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Emulsified Water/Fuel Separation Test Procedure

RATIONALE

SAE J1488 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 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 large water droplets predominate is also recommended. This procedure is given in SAE J1839.)

The following test procedure is relevant to finely dispersed or emulsified 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. The procedure is well suited to lower flow rates, although it may be applied with due caution to flow rates up to 100 Lpm using parallel emulsion generating circuits. It has been designed to approximate field conditions in a practical manner. A 3500-rpm centrifugal pump is used to disperse water in the fuel, simulating most fuel loading pumps. 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 fuel or biodiesel. 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 ability of a fuel/water separator to separate emulsified or finely dispersed water from fuels. This test method is applicable for 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), www.sae.org.

SAE J1839	Coarse Droplet 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, www.astm.org.

ASTM D 971	Test Method for Interfacial Tension of Oil Against Water by the Ring Method
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 replace with ISO 12937
ASTM D 7261-08	Standard Test Method for Determining Water Separation Characteristics of Diesel Fuel by Portable Separometer

3. TEST APPARATUS

A test system, as illustrated in Figure 1. This system generates test emulsion populations in ultra low sulfur diesel fuel (IFT 15-19 mN/m). Test droplet size distribution in this procedure is controlled for changes in test flow rate at recommended interfacial tensions, but may vary significantly in test fuels where interfacial tension is not controlled.

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

3.1 A flat bottom, corrosion resistant fuel container with a fuel outlet not less than 4 cm from the bottom of the container.

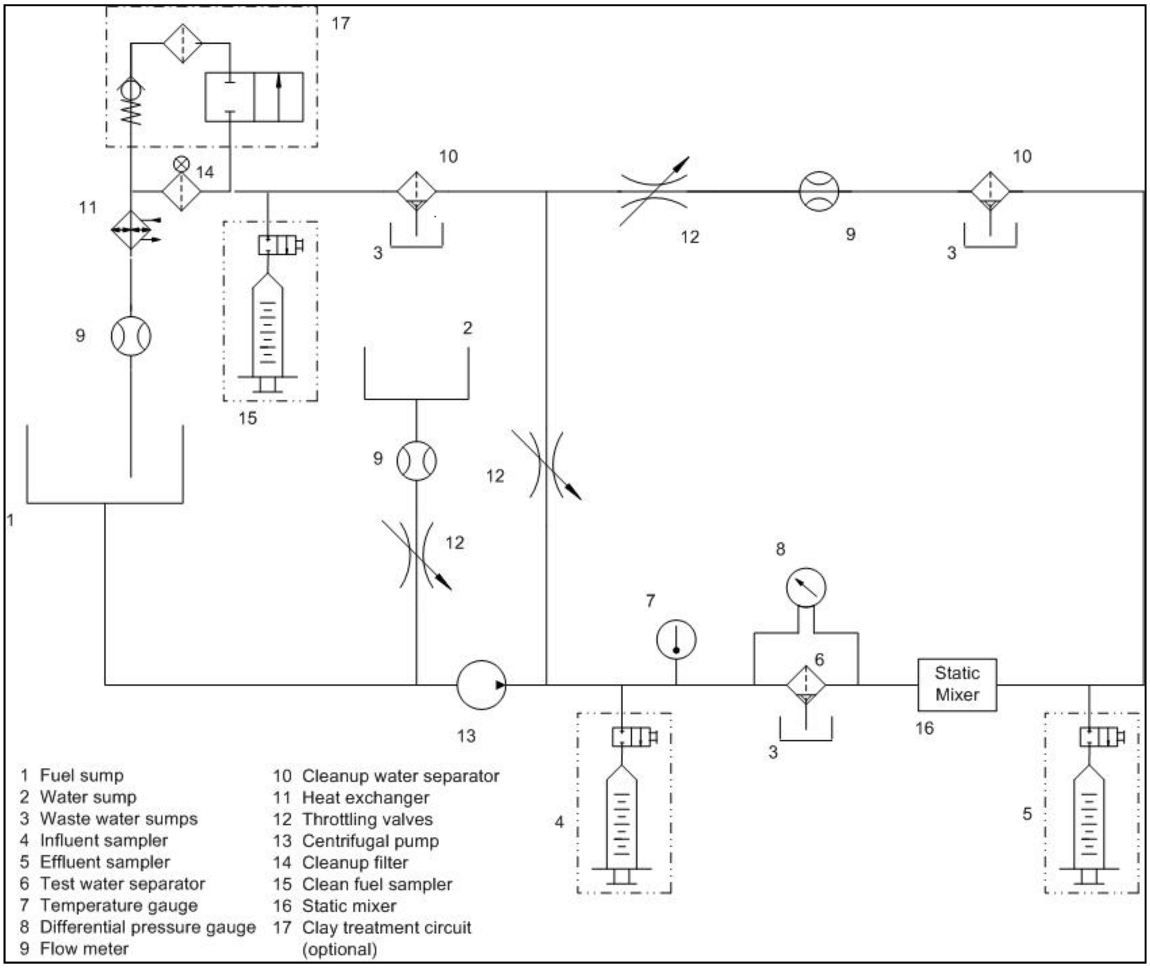

FIGURE 1 - TEST SYSTEM

3.2 A 3500 rpm ± 100 rpm, 1 Hp centrifugal pump, ITT Gould model 1ST 1E5D4.

3.3 Fuel flow meter capable of measuring with an accuracy of better than or equal to 5% of actual flow.

3.4 Temperature indicator with an accuracy of ±1.5 °C.

3.5 A cleanup water separator assembly such that not more than 50 ppm of undissolved water is recycled on an average basis under test conditions.

3.6 A corrosion resistant water sump with approximate capacity of 19 L.

3.7 Water flow meter (0 to 100 mL/min, or as required) with flow regulating valve, capable of measuring water at 0.25% of test flow rate, with an accuracy of 5% of actual flow.

3.8 Automatic Karl Fischer Coulemetric Titration Apparatus for water content analysis.

3.9 Leave static mixer in system. Full flow needs to go through this “mixer”. Minimum of 50 ml sample size. Sonicate in a sealed container before Karl Fischer analysis.

3.10 The fuel/water separation stand piping should have a velocity equal to or greater than 0.72 m/s. Any nonrusting and nonreacting pipe material may be used.

3.11 A differential pressure gauge with a readability of 340 Pa and an accuracy of 340 Pa.

3.12 Syringe sampler in accordance with Figure 2. Note that the influent sampler does not have an in-line mixer while the effluent sampler does, this is because the water at the influent section is emulsified and an in-line mixer at this point is not needed. The syringe sampler may be replaced with a beaker or vile to collect the sample. The sample should be sealed and sonicated before Karl Fischer analysis.

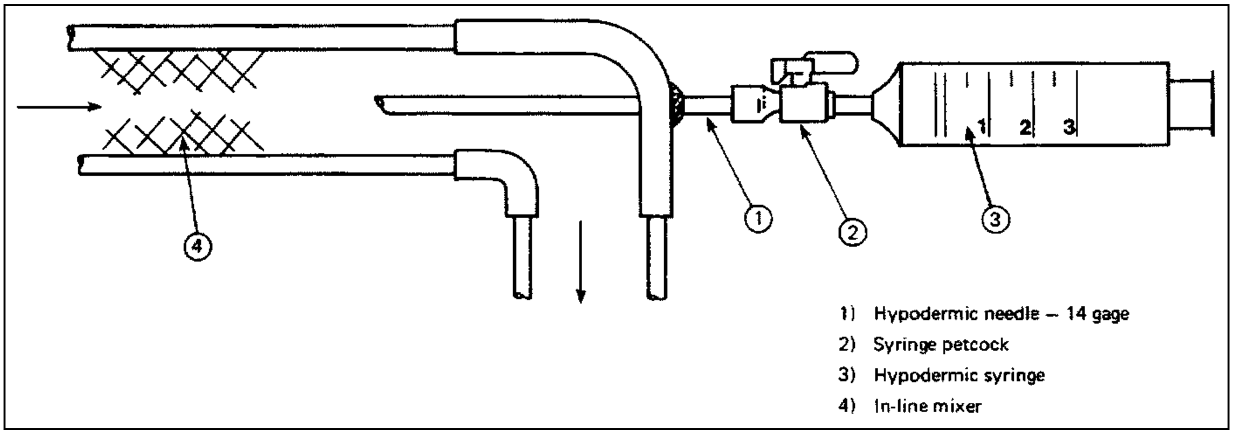

FIGURE 2 - EFFLUENT SAMPLER

3.13 Temperature control system capable of maintaining test temperature as specified in 5.2.

3.14 Double pipe heat exchanger (cooling and heating, if required) with fuel in the tube side. This should be a single pass, single tube, double pipe exchanger. If adequate cooling can be accomplished after the cleanup fuel/water separator (see Figure 1), then any type of heat exchanger may be used.

3.15 Fuel/water interfacial tension measuring device. Preferably a platinum ring detachment method (ASTM D 971) should be used.

3.16 For testing at flow rates greater than 25 Lpm it is required that separate emulsion generating circuit(s) with a common sump be connected in parallel to generate the emulsions for the test circuit.

4. TEST MATERIALS

4.1 Test Fluid

Since commercially available diesel fuel oil (particularly biodiesel) contains highly variable constituents, that can significantly influence the outcome of emulsion removal testing, 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 A.

For the most standardized laboratory comparisons, test fluid b is recommended as the least variable of the test fluids categorized above.

In all these cases, it should be understood that the results are relevant to each individual fuel and that significant 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 recommended in item b should 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 Clean, distilled, or deionized water with a surface tension of 72 mN/m ± 2 mN/m at 20 °C ± 1.5 °C.

5. TEST CONDITIONS

5.1 Volume of Fuel

Shall be five times the flow rate, per minute, with a minimum of 38 L.

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 the emulsion generated circuit fuel flow rate.

6. TEST STAND VALIDATION

6.1 Determine the minimum and maximum flow rates at which the test stand is to be qualified, and test first at the minimum flow rate.

6.2 Install a straight pipe in the location where the test water separator would be located.

6.3 Adjust the emulsion generation circuit flow to 25 Lpm using the throttling valves, while maintaining the minimum test flow rate in the filter test circuit.

6.4 Open the water valve and adjust water flow rate to be 0.25% of the emulsion generation circuit flow rate (0.25% of 25 Lpm = 63 ml/min).

6.5 Run the validation test for 1 h, and take samples from the influent, effluent and cleanup sample ports at 20 min intervals, starting 20 min after the water injection has commenced.

6.6 Analyse the samples using the Karl Fischer apparatus and report water content as ppm.

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:

$$Titration\ Reading\big(in\ ppm\ (volume\ fraction)\big) = \frac{Titration\ Reading\ (in\ ppm\ (mass\ fraction)) * Fuel\ Density\ (g/l)}{Water\ Density\ (g/l)} \tag{1}$$

Report water concentrations in ppm by volume

6.7 Verify that the water content of all the influent and effluent samples is within the range of 2200 to 2800 ppm, and the cleanup samples are less than 50 ppm.

6.8 The system is qualified if all the samples have the required water content.

6.9 Repeat the validation process, 6.3 to 6.8, for the maximum test flow rate for which the stand is to be qualified.

7. TEST PROCEDURE

NOTE: Fuller's Earth treated fuel and non-Fuller's Earth treated fuel [4.1(a), and (b)] 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.

7.1 For every fresh batch of fuel, determine the water saturation level in test fuel according to Appendix B.

7.2 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).

7.3 Install test fuel/water separator or filter on the discharge side of the emulsion generation pump circuit and in the filter test circuit (see Figure 1) . Adjust the emulsion generation circuit flow to 25 Lpm using the throttling valves, while meeting the required test flow rate in the filter test circuit. Air should be bled from all filters in the system at this time. The systems should circulate for at least two turnovers to adequately coat test filter media. Take initial pressure drop reading across the test filter at the rated flow. To determine turnover time use Equation 2:

$$\frac{\text{System Volume (L)}}{\text{Flow Rate (L/min)}} = 1\ \text{turnover}\ (\text{min}) \qquad \text{(Eq. 2)}$$

7.4 Open the water valve and adjust water flow rate to be 0.25% of the emulsion generation circuit flow rate (0.25% of 25 lpm = 63 ml/min). Start the clock at the same time water begins to flow and water flow rate is set. This point is zero test time. Water must be injected at the suction side of the pump.

NOTE: To insure the proper water flow rate, the water line from water sump to pump should be free of air and completely full of water. Further, the water line must feed into the fuel line, as close to the suction line fitting of the pump as is practical.

7.5 Periodically, drain the water from the water collection sump of the unit under test and the cleanup water separator collection sump 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 C). 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.

7.6 At approximately 10 min, collect 50 mL of the fuel sample from the petcock of the effluent sample any suitable vessel with a lid (use a beaker or vial or syringe) from the effluent sample port in Figure 1. Record this sample time. Flush the vessel 3 to 5 times and slowly withdraw 50 mL of sample over a period of approximately 10 to 15 s. Ultrasonicate fuel sample before analysis. Analyze a portion of the sample, using the Karl Fischer automatic titration apparatus. For flow rates greater than 23 Lpm, larger sample volumes should be withdrawn. Determine water concentration in ppm by volume. Repeat this sampling procedure every 20 min thereafter until termination of the test.

7.7 After approximately 10 min, take a 50 ml sample from the influent sampling port (Figure 1), using the same technique as the effluent sample. Analyze approximately 0.5 mL of this sample using the Karl Fischer automatic titration apparatus.Record the exact sampling time and the water concentration. Verify that the concentration measured is within the designated range of 2500 ppm ± 300 ppm of water is being emulsified into the fuel stream. Reconfirm this influent water concentration at approximately the same time as every alternate effluent sample (for example, take influent samples at 10 min, 50 min, 90 min, etc.; that is, every 40 min after the first 10 min sample). Record the influent concentrations with respect to time.

Periodically (suggested 30 min intervals), take cleanup water separator effluent samples at the cleanup sampling port (Figure 1) to ensure that not more than 50 ppm of water is being recirculated during the test. Analyze using the Karl Fischer automatic titration apparatus the filter influent and effluent analysis. The Cleanup water separator needs to be serviced if more than 50 ppm undissolved water appears in the effluent during a test.

7.8 Record the differential pressure across the test fuel/water separator at each effluent sample interval.

7.9 Terminate test if one or more of the following conditions is met:

a. Water concentration in effluent fuel is above acceptable level, to be specified by manufacturer or user, or

b. An equilibrium pressure drop has been reached and a minimum of 2-1/2 h of test time has been attained. Both conditions must be met. The pressure drop is said to have reached equilibrium if after 2 h the pressure drop does not increase by more than 680 Pa over a 30-min period, or

c. Differential pressure exceeds an upper limit specified by the manufacturer or user for the element or application.

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

7.11 Take a final effluent sample for analysis at test termination

8. PRESENTATION OF DATA

8.1 Plot concentration of undissolved water in effluent versus time (minutes) on a linear graph. Undissolved water = total water minus dissolved water (see Appendix B).

8.2 Plot of pressure drop (mm Hg or kPa) versus time (minutes) is optional. If plot is not presented, total pressure drop should be reported.

8.3 Note the test fluids used, IFT, DSEP, flow rate, test temperature, total test time, equilibrium pressure drop, and dissolved water saturation level in the fuel at test temperature (see Appendix B). Also, explain reason for test termination.

8.4 Calculate and report the time average undissolved effluent water level (see Equation 3).

$$Cav = \sum_{i=1}^{n} ci \left(ti - \frac{t(i-1)}{ttotal}\right) \qquad \text{(Eq. 3)}$$

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

8.5 Calculate and report average dispersed water separation efficiency (see Equation 4).

$$\text{Average Efficiency} = \left(1 - \frac{\text{Cav}}{2500}\right) * 100\% \qquad \text{(Eq. 4)}$$

9. NOTES

9.1 Marginal Indicia

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PREPARED BY THE SAE FILTER TEST METHODS STANDARDS COMMITTEE

APPENDIX A

A.1 FUEL TREATMENT TO OBTAIN FLUID AS SPECIFIED IN 4.1

A.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.

A.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.

A.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.

A.1.4 Remove the Fuller's Earth filters from the test loop, or adjust valving to isolate them from the test loop.

A.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 A1 and A2 are graphical representations of IFT, and DSEP values of one diesel fuel containing monoolein in different concentrations. It must be noted, Figures A1 and A2 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.

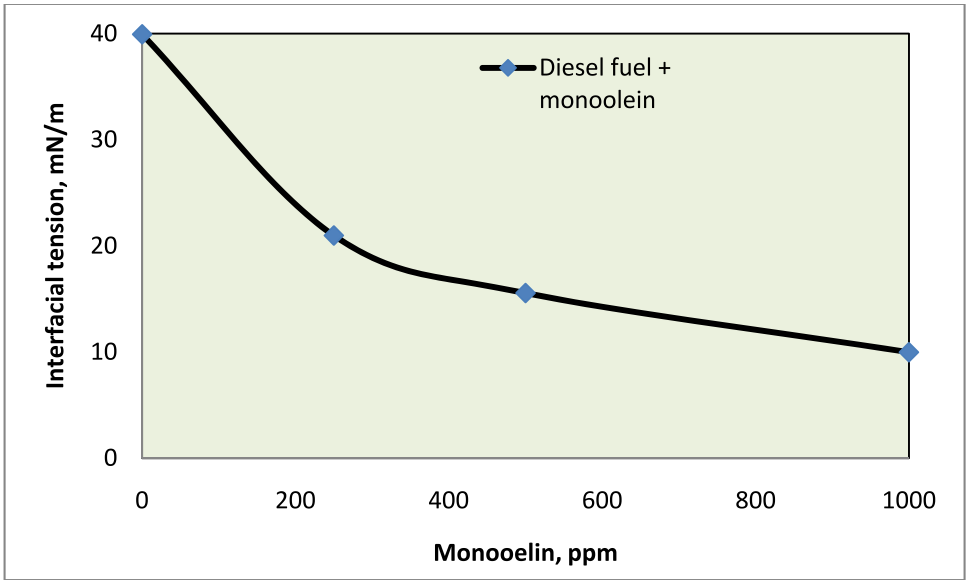

FIGURE A1 - INTERFACIAL TENSION VS. MONOOLEIN, PPM

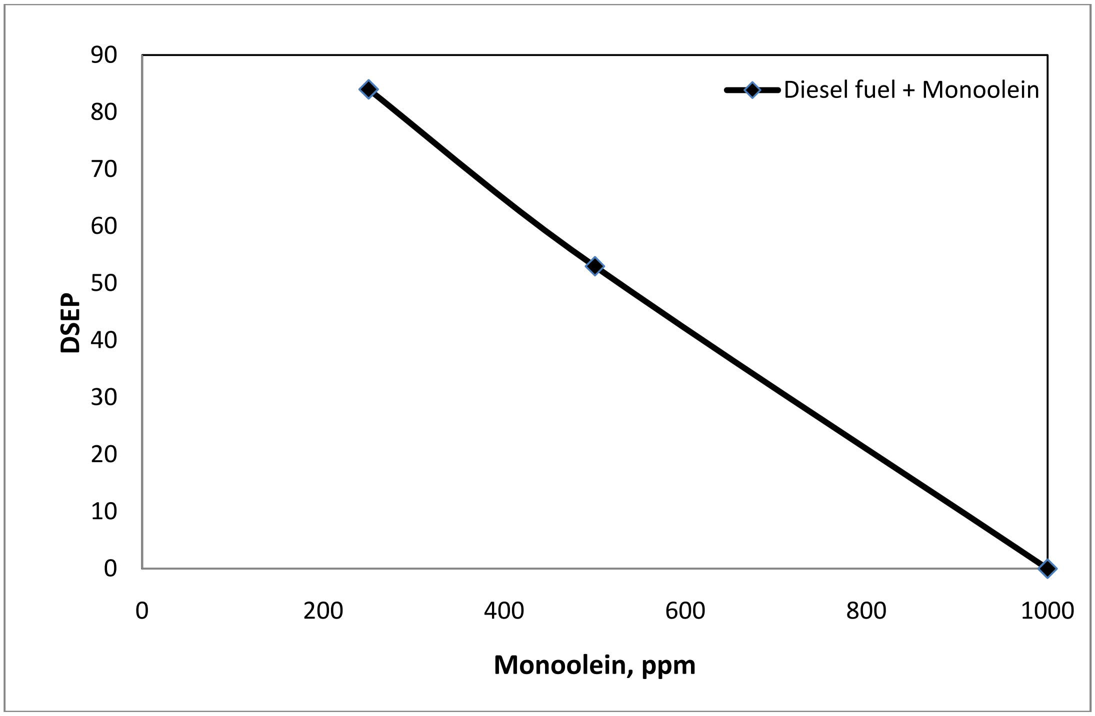

FIGURE A2 - DSEP VS. MONOOLEIN, PPM

A.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 B

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

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

B.1.1 Wash a clean sample bottle (with a rubber diaphragm cap; minimum 100 mL capacity) with distilled or deionized water so as to remove traces of detergent; dry bottle thoroughly.

B.1.2 Place 75 mL of the fuel into the dried clean 100-mL sample bottle. Insert a clean PTFE coated magnetic stirrer.

B.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 fluid/water interface is not strongly agitated and that no appreciable vortex develops as a result of mixing.

B.1.4 After mixing for at least 18 h (overnight), place the bottle gently in a water bath at the test temperature (26.2 °C) for 2 h; insert a clean, dried hypodermic syringe through the rubber diaphragm in the cap; gently withdraw 2 mL 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.

B.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.

APPENDIX C - METHOD FOR VERIFYING WATER REMOVAL EFFICIENCY

C.1 SCOPE

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.

C.1.1 Obtain volumetric reading of water collected from the test filter (V1) and cleanup water separator (V2).

C.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 6.4). This value should be within the range of water added as specified in 6.7.

C.1.3 Calculate the water balance verification efficiency (see Equation C1). This value should be compared to the time-weighted average efficiency calculated in Equation 3.

$$Water\ Balance\ Efficiency(\%) = \frac{V1}{(V1+V2)} * 100 \qquad \text{(Eq. C1)}$$