sqrt{LPM \times .0000189}$ for 7.0 m/s velocity

(See Figures A2 and A3 for nozzle I.D. and gauge at various test flow rates.)

This water dispersing method has been found to produce dispersed phase droplet populations with mean diameters of approximately 180 to 260 µm.

FIGURE A1 - WATER DISPERSING DEVICE



FIGURE A2 - DISPERSING NOZZLE SIZE AND GAUGE FOR 0 TO 70 L/M TEST FLOW AT 4.7 TO 7.0 M/S VELOCITY THROUGH NOZZLE (ASSUMES 0.25% WATER ADD RATE)



FIGURE A3 - DISPERSING NOZZLE SIZE AND GAUGE FOR 0 TO 15 L/M TEST FLOW AT 4.7 TO 7.0 M/S VELOCITY THROUGH NOZZLE (ASSUMES 0.25% WATER ADD RATE)

APPENDIX B

- B.1 FUEL TREATMENT TO OBTAIN FLUID AS SPECIFIED IN 4.1.
- B.1.1 Take the required volume of fuel oil and continuously contact the fuel with Fuller's Earth. This may be done by filtering the fuel through commercially available Fuller's Earth or clay cartridge filters. The test fluid sump should be used.
- B.1.2 Periodically [about every 20 min or 2 turnovers (see 6.3)], take a sample of the fuel in a beaker and measure the interfacial tension (IFT) with distilled or deionized water at 20 °C ± 5 °C. The platinum ring detachment method (ASTM D 971) is recommended, although other correlatable methods may be used.
- B.1.3 If the IFT is above 20 mN/m, stop further contacting the Fuller's Earth or clay. Generally 2 to 4 turnovers will more than adequately ensure that this condition is met. Report the IFT of the treated fuel.
- B.1.4 Remove the Fuller's Earth filters from the test loop, or adjust valving to isolate them from the test loop.
- B.1.5 Add to the Fuller's Earth treated fuel (in the test sump) additive monoolein in parts per million. Monoolein is a pure compound composed of monoglycerides; it can be added to the test fuel to simulate biodiesel or ultra low sulfur diesel fuel with low interfacial tension. At room temperature, monoolein is a solid, and should be heated to convert it to a liquid. Add liquid monoolein to fuel (in the test sump) in a volume percent depending on the target IFT between 15 and 19 mN/m (or per client's specification). The modification of fuel/water interfacial tension and DSEP may have significant influences on test results. NOTE: It is advisable to check the interfacial tension of this treated fuel (with monoolein) against distilled or deionized water (ASTM D 971). Figures B1 and B2 are graphical representations of IFT, and DSEP values of one diesel fuel containing monoolein in different concentrations. It must be noted, Figures B1 and B2 are representation of one diesel fuel. The amount of monoolein required to achieve the target IFT will be dependent on the chemical nature of the particular type diesel fuel.



FIGURE B1 - INTERFACIAL TENSION VS. MONOOLEIN, PPM



FIGURE B2 - DSEP VS. MONOOLEIN, PPM

B.1.6 Circulate the fuel with additive through the pump until there has been at least two complete turnovers of the fuel volume in the sump. The fuel treatment is now complete.

APPENDIX C

C.1 METHOD OF DETERMINING SATURATION LEVEL OF DISSOLVED WATER IN FUEL

Use this method for maximum accuracy or when tables or charts are unavailable. This method should be performed once for each new batch of fuel.

- C.1.1 Wash a clean sample bottle (with a rubber diaphragm cap; minimum 100 mL capacity) with distilled water so as to remove traces of detergent; dry bottle thoroughly.
- C.1.2 Place 75 mL of the filtered fuel into the dried clean 100 mL sample bottle. Insert a clean PTFE coated magnetic stirrer.
- C.1.3 Fill a 50-mL hypodermic syringe fitted with a long, large diameter needle with clean distilled or deionized water and remove any air. Carefully insert 25 mL of the water into the bottom of the sample bottle taking care to create and maintain the water/fuel interface with minimal agitation. Completely fill the bottle with fuel. Place the bottle on a magnetic stirrer and stir at the lowest possible speed. Make sure that the water/fuel interface is not strongly agitated and that no appreciable vortex develops as a result of mixing.
- C.1.4 After mixing for approximately 18 h (overnight), place the bottle gently in a water bath at the test temperature (26.6 °C) for 2 h; insert a clean, dried, hypodermic syringe through the rubber diaphragm in the cap; gently withdraw 2 mL of fuel from the top and analyze for water content using the Karl Fischer method (ASTM D 6304). Take three readings. The average is the water saturation.
- C.1.5 Report this dissolved water saturation level in the fuel as ppm by volume.
- NOTE: Typical fuel saturation levels are between 100 to 150 ppm.



FIGURE D1 - SOLUBILITY OF WATER IN NO. 2 FUEL OIL

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APPENDIX E - METHOD FOR VERIFYING WATER REMOVAL EFFICIENCY

- E.1 This method can be used to verify the total water removed at the end of a test. This value is not to be reported, but used only to verify water titration analysis.
- E.1.1 Obtain volumetric reading of water collected from the test filter (V1) and downstream clean-up filters (V2).
- E.1.2 Calculate the total water fed by adding the volumetric reading from the test filter (V1) and clean-up filters (V2). Compare this amount to the theoretical amount of water added (see section 6.5).
- E.1.3 Calculate the water balance verification efficiency (see Equation E1). This value should be compared to the time-weighted average efficiency calculated in Equation 3.

Water Balance Verification Efficiency (%) = (V1/(V1+V2)) * 100 (Eq. E1)