

SURFACE VEHICLE RECOMMENDED PRACTICE

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(R) 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 SAE J1839.)

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FOREWORD (Continued):

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.¹ The procedure is well suited to lower flow rates, although it may be applied with due caution to higher flow rates [up to approximately $1.6 \times 10^{-3} \text{ m}^3/\text{s}$ (~25 gpm)]. 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 (5 to 10 μm mean droplet size). 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.

2. REFERENCES:

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

ASTM D 971

3. TEST APPARATUS:

(A test stand meeting the following requirements is available from American Filtrona Company, P.O. Box 34668, Richmond, VA 23234.)

A test system,² as illustrated in Figure 1, is to include:

¹This 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.

²For test flow rates lower than approximately $4.73 \times 10^{-5} \text{ m}^3/\text{s}$ (0.75 gpm), 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.

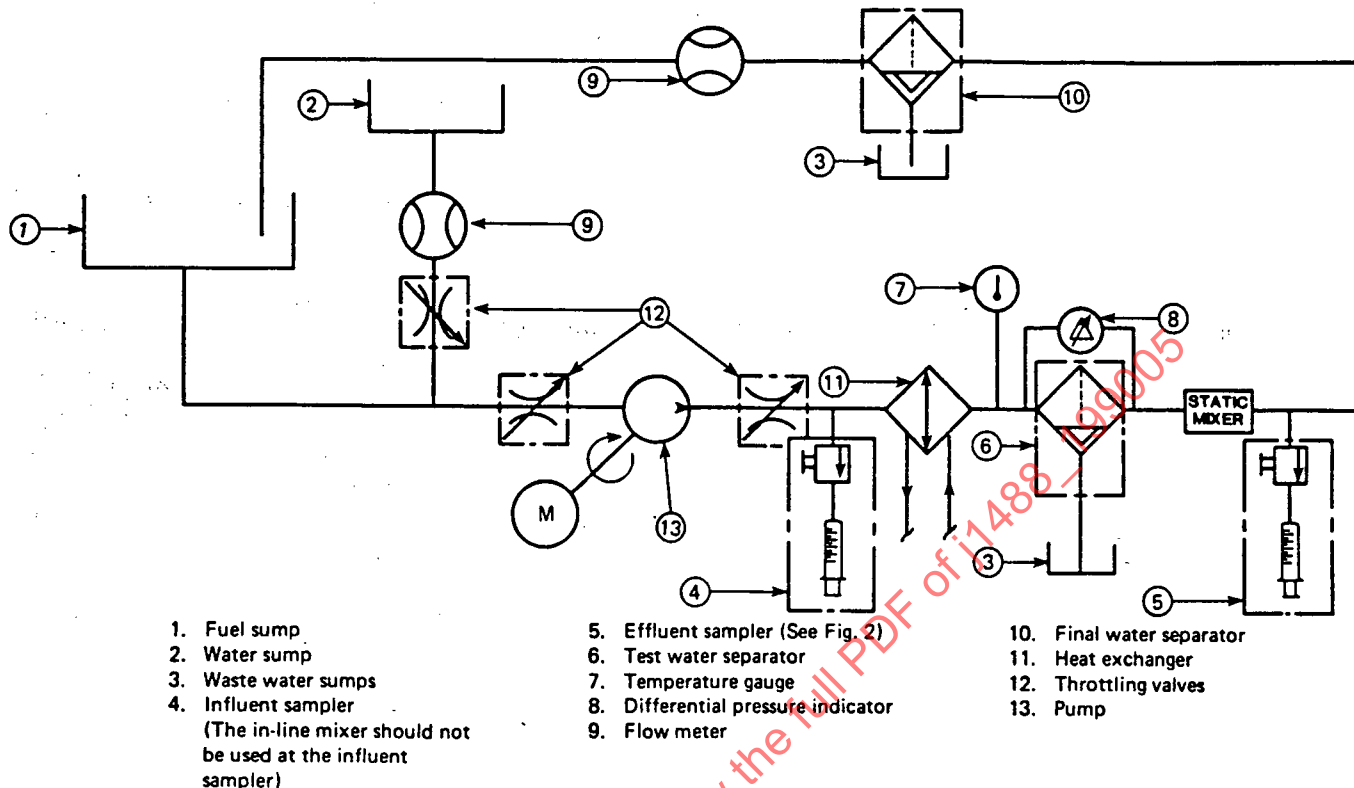


FIGURE 1

- 3.1 A flat bottom, corrosion resistant (for example, polyethylene) 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.
- 3.2 A 3500 rpm \pm 100 centrifugal pump capable of delivering at least five times (but not greater than 20 times) the test flow rate at 1×10^5 Pa (15 psi). Note that magnetically coupled and multistaged centrifugal pumps should not be used.³ 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 $\pm 1.5^\circ\text{C}$.
- 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 (for example, polyethylene) water sump with approximate capacity of $2 \times 10^{-2} \text{ m}^3$ (5 gal).

³Magnetically coupled pumps have some slippage and this will affect the drop size distribution. Similarly multistaged centrifugal pumps will result in different drop size distribution.

- 3.7 Water flow meter (0 to $8 \times 10^{-7} \text{ m}^3/\text{s}$, 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 Karl Fischer Titration Apparatus for water content analysis (for example, Seragen Diagnostics, Indiana, or equivalent).
- 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 $3.785 \times 10^{-4} \text{ m}^3/\text{s}$ (0 to 6 gpm): 1.27×10^{-2} ($1/2 \text{ in}$) ID mixer (for example, Kenics Corp., Model No. 1/2-KMS-6 304 s s, or equivalent).
- 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 (4 ft/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 338.64 Pa (0.1 in Hg) and an accuracy of at least 340 Pa ($\sim 0.1 \text{ in Hg}$).
- 3.12 Syringe sampler in accordance with Figure 2. Note⁴ that the influent sampler does not have an in-line static mixer while the effluent sampler does.
- 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 (for example, Cenco Instruments, or equivalent).

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 sample of the fluid used in the application.
- No. 2 diesel fuel, locally available.
- Specially treated fluid, per Appendix C.
- A standard reference fluid to be specified.

⁴Since the water at the influent section is emulsified, an in-line mixer is not needed prior to the influent sampler.

4.1 (Continued):

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.

4.2 Clean, distilled, or deionized water with a surface tension greater than 6×10^{-2} N/m (60 dynes/cm) at $20^{\circ}\text{C} \pm 1.5$.

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 3.8×10^{-2} m^3 (38 L).

5.2 Temperature:

$26.6^{\circ}\text{C} \pm 2.5$ measured at the test separator inlet.

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 A.

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 After 10 min, insert a clean, water-free 5 mL syringe in the syringe petcock of the effluent sampler (see Figure 2). 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 Karl Fischer Titration Apparatus. For flow rates greater than $3.875 \times 10^{-4} \text{ m}^3/\text{s}$ (6 gpm), larger sample volumes should be withdrawn. Determine water concentration in ppm by volume.⁵ Repeat this sampling procedure every 20 min thereafter until termination of the test.

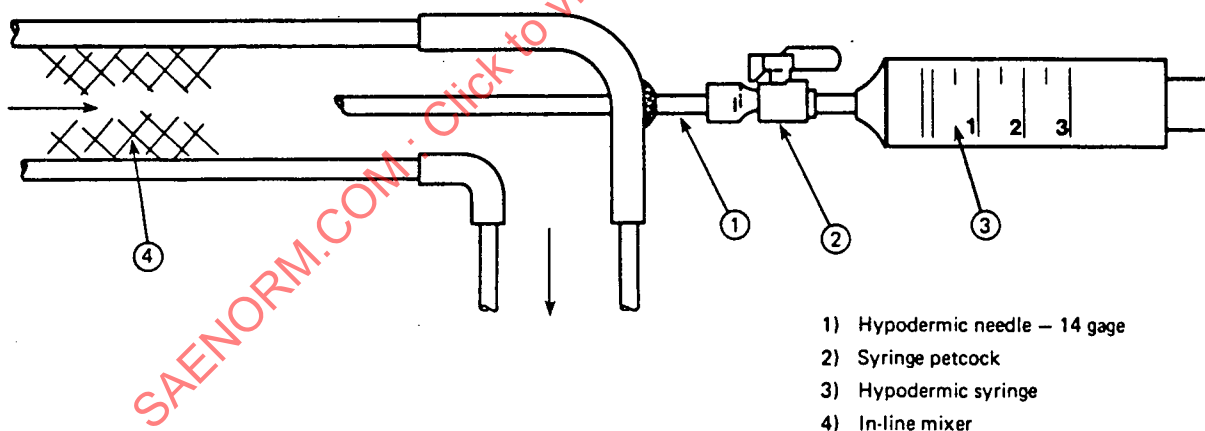


FIGURE 2 - Effluent Sampler

⁵Many Karl Fischer devices will determine micrograms. Convert this to ppm by volume.

$$\text{ppm by volume} = \frac{\text{Karl Fischer reading } (\mu\text{g})}{\text{sample volume (mL)}}$$

$$\text{or} = \frac{\text{Karl Fischer reading } (\mu\text{g}) \times \text{fuel sp. gr.}}{\text{sample wt. (gms)}}$$

- 6.7 Within the first 10 min of the test, take a sample (approximately 0.5 mL) of the fuel/water mixture entering the test separator and analyze the sample using the Karl Fischer Titration device, as described in 6.6. This is to confirm that approximately 2500 ppm \pm 300 of water are 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 678 Pa (0.2 in Hg) over a 1/2 h 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 A).
- 7.2 Plot pressure drop (in Hg) 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 A). Also, explain reason for test termination.
- 7.4 Calculate and report the time average undissolved effluent water level in ppm by volume.

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

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.

$$\text{Average efficiency} = \frac{2500 - E_{av}}{2500} \times 100 \quad (\text{Eq.2})$$

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The (R) is for the convenience of the user in locating areas where technical revisions have been made to the previous issue of the report. If the symbol is next to the report title, it indicates a complete revision of the report.

APPENDIX A

Method for Determining Saturation Level of Dissolved Water in Fuel

- A.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 in oven.
- A.2 Take about 150 mL of test fuel [4.1(a) or 4.1(b)] and filter fuel through a 0.45 μ m membrane compatible with fuel oil.
- A.3 Determine total water concentration in the fuel by the Karl Fischer method in ppm by volume.
- A.4 If the water concentration is below or equal to 100 ppm, proceed to Section A.5; if not, repeat Sections A.2 and A.3. If necessary, cool fluid to -4°C before filtering (Section A.2).
- A.5 Fill the dried, clean sample bottle almost to the top with filtered fuel.
- A.6 Place the bottle on a magnetic stirrer. Using a hypodermic syringe with a long needle, gently insert 20 to 30 mL of clean distilled water in the bottom of the sample bottle; gently insert a magnetic mixer and gently fill the bottle up to the top with "dewatered" fuel; cap the bottle. DO NOT SHAKE OR MOVE THE BOTTLE after the water has been injected. Start the stirrer 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.
- A.7 After mixing for at least 18 h (overnight), gently remove from mixer and place bottle in a water bath at the test temperature for 2 h. Then, insert a clean, dried hypodermic syringe through the rubber diaphragm in the cap; gently withdraw 2 mL of the top fuel layer and analyze for water content using the Karl Fischer method. Take three readings. The average is the water saturation.
- A.8 Convert water saturation to ppm by volume; if the concentration is not between 100 to 150 ppm by volume, then repeat steps A.6 through A.8 to confirm. Report this as the dissolved water saturation level in fuel.

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 μ m 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 15 in (381 mm) 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 15 in (381 mm) 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.3.3.10 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.3.3.10, expressed in % w/w:

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

$$\frac{\text{Weight of contaminant solids collected}}{\text{Weight of oil sample analyzed}} \times 100$$

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 90 to 100°F (32 to 38°C). 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/s².
- 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 $105^{\circ}\text{C} \pm 3$ 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 180°F (82°C) 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.
 - B.4.3.10 Shake tube until mixture is homogeneous.
 - B.4.3.11 Centrifuge for 20 min at 98 km/s^2 .
 - B.4.3.12 Decant supernatant liquid being careful not to disturb the precipitate.
 - B.4.3.13 Rinse only the outside of the centrifuge tube with acetone (during and after rinse, centrifuge tube should not be touched by hands).
 - B.4.3.14 Place tube in an oven at $105^{\circ}\text{C} \pm 3$ for 45 min.
 - B.4.3.15 Place tube in a desiccator until centrifuge tube returns to room temperature.
 - B.4.3.16 Weigh centrifuge tube to the nearest 0.0001 g and calculate weight of solids.