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Emulsified Water/Fuel Separation Test Procedure

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. 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 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. 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. Furthermore, testing unused "clean" water separators may provide water removal efficiencies which are far superior to those obtained from the same water separators after very short exposure to natural fuel and natural fuel contaminants.

1. ***Scope***—To determine the ability of a fuel/water separator to separate emulsified or finely dispersed water from fuels.

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2. *References*

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

2.1.1 SAE PUBLICATION—Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001.

SAE J1839—Fuel/Water Coarse Droplet Separation Test Procedure for Suction Side Applications

2.1.2 ASTM PUBLICATION—Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

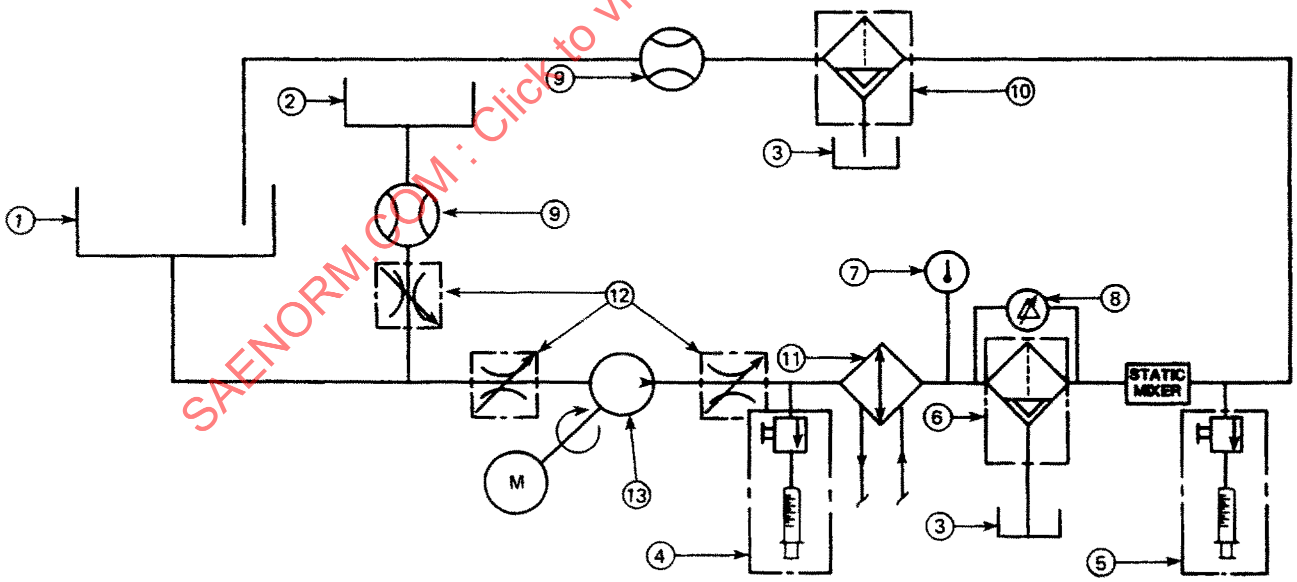
ASTM D 971—Test Method for Interfacial Tension of Oil Against Water by the Ring Method

3. *Test Apparatus*

NOTE—For test flow rates lower than approximately 3 Lpm, the test system may be operated at a minimum design flow rate, with a small (desired flow rate) portion of this flow diverted into the test fuel/water separator. For this "slip stream method," generally two final (cleanup) fuel/water separators are required. Using this slip stream method, all the piping velocity and pump pressure flow rate requirements can be met.

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. The container must have a volume of at least five times the test flow rate per minute.



1. Fuel sump
2. Water sump
3. Waste water sumps
4. Influent sampler (The in-line mixer should not be used at the influent sampler)
5. Effluent sampler (See Fig. 2)
6. Test water separator
7. Temperature gauge
8. Differential pressure indicator
9. Flow meter
10. Final water separator
11. Heat exchanger
12. Throttling valves
13. Pump

FIGURE 1—TEST SYSTEM

3.2 A 3500 rpm ± 100 rpm centrifugal pump capable of delivering at least five times (but not greater than 20 times) the test flow rate at 100 kPa. Note that magnetically coupled and multistaged centrifugal pumps should not be used. Magnetically coupled pumps have some slippage and this will affect the drop size distribution. Similarly multistaged centrifugal pumps will result in different drop size distribution The pump should be capable of producing the required pressure to overcome the system and fuel/water separator resistances.

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 final water separator assembly such that not more than 30 ppm by volume of undissolved water are 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 8 mL/s, 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 titration apparatus for water content analysis.

3.9 In-line static mixer with at least three internal mixing units. The inner pipe diameters of the mixers should be as follows:

0 to 23 Lpm: 12.7 mm I.D. mixer.

For larger flow rates, the inner diameter should be such that the fuel velocity through an equivalent empty pipe is greater than 1.22 m/s.

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 static 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.

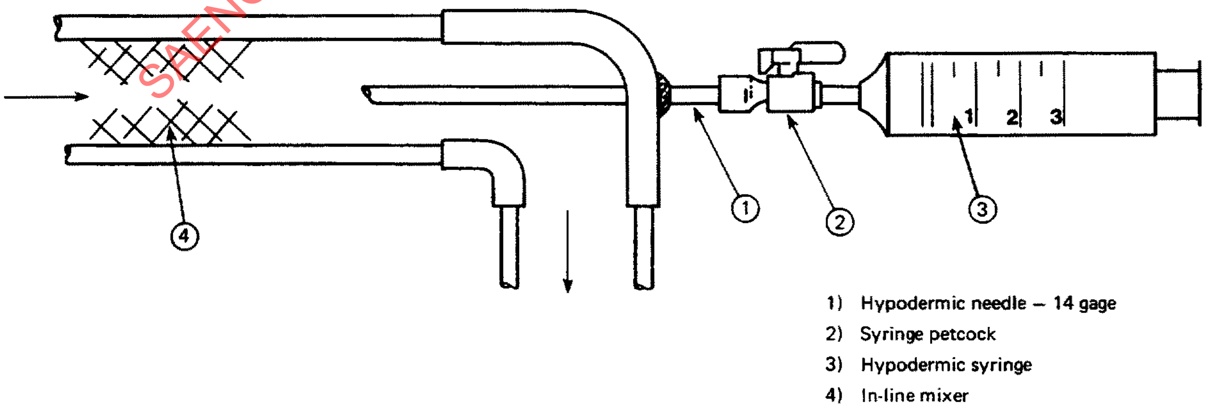


FIGURE 2—EFFLUENT SAMPLER

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 final (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.

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.
b. No. 2 diesel fuel, locally available.
c. Specially treated fluid, per Appendix A.
d. A standard reference fluid to be specified.

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.

The IFT of the test fluid should be between 25 and 30 dynes/cm.

4.2 Clean, distilled, or deionized water with a surface tension of 72 dynes/cm ± 2 dynes/cm at 20 °C ± 1.5 °C.

4.3 A 0.45 µm filter and associated equipment as listed in Appendix B.

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 **Flow Rate of Fuel—**Rated flow of unit to be tested or as specified.

5.4 **Water Flow Rate—**0.25% of fuel flow rate.

5.5 Water must be injected at the suction side of the pump.

6. Test Procedure

NOTE—If Fuller's Earth treated fuel [4.1(c)] is selected as the fuel, then it may be reused after treating again. If Fuller's Earth treated fuel is not the selected fluid, use a fresh quantity of fluid.

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

6.2 Install cleanup filter (95% efficiency at 5 µm), in place of test filter, fill fuel tank; start circulation of fuel through the cleanup filter flow until a 500 mL sample of fluid has an insoluble contaminant level less than 5 mg/L (see Appendix B) and the total undissolved water concentration is less than 30 ppm.

6.3 Install test fuel/water separator or filter on the discharge side of the pump (see Figure 1); adjust fuel flow rate by throttling the discharge side valve; take the initial (fuel only) pressure drop reading at the rated flow.

6.4 Open the water valve and adjust water flow rate to be 0.25% of fuel flow. Start the clock at the same time water begins to flow. This point is zero test time.

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.

6.5 Periodically, drain the water from the water collection sump of the unit under test. (Obviously, this is not 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.

6.6 At a point during the 10 min, insert a clean, water-free 5 mL syringe in the syringe petcock of the effluent sample (see Figure 2). Record this sample time. Flush syringe 3 to 5 times and slowly withdraw 2 to 3 mL of sample over a period of approximately 10 to 15 s. Analyze the sample immediately, using the 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.

Many titration devices will determine micrograms. Convert this to ppm by volume. (See Equation 1.)

$$\text{ppm by volume} = \frac{\text{titration reading }(\mu g)}{\text{sample volume (mL)}} \qquad \text{(Eq. 1)}$$

$$\text{or} = \frac{\text{titration reading }(\mu g) \times \text{fuel sp. gr.}}{\text{sample mass (g)}}$$

6.7 At a point in the first 10 min, take a sample (approximately 0.5 mL) of the fuel/water mixture entering the test separator and analyze the sample using the water titration device, as described in 6.6. Record the exact sampling time. This is to confirm that approximately 2500 ppm ± 300 ppm of water is being emulsified into the fuel stream. If this requirement is not met, adjust the water flow rate accordingly. Reconfirm this influent water concentration between 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.

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

6.9 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. Presentation of Data

7.1 Plot concentration of undissolved water in effluent (ppm by volume) versus time (minutes) on a linear graph paper. Undissolved water = total water minus dissolved water (see Appendix C).

7.2 Plot pressure drop (mm Hg or kPa) versus time (minutes).

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

7.4 Calculate and report the time average undissolved effluent water level in ppm by volume. (See Equation 2.)

$$E_{av} = \Sigma E_i(t_i - t_{i-1}) / t_{total} \qquad \text{(Eq. 2)}$$

where:

E_i is the effluent water concentration, ppm by volume at time t_i, min
t_{total} is the total test time, min

7.5 Calculate and report average dispersed water separation efficiency. (See Equation 3.)

$$\text{Average efficiency} = (1 - E_{av} / 2500) \times 100 \qquad \text{(Eq. 3)}$$

8. Notes

8.1 Marginal Indicia—The change bar (l) located in the left margin is for the convenience of the user in locating areas where technical revisions have been made to the previous issue of the report. An (R) symbol to the left of the document title indicates a complete revision of the report.

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 No. 2 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 may be used.

A.1.2 Periodically (about every 2 h), take a sample of the fuel in a beaker. Filter the sample through a 0.45 µm membrane and measure the interfacial tension (IFT) with distilled water at 20 °C ± 1.5 °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 between 25 and 30 dynes/cm, stop further contacting the Fuller's Earth or clay. Generally 2 to 4 h of contacting 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) 0.1% (1000 ppm) of cetane improver Ethyl D113 (Ethyl Corporation). The additive is used to simulate additives normally found in diesel fuel which, trough modification of fuel/water interfacial tension, may have significant influences on test results. NOTE: Since D113 is a commercially available additive and since it may have a finite shelf life, it is advisable to check the interfacial tension of this treated fuel (with D113) against distilled water (ASTM D 971). If the fuel treatment is successful and the D113 additive is acceptable, the interfacial tension within 1 h of D113 addition should be between 23 and 28 dynes/cm.

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

METHODS FOR OIL SAMPLE ANALYSIS

B.1 Scope—These methods cover the gravimetric procedures for the determination of n-Pentane or petroleum ether insolubles found in a representative sample of oil drawn from the laboratory test stand in the manner specified by the filter test procedure.

B.2 Summary of Methods

B.2.1 The preferred procedure, Method A, uses a microporous filter membrane technique for the separation of solids from the test fluid. This procedure can be effectively used for most sample analyses; however, where extreme accuracy is required or where the level of contaminant is very low, additional steps are recommended. These steps are described at the end of this procedure.

B.2.2 An alternate procedure, Method B, uses centrifugation for solids separation.

B.3 Analysis Method A (Membrane Filtration)

B.3.1 Test Apparatus and Materials

B.3.1.1 Membrane filters, white, plain, 47 mm diameter, 0.45 µm pore size (1).

B.3.1.2 Filter holder, 47 mm, consisting of a borosilicate glass or stainless steel funnel and funnel base, plus clamp (2).

B.3.1.3 Analytical balance, accurate to 0.0001 g.

B.3.1.4 Petri dishes, 60 mm ID.

B.3.1.5 Noncirculatory air oven, capable of maintaining a temperature of 90 °C.

B.3.1.6 Forceps, flat bladed (3).

B.3.1.7 Washing bottles and storage bottles.

B.3.1.8 Vacuum system, aspirator or pump, capable of producing 381 mm (15 in) Hg vacuum. If pump is used, a cold trap should be installed.

B.3.1.9 Petroleum ether (filtered) 30 to 60 °C boiling point range.

B.3.1.10 n-Pentane (filtered).

B.3.2 Test Preparations

B.3.2.1 SOLVENTS—Solvents used for flushing and dilution of sample must be prefiltered through a 0.45 µm membrane and stored in a container, which has been thoroughly washed, dried, and flushed three times with filtered solvent.

B.3.2.2 FILTER MEMBRANES (0.45 µm PORE SIZE)—Identify each membrane with a sample number by marking the rim (sealing edge) with a ballpoint pen.

B.3.2.3 EQUIPMENT—The filter funnel, petri dishes, and all other glassware used must be washed with detergent, rinsed thoroughly, oven dried, and flushed with filtered solvent before use.

B.3.3 Test Procedure

B.3.3.1 With clean forceps, place filter membrane in an open petri dish and oven dry for 30 min minimum at 90 °C.

B.3.3.2 Stand petri dish, with the cover slightly ajar, in the balance room or area near balance. Membrane must be protected from airborne contamination.

B.3.3.3 Allow 30 min (minimum) for the membrane to equilibrate with the ambient temperature and humidity before weighing.

B.3.3.4 Weigh membrane to the nearest 0.0001 g.

B.3.3.5 Place membrane centrally on funnel base, center funnel position, and clamp assembly securely. Place a large watchglass on top of funnel to protect the membrane until used.

NOTE—Static electricity is frequently generated during the filtration process. Since the solvents are highly flammable, it is recommended that the equipment and operator be safely grounded. In addition, the vacuum pump must be located in a well-ventilated area and/or the pump exhaust vented to a safe area.

B.3.3.6 If the liquid sample is stored in a container and the weight of the sample must be known, the outside surface of the container should be rinsed thoroughly with prefiltered solvent, and then the cap removed. Allow the container to reach room temperature again (about 5 min), then record weight of container and sample (no cap) to nearest 0.0001 g.

B.3.3.7 Pour 0.150 L of solvent into the filter funnel and add the sample, rinsing the sample bottle inner surface only. Put the clean sample bottle aside for reweighing. Apply 381 mm (15 in) Hg vacuum to the flask and maintain a liquid head in the funnel until filtration is completed. During this operation, rinse the inner surface of the funnel, using the wash bottle. Repeat this wash down several times, using at least 0.150 L of solvent. About 0.400 L of solvent should be used in the entire analysis.

B.3.3.8 With the vacuum still applied, carefully remove the clamp and funnel. Wash the sealing rim of the membrane with solvent by directing a gentle stream from the wash bottle. Direct the stream toward the center of the membrane, taking care not to wash off any of the contaminant. Also wash the sealing rim of the filter funnel onto the membrane.

B.3.3.9 Disconnect vacuum and carefully remove filter membrane and place into a covered petri dish. Use clean forceps for handling.

B.3.3.10 Dry membrane and reweigh as described in B.3.3.1 and B.3.3.4.

B.3.3.11 Reweigh the empty sample container and record the weight. Be sure that the outside of the container is free from oil before reweighing.

B.3.4 Evaluation of Results—The difference in weight between B.3.3.1 and B.3.3.4 is the weight of contaminant solids collected. The weight of oil analyzed is the difference in weight between B.3.3.6 and B.3.3.11, expressed in % w/w:

Percent n-Pentane (or P.E.) insolubles (see Equation B1):

$$\frac{\text{Height of contaminant solids collected}}{\text{Height of oil sample analyzed}} \times 100 \qquad \text{(Eq. B1)}$$

B.3.5 Additional Suggestions of Technique

B.3.5.1 During periods of high humidity, the cooling effect of the solvent evaporation will cause moisture condensation on the disc. This may retard the complete solvent washing and solid deposit; an intermediate air drying step followed by an additional wash with solvent may be necessary.

B.3.5.2 The analyst may insert, between the filter membrane and the base, a porous absorbent pad to insure a clean surface for the membrane and to minimize "freezing" of the filter to the base.

B.3.5.3 Visual examination of the membrane under ultraviolet light for absorbed oil residue will quickly determine if the rinsing operation was thorough. Traces of residual oil will show a fluorescent effect, whereas a clean membrane will not.

B.3.6 Additional Operations for Improved Accuracy of Analysis

B.3.6.1 CONDITIONING FILTER MEMBRANE

B.3.6.1.1 All filter membranes must be conditioned by a warm water soak to remove extraneous manufacturing material and to assure accurate and constant tare weight. Place the filter membrane in a clean beaker containing prefiltered distilled water at a temperature of 32 to 38 °C (90 to 100 °F). Cover the beaker and soak the filter membrane for 1 h.

B.3.6.1.2 Carefully remove the filter membrane with clean forceps. Allow all surface water to run off the membrane.

B.3.6.1.3 Place filter membrane in an open petri dish. Place petri dish, with cover slightly ajar, in the oven and dry for 60 min at 90 °C minimum.

B.3.6.1.4 Remove the petri dish, with membrane, from the oven and place in a dessicator, or constant temperature and humidity area, with cover slightly ajar. Allow 30 min (minimum) for the membrane to equilibrate with environmental conditions before weighing.

B.3.6.2 CONTROL FILTER MEMBRANE

B.3.6.2.1 A control filter membrane should be carried along for all analysis work. Subject this membrane to all preparation, handling, and weighing techniques of B.3.6.1.1 through B.3.6.1.4.

B.3.6.2.2 Insert the control filter membrane directly beneath a test membrane in the filter holder in at least one instance during a single or group test run. Subject it to all handling and weighing techniques specified in B.3.3.9 and B.3.3.10.

B.3.6.2.3 Apply the weight change of the control filter as a correction factor to test results, subtracting this factor when the control filter shows a weight increase or adding the factor when the control shows a weight decrease.

B.4 Analysis Method B (Centrifugation), Alternate

B.4.1 Test Apparatus and Materials

B.4.1.1 Air oven capable of maintaining 105 °C.

B.4.1.2 Clamp curved type.

B.4.1.3 Centrifuge tube(s), 0.100 L size.

B.4.1.4 Centrifuge, capable of 98 km/s^2.

B.4.1.5 Desiccator (5).

B.4.1.6 Analytical balance.

B.4.1.7 ACETONE—30 to 60 °C boiling point range (or equivalent).

B.4.1.8 n-Pentane or equivalent.

B.4.1.9 Stiff Wire—Suggest stainless steel.

B.4.1.10 Wash bottle.

B.4.2 Test Preparation

B.4.2.1 SOLVENTS—See Section B.3.

B.4.2.2 Wash forceps and centrifuge tubes in a detergent.

B.4.2.3 Rinse with water to remove suds.

B.4.2.4 Completely rinse inside and outside of centrifuge tube(s) with acetone. During this procedure, centrifuge tube should not be touched by hand.

B.4.2.5 Place centrifuge tube(s) in a 105 °C ± 3 °C oven for 45 min.

B.4.2.6 Place centrifuge tube(s) in desiccator while cooling to room temperature.

B.4.2.7 Weigh centrifuge tube(s) and record weight.

B.4.3 Test Procedure

B.4.3.1 Heat sample to 82 °C (180 °F) or until sufficiently mobile to allow shaking of sample. The sample shall be shaken until the sediment is homogeneously suspended in the oil.

B.4.3.2 Weigh 10 g of sample into a preweighed centrifuge tube.

B.4.3.3 Fill centrifuge tube to the 0.100 L mark with n-Pentane and place stopper in centrifuge tube.

B.4.3.4 Shake centrifuge tube well so that contents are thoroughly mixed.

B.4.3.5 Centrifuge for 20 min at 98 km/s^2 in a high-speed centrifuge.

B.4.3.6 Decant all but 0.003 L from the centrifuge tube without disturbing the precipitate.

B.4.3.7 Add 0.010 L of n-Pentane to the centrifuge tube.

B.4.3.8 With a clean, stiff wire, dislodge the precipitate and disperse it in the n-Pentane.

B.4.3.9 Wash all insolubles adhering to the wire back into the centrifuge tube using n-Pentane and fill the tube to the 0.050 L mark.