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# SAE J1488 JUN85

# Emulsified Water/Fuel Separation Test Procedure

**SAE Recommended Practice**
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**An American National Standard**

# EMULSIFIED WATER/FUEL SEPARATION TEST PROCEDURE—SAE J1488 JUN85

**SAE Recommended Practice**

Report of the Engine Committee, approved June 1985.

## INTRODUCTION

Water in fuels is one of the major causes of engine maintenance problems. The effects of water in fuel can be briefly summarized as (a) corrosion and (b) plugging of orifices. Water in fuels often extracts sulfur compounds, turns acidic, and enhances corrosion in engine injection systems and within the engine itself. The presence of water also encourages microbiological growth that generates sludge. This tends to plug devices with orifices, such as injectors, filters, etc. Further, due to surface tension forces, water by itself can restrict injectors in diesel engines. These effects can occur even at low concentrations of water. With the lowering of crude oil quality and increased demand, the problem of water in fuel is increasing and has received more attention.

This test code has been written to evaluate the devices designed to separate emulsified[1] water from primarily diesel and gasoline fuels,[2] although the general terminology "fuel" has been used. The procedure is more suited to low flow rates, although it may be adapted with due caution for higher flow rates [up to approximately $1.6 \times 10^{-3}$ m$^3$/s (~ 25 gpm)].

The purpose of this document is to evaluate *devices* for separating emulsified water from fuels. This should not be confused with other test methods (for example, ASTM D2550, D3602, and D3948) whose primary intent is to evaluate *fuels* in terms of their water separation characteristics.

*1. **General Information***—During the process of transportation and loading in engine fuel tanks, water in fuel gets "emulsified" or dispersed by high speed pumps. Most of this water tends to remain dispersed in the fuel due to the small drop size and viscosity of the fuel. This problem is enhanced when additives and surfactants are present in the fuel since these materials lower the interfacial tension, resulting in smaller drop sizes.

The effect of condensation in fuel tanks is similar. Water that condenses on the *sides* of fuel tanks tends to coalesce and settle into the fuel tank bottom as free water. However, water or moisture that condenses on the fuel-air interface tends to stay as a dispersion due to the extremely small drop size.

Although some of the water will settle down into the fuel tank, the more general result is that finely dispersed water tends to enter the fuel lines. This test procedure is aimed at evaluating devices designed for separation of dispersed or emulsified water from fuels. Based on the stability of the water dispersions (once dispersed, the water tends to stay in a dispersed state), this test procedure is relevant to dispersed water separation devices whether applied on the suction or discharge side of engine fuel transfer pumps.

The test procedure 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 the fuel may be #2 fuel oil that has been clay treated (conditioned) so as to enable equal and reproducible comparison of various test devices. Test fuel conditioning is an important part of the test procedure since this minimizes variability and enhances test repeatability.

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

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

The test system,[3] as illustrated in Fig. 1, is to include:

**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** 3500 ± 100 rpm centrifugal pump capable of delivering at least five times (but not greater than 20 times) the test flow rate at $1 \times 10^5$ Pa (15 psi). Note that magnetically coupled and multistaged centrifugal pumps should not be used.[4] 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 flow 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 (for example, polyethylene) water sump with approximate capacity of $2 \times 10^{-2}$ m$^3$ (5 gal).

**3.7** Water flow meter ($0–8 \times 10^{-7}$ m$^3$/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 analysis (for example, Seragen Diagnostics, Indiana).

**3.9** In-line static mixer with at least three internal mixing units. The inner pipe diameters of the mixers should be as follows: $0–3.785 \times 10^{-4}$ m$^3$/s (0–6 gpm): $1.27 \times 10^{-2}$ (½ in) ID mixer (for example, Kenics Corp., Model No. ½-KMS-6 304 s s).

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

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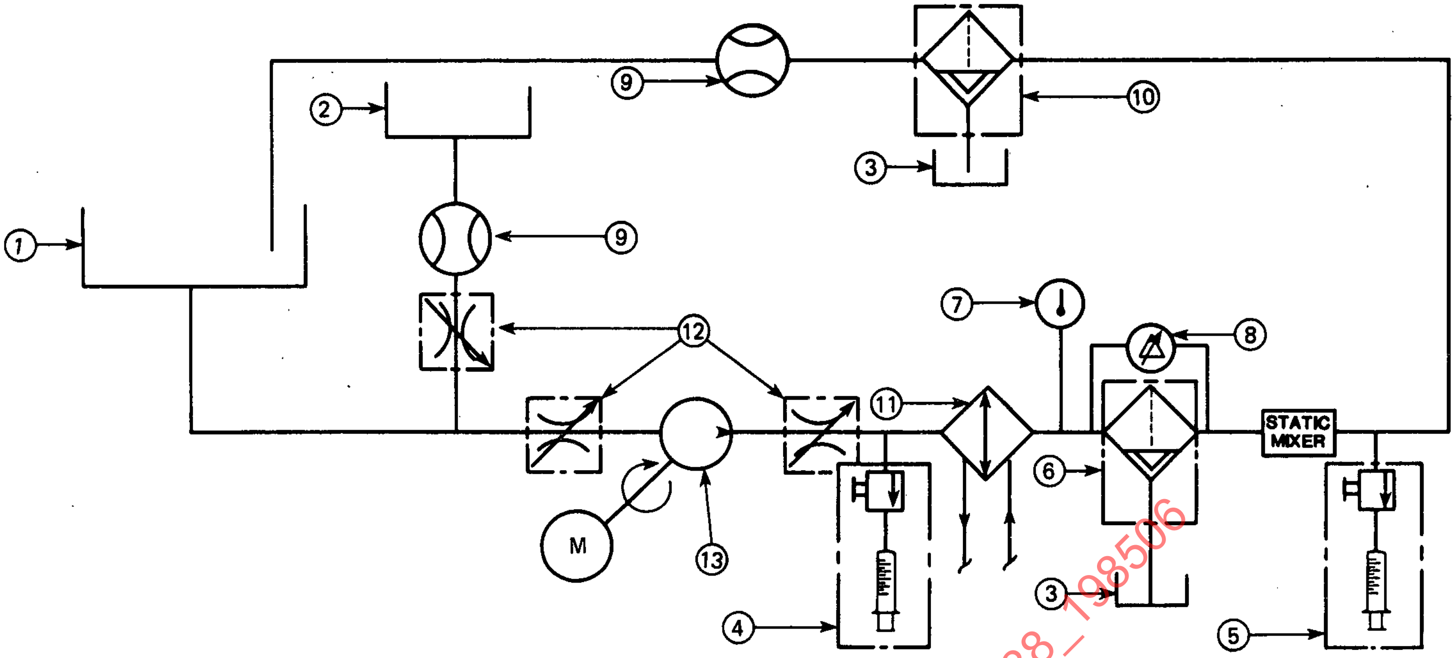
[1] Please note that the SAE Filter Test Method Subcommittee is currently in the process of evaluating a test procedure for evaluating devices designed to separate larger droplets of water from fuel.

[2] Appropriate safety precautions should be taken in construction of the test system and in conducting tests, especially when gasoline is involved.

[3] For test flow rates lower than approximately $4.73 \times 10^{-5}$ m$^3$/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 (clean-up) fuel-water separators are required. Using this slip stream method, all the piping velocity and pump pressure-flow rate requirements can be met.

[4] 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.



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

FIG. 1

equal to or greater than 0.72 m/s. Any non-rusting and non-reacting 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 (~0.1 in Hg).

**3.12** Syringe sampler in accordance with Fig. 2. Note[5] 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 paragraph 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 (clean-up) fuel water-separator (Fig. 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 D971) should be used (for example, Cenco Instruments).

### 4. *Test Materials*

**4.1 Test Fluid**—Since fuel oil is a variable quantity, the test fluid should be either of the following:

(a) #2 fuel oil treated (see Appendix C) with Fuller's Earth, followed by the addition of cetane number improver, Ethyl D113. (This treatment of the #2 fuel oil will tend to eliminate differences in testing fuel-water separators at different laboratories in different geographic locations.)

(b) A sample of the fluid used in the application.

In this case, 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. Measure and report the fuel-water interfacial tension (ASTM D971).

**4.2** Clean distilled or deionized water with a surface tension greater than $6 \times 10^{-2}$ N/m (60 dynes/cm) at 20 ± 1.5°C.

**4.3** 0.2 μm filter and associated equipment as listed in Appendix B.

### 5. *Test Conditions*

**5.1 Volume of Fuel**—Five times the flow rate, per minute, with a minimum of $3.8 \times 10^{-2}$ $m^3$ (38 L).

**5.2 Temperature**—26.6 ± 2.5°C 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 [paragraph 4.1(a)] 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 clean-up filter (90% efficiency at 5 μm or better, in place of test filter; fill fuel tank; start circulation of fuel through the clean-up 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 water concentration is less than 250 ppm.

**6.3** Install test fuel-water separator or filter on the discharge side of the pump (Fig. 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. 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** Every 20 min, insert a clean, water-free 5 mL syringe in the syringe petcock of the effluent sampler (Fig. 2), flush syringe three-five

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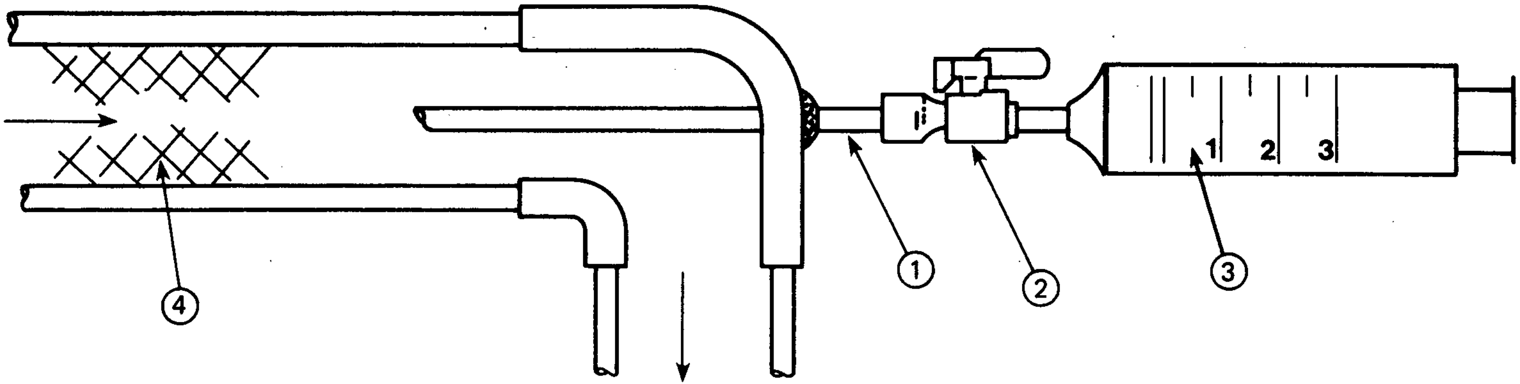
[5] Since the water at the influent section is emulsified, an in-line mixer is not needed prior to the influent sampler.



1) Hypodermic needle – 14 gage
2) Syringe petcock
3) Hypodermic syringe
4) In-line mixer

FIG. 2—EFFLUENT SAMPLER

times and slowly withdraw 2–3 mL of sample over a period of approximately 10–15 s and analyze sample immediately using the *automatic* Karl Fischer Titration apparatus. For flow rates greater than $3.785 \times 10^{-4}$ $m^3/s$ (6 gpm), larger sample volumes should be withdrawn. Determine water concentration in ppm by volume.[6]

**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 paragraph 6.6. This is to confirm that approximately 2500 ± 300 ppm 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:

(a) Water concentration in effluent fuel is above acceptable level as specified, or

(b) An equilibrium pressure drop is reached and a minimum of 2½ 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 ½ h period, or

(c) Differential pressure exceeds the upper limit specified 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:

$$E_{av} = \sum_i E_i\,(t_i - t_{i-1})/t_{total}$$

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:

$$\text{Avg. efficiency} = \left(\frac{2500 - E_{av}}{2500}\right) \times 100$$

## APPENDIX A
## METHOD FOR DETERMINING SATURATION LEVEL OF DISSOLVED WATER IN FUEL

*A1.* 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.

*A2.* Take about 150 mL of test fuel [paragraph 4.1(a) or 4.1(b)] and filter fuel through a 0.45 μm membrane compatible with fuel oil.

*A3.* Determine total water concentration in the fuel by the Karl Fischer method in ppm by volume.

*A4.* If the water concentration is below or equal to 100 ppm, proceed to Section A5; if not, repeat Sections A2 and A3. If necessary, cool fluid to −4°C before filtering (Section A2).

*A5.* Fill the dried, clean sample bottle almost to the top with filtered fuel.

*A6.* Place the bottle on a magnetic stirrer. Using a hypodermic syringe with a long needle, gently insert 20–30 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.

*A7.* 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.

*A8.* Convert water saturation to ppm by volume; if the concentration is not between 100–150 ppm by volume, then repeat Sections A6–A8 to confirm. Report this as the dissolved water saturation level in fuel.

## APPENDIX B
## METHODS FOR OIL SAMPLE ANALYSIS

***B1. 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.

***B2. Summary of Methods***

**B2.1** 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.

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[6] Many Karl Fischer devices will determine micrograms. Convert this to ppm by volume.

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

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

**B2.2** An alternate procedure, Method B, uses centrifugation for solids separation.

*B3. Analysis Method "A" (Membrane Filtration)*

**B3.1 Test Apparatus and Materials**

B3.1.1 Membrane filters, white, plain, 47 mm diameter, 0.45 μm pore size (1).

B3.1.2 Filter holder, 47 mm, consisting of a borosilicate glass or stainless steel funnel and funnel base, plus clamp (2).

B3.1.3 Analytical balance, accurate to 0.0001 g.

B3.1.4 Petri dishes, 60 mm ID.

B3.1.5 Noncirculatory air oven, capable of maintaining a temperature of 90°C.

B3.1.6 Forceps, flat bladed (3).

B3.1.7 Washing bottles and storage bottles.

B3.1.8 Vacuum system, aspirator or pump, capable of producing 15 in (381 mm) Hg vacuum. If pump is used, a cold trap should be installed.

B3.1.9 Petroleum ether (filtered) 30–60°C boiling point range.

B3.1.10 n-Pentane (filtered).

**B3.2 Test Preparations**

B3.2.1 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.

B3.2.2 Filter Membranes (0.45 μm pore size)—Identify each membrane with a sample number by marking the rim (sealing edge) with a ball-point pen.

B3.2.3 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.

**B3.3 Test Procedure**

B3.3.1 With clean forceps, place filter membrane in an open Petri dish and oven dry for 30 min minimum at 90°C.

B3.3.2 Stand Petri dish, with the cover slightly ajar, in the balance room, or area near balance. Membrane must be protected from airborne contamination.

B3.3.3 Allow 30 min (minimum) for the membrane to equilibrate with the ambient temperature and humidity before weighing.

B3.3.4 Weigh membrane to the nearest 0.0001 g.

B3.3.5 Place membrane centrally on funnel base, center funnel position, and clamp assembly securely. Place a large watchglass on top of tunnel 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.

B3.3.6 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.

B3.3.7 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 washbottle. Repeat this washdown several times, using at least 0.150 L of solvent. About 0.400 L of solvent should be used in the entire analysis.

B3.3.8 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 washbottle. 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.

B3.3.9 Disconnect vacuum and carefully remove filter membrane and place into a covered Petri dish. Use clean forceps for handling.

B3.3.10 Dry membrane and reweigh as described in paragraphs B3.3.1 and B3.3.4

B3.3.11 Reweigh the empty sample container and record the weight. Be sure that the outside of the container is free from oil before reweighing.

**B3.4 Evaluation of Results**—The difference in weight between paragraphs B3.3.10 and B3.3.4 is the weight of contaminant solids collected. The weight of oil analyzed is the difference in weight between paragraphs B3.3.6 and B3.3.11, expressed in % w/w:

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

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

**B3.5 Additional Suggestions of Technique**

B3.5.1 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.

B3.5.2 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.

B3.5.3 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.

**B3.6 Additional Operations for Improved Accuracy of Analysis**

B3.6.1 Conditioning Filter Membrane

B3.6.1.1 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 90–100°F (32–38°C). Cover the beaker and soak the filter membrane for 1 h.

B3.6.1.2 Carefully remove the filter membrane with clean forceps. Allow all surface water to run off the membrane.

B3.6.1.3 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.

B3.6.1.4 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 equilibriate with environmental conditions before weighing.

B3.6.2 Control Filter Membrane

B3.6.2.1 A control filter membrane should be carried along for all analysis work. Subject this membrane to all preparation, handling, and weighing techniques of paragraphs B3.6.1.1—B3.6.1.4.

B3.6.2.2 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 paragraphs B3.3.9 and B3.3.10.

B3.6.2.3 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.

*B4. Analysis Method "B" (Centrifugation), Alternate*

**B4.1 Test Apparatus and Materials**

B4.1.1 Air oven capable of maintaining 105°C.

B4.1.2 Clamp—Curved type.

B4.1.3 Centrifuge tube(s), 0.100 L size.

B4.1.4 Centrifuge, capable of 98 km/s².

B4.1.5 Desiccator (5).

B4.1.6 Analytical balance.

B4.1.7 Acetone—30–60°C boiling point range (or equivalent).

B4.1.8 n-Pentane or equivalent.

B4.1.9 Stiff Wire—Suggest stainless steel.

B4.1.10 Wash bottle.

**B4.2 Test Preparation**

B4.2.1 Solvents—See Section B3.

B4.2.2 Wash forceps and centrifuge tubes in a detergent.

B4.2.3 Rinse with water to remove suds.

B4.2.4 Completely rinse inside and outside of centrifuge tube(s) with acetone. During this procedure, centrifuge tube should not be touched by hand.

B4.2.5 Place centrifuge tube(s) in a 105 ± 3°C oven for 45 min.

B4.2.6 Place centrifuge tube(s) in desiccator while cooling to room temperature.

B4.2.7 Weigh centrifuge tube(s) and record weight.

**B4.3 Test Procedure**

B4.3.1 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.

B4.3.2 Weigh 10 g of sample into a preweighed centrifuge tube.

B4.3.3 Fill centrifuge tube to the 0.100 L mark with n-Pentane and place stopper in centrifuge tube.

B4.3.4 Shake centrifuge tube well so that contents are thoroughly mixed.

B4.3.5 Centrifuge for 20 min at 98 km/s² in a high-speed centrifuge.

B4.3.6 Decant all but 0.003 L from the centrifuge tube without disturbing the precipitate.

B4.3.7 Add 0.010 L of n-Pentane to the centrifuge tube.

B4.3.8 With a clean stiff wire, dislodge the precipitate and disperse it in the n-Pentane.