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SURFACE VEHICLE RECOMMENDED PRACTICE

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Test Procedure to Determine the Hydrocarbon Losses from Fuel Tubes, Hoses, Fittings, and Fuel Line Assemblies by Recirculation

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1. Scope—This SAE Recommended Practice is intended for the determination of the losses of hydrocarbon fluids, by permeation through component walls as well as through "microleaks" at interfaces of assembled components while controlling temperature and pressure independently of each other. This is achieved in a recirculating system in which liquids which are transported through walls and joints are collected by a controlled flow of nitrogen (dry) and adsorbed by activated charcoal.

2. References

2.1 Applicable Publication—The following publication forms a part of the specification to the extent specified herein. Unless otherwise indicated, the latest revision of SAE publications shall apply.

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

SAE J1681—Gasoline/Methanol Mixtures for Material Testing

3. Background Information—New State and Federal Government regulations are establishing more stringent standards for the total quantity of evaporative hydrocarbon emissions that are permitted to come from a vehicle. Besides indicating much lower levels of emissions to be achieved, they are also establishing schedules and deadlines for achieving those levels.

Hydrocarbon evaporative emissions can arise from many sources on a vehicle. A major part of those are associated with the fuel and fuel vapor systems. These hydrocarbon emissions can come from the many different parts of the fuel system and from its general usage:

a. Refueling the vehicle
b. Normal operation of the vehicle (running losses)
c. Permeation of the fuel constituents through the walls of the components that contain the fuel
d. Migration of the fuel or vapor through connections, interfaces, and any other potential leak paths of the fuel system (microleaks)

3.1 Sealed Housing Evaporative Determination (S.H.E.D.) Units—One of the primary techniques for measurement of vehicle hydrocarbon emissions is the Sealed Housing Evaporative Determination (S.H.E.D.) test. This test uses enclosed cells or structures that contain the item being tested (vehicle, system, or component). The environment of the unit is controlled and periodically analyzed to determine the quantities of hydrocarbons that are present. This technique is specified in the overall governing regulations from the Environmental Protection Agency (EPA) and the California Air Resources Board (CARB). They are used primarily for three of the aspects of the hydrocarbon emissions: static testing, normal operation, and refueling; large components (e.g., tanks) and fuel systems typically utilize this technique as well. Other procedures, such as mini-shed, and some weight change methods are normally used to measure permeability of materials or smaller components and the performance of specific interfaces.

3.2 **Other Measurement Techniques—**With the development of the lower emissions standards, the ability of parts and materials to contain hydrocarbons must improve significantly. There are two different aspects of the emissions of the hydrocarbons in a system:

a. Rate of permeation of constituents of fuel or fuel vapor through the walls of a component into the external environment
b. Performance of connections, seals, interfaces, and any other areas where different parts of a fuel or vapor system are assembled or joined together (sometimes referred to as microleaks)

In the past, permeability of materials or components has been measured by a fairly simple weight-loss technique. In general, these have involved filling a component (or a test assembly consisting of a component and a reservoir) with fuel and then measuring any weight changes that occur over a prescribed period of time and under a specific set of conditions. The most common of these long-used techniques are those described by SAE J30 and J1527; generally these do not have the level of sensitivity that is required to measure the very low rates of permeation necessary to meet the new government regulations.

When parts or materials have significantly reduced rates of permeation, the measurement techniques that are used must be able to detect much smaller quantities and changes in hydrocarbon levels. This document describes a technique that has the level of sensitivity for measurement of very low rates of permeation that is required.

3.3 **Measuring Steady-State Conditions—**When rates of permeation are measured, the initial performance of a component or material is not the same as the eventual equilibrium value. It takes a certain amount of time for the migration of the fuel through the wall or at the interfaces to achieve its steady-state rate after first exposure to the fuel. Also, with some materials, there can be other components present (e.g., plasticizer) that are driven off by the fuel, temperature, or other test conditions. Time must be allowed for these other emanations to stop contributing to any weight-loss measurements. There may also be a change in crystallinity when measuring at elevated temperatures.

It is important to note that the better a material or component's resistance to permeation, the longer it takes to achieve the steady-state conditions. For this reason, these new measurement techniques are operated at conditions that cause permeation to be accelerated. The more permeation is accelerated, the sooner equilibrium is achieved. The amount of time it takes to reach equilibrium in the permeation measurement unit can be shortened by preconditioning the specimen. Refer to 7.2 for details concerning this.

3.3.1 TIME TO REACH STEADY-STATE—For an indication of the amount of time involved in achieving steady-state conditions, experience has shown that it can take several hundreds of hours. Table 1 was developed from experience with (10% to 25%) range of alcohol-blend test fuels and 60 °C permeation test temperatures.

TABLE 1—STEADY-STATE PERMEATION RESISTANCE MEASUREMENT (GRAMS/METER^2/DAY)

Methanol Blend Test Fuels	Ethanol Blend Test Fuels	Approximate Time Necessary to Achieve Steady-State Permeation Measurement (Hours)
0 to 25	0 to 10	over 1000
25 to 50	10 to 20	1000
50 to 100	20 to 40	800
100 to 200	40 to 80	600
200 to 400	80 to 160	500
over 400	160 and over	400

This time is the continuous exposure time to the identified test conditions. It is a summation of the preconditioning step and time in the permeation test unit (see 7.2). If temperatures are lower than 60 °C or test fuels are "less active" than those containing alcohol, the overall time will be longer than shown in Table 1.

3.3.2 DETERMINING STEADY-STATE—Equilibrium can be determined by three different techniques. Each of them are described as follows in general terms. In all such procedures, however, there are small measurement errors that can arise because of rounding-off of units, seemingly insignificant changes in test conditions, minuscule differences in procedures followed, etc. The second section describes a procedure for determining if steady-state is achieved that includes the effects of these variations.

3.3.2.1 General Means to Determine Steady State

3.3.2.1.1 Length Change Measurement—Many materials being tested for permeation will simultaneously experience changes in length when exposed to the test fuel. The point at which all length changes have ceased, can give an estimate that the effects of the fuel on the specimen have reached steady state. This means that the permeation of the fuel through the walls of the specimens are at steady state also. This is not always practical to follow as a procedure because permeation rate can be at a steady-state level (for all practical purposes) well before dimensional changes of the component being tested have truly stopped altogether.

3.3.2.1.2 Rate of Weight Change—Plot canister weight as a function of time. Before steady state is reached, the line that is plotted will show a changing slope. When such a plot shows that at least 5 successive points establish a straight line, steady-state has been achieved. The slope of that line (gm/day) is the equilibrium rate of permeation for the specific specimen being tested.

3.3.2.1.3 Weight Gain—Plot canister weight change that occurs at each measurement time. Before steady state is achieved, the weight change will fluctuate or will show some sort of a trend. When the plot shows that at least 5 successive measurements yield the same weight change, then the steady-state weight change per unit time is the equilibrium rate.

3.3.2.2 Caution Concerning Steady-State Conditions—Care must be taken to be sure that the conditions described in the "Rate of Weight Change" and "Weight Gain" sections are not due to the inability of the charcoal to adsorb any more fuel. Complete saturation of the charcoal is not steady-state permeation (refer to 4.1.17, 5.3, 7.4, and Appendix A).

3.3.2.3 Practical Means to Determine Steady State—As the canisters are weighed, the permeation rate that the specific weight measurement represents should be calculated. That rate would be expressed in terms of grams/day, grams/meter^2/day, etc. (refer to 3.5 and 8.3). The calculated rate on 2 successive days should be used to determine a 2-day moving average (average of Day 1 and Day 2 measurements is first data point; average of Day 2 and Day 3 measurements is second data point, and the average of next to last and last days measurements is last data point). Once the moving 2-day averages the rate of permeation have been calculated for at least 3 to 4 weeks of test results, the following steps should be followed:

a. Plot 2-day moving average as a function of time (days).
b. As the rate of permeation nears steady state, that plot will approach horizontal.
c. Calculate a least - squares line for the 5 most recent points used to make the plot.
d. Steady state can be defined as the time when that least squares line is horizontal within ±5% of the absolute value of the rate of permeation indicated as the steady-state value.

EXAMPLE—An example of this are an absolute rate of 20 grams/meter^2/day would have to show a 5-day least squares plot of the five 2-day moving average points within ±1 g/m^2/day of being horizontal over the whole 5-day period.

3.4 Test Conditions—For permeation rate to be measured accurately, many different aspects of the test procedure must be controlled carefully. They are all factors to consider, but can have a wide variation in importance. The following paragraphs form the list of common test conditions which control permeation and give an indication of their importance in obtaining accurate permeation test results. Whenever the permeation performance of one material, construction, or part is compared to another, test conditions must be as consistent as possible. No comparison is possible when these aspects of the test differ.

3.4.1 TEMPERATURE—This is a significant factor that must be controlled very carefully because the rate of permeation will change significantly with relatively small temperature changes. For example, the permeation rate of most hydrocarbons through Nylon 12 will double with a temperature increase of only 10 or 15 °C. The rate can increase even more when multilayer wall constructions are involved. For such multilayer walls, the rate of permeation can increase 10 or even 20 times as temperature rises from ambient to 60 °C. Another example are materials that change dramatically when you go from 40 °C to 60 °C (such as EVOH). Because EVOH's glass transition temperature is ≈ 55 °C, the material changes structure during the transition to 60 °C; the result is a greater change in rate of permeation. For precise permeation measurement, the temperature of the fuel must be controlled very closely in the test.

3.4.2 TYPE OF FUEL—Alcohol-blend fuels have been used to evaluate materials for potential use in fuel and emissions applications. The effect of such blends on specific materials and composites of more than one material can be significant. For this reason, the test fuel used should be representative of what is likely to be actually encountered in the field. Also, if any comparisons among materials in the area of permeation resistance are ever made, the test fuel used must be as much the same as possible. Typical fuels used in this test are alcohol blends between 5% and 25%. The basic test fuel for use with this procedure should be CM15; with CE10, (ASTM Ref. Fuel C and 10% Ethanol by volume) used a second test fuel; refer also to 5.1, 7.5, and SAE J1681).

Permeation is a result of the solubility and diffusivity of a fluid in a material. Therefore, fuel permeability is affected by the solubility of a given fuel constituent in a given fuel containment material. This solubility effect can be readily observed by measuring the volume swell of a given material when exposed to a specific type of fuel. In many cases, when comparing two different materials that do not have a plasticizer, the greater the swell, the more easily the fuel is dissolved into the material; the resulting permeation rate will then be higher. In other cases, differences in diffusion will cause permeation to be different from predictions based on solubility alone.

Fuel constituents can be broadly classified in two ways: as polar/non-polar and as solvents/co-solvents. Polar or non-polar types will mix readily with their own kind but not with their opposites. Co-solvents permit polar and non-polar solvents to mix. The majority of hydrocarbon compounds in fuel are non-polar. Methanol is one of the most strongly polar fuel constituents. Aromatic hydrocarbons are co-solvents. Their presence is essential for methanol to mix in gasoline.

Plastic and elastomeric materials can also be broadly classified as polar and non-polar. For example, Polyethylene is non-polar, Nylon is polar. Therefore, gasoline (a primarily non-polar solvent) tends to permeate Polyethylene readily. However, Polyethylene is quite resistant to permeation of pure methanol. The opposite is true for Nylon which is permeated by methanol much more readily than gasoline.

The behavior of mixtures of gasoline is non-ideal in that the resulting properties or effects are not a linear relationship between the effects measured in a pure sample of either liquid. Gasoline/Methanol mixtures tend to produce much greater effects on materials at a methanol concentration of at between 5% and 60%. If one is designing a fuel system for minimum permeation, it is essential to know what fuel mixtures will be encountered.

3.4.3 PRESSURE—The pressure of the fuel/fuel vapor in the component must also be controlled even though its effect is far less than that of temperature. The pressure can have a small influence on the rate of permeation through the wall, when fuel vapor is present, to the extent that the pressure can affect the concentration of the vapor. For a liquid, the concentration is already 100% and cannot be changed by a pressure change.

Another consideration for changes in pressure and the effect on apparent permeation rate is in the sealing of the joints where the tubing or component being tested is attached to another component or the test cell. When the described test procedure is used to determine the leakage of quick connectors (or other attachment devices), changes in pressure may lead to different results because of the effect of pressure on microleakage at the joints. Problems that may arise can be solved by connecting the tubing being tested to the fuel supply at a location outside of the measurement test cell.

3.4.4 WALL THICKNESS—According to the basic equations for permeation, permeation rate is directly related to wall thickness, therefore (theoretically), doubling wall thickness would cause permeation rate to be decreased by a factor of two. For materials that are crystalline in nature, permeation resistance tends to be better when the amount of crystallinity in the molecular structure is greater. For parts with thicker walls, the amount of crystallinity is usually higher because of the heat retained by the greater thickness during processing. As a result, if wall thickness is doubled, the permeation rate can decrease by more than a factor of two.

3.4.5 CONSTRUCTION OF THE PART—A given part can be made of a single material or be composed of different layers of unlike materials. This again should be noted so the most accurate comparisons and conclusions can be made when comparing different products.

3.4.6 MOISTURE CONTENT—The permeation rate being measured can be affected by the moisture content of the fuel. The barrier properties of some materials used to reduce permeation can deteriorate as moisture is absorbed. To make consistent comparisons of one material to another, the moisture content of the fuel used should be kept to a minimum by using reagent grade materials, and stored in sealed containers.

3.5 Units of Measurement

3.5.1 TYPES OF UNITS OF MEASUREMENT—For permeation resistance, the units of measurement are expressed in one of three ways:

3.5.1.1 *Grams/Day*—For a given vehicle, system, or complex component, it is most practical to measure permeation in terms of amount of emissions per unit of time. This is usually expressed in grams/day. When directly comparing the performance of two different components or systems, it is important that they be the same size or are designed to perform exactly the same task. If the overall surface area of a given part being tested is significantly different than another, similar part, conclusions on permeation performance could be misleading. Care must be taken to compare systems or parts on as much of an equal basis as possible.

3.5.1.2 *Grams/Meter/Day*—When the specimen being tested is some sort of tube or hose configuration, the amount of permeation is expressed in terms of amount of emissions per unit of length and per unit of time.

3.5.1.3 *Grams/Meter^2/Day*—When parts tested have a very simple geometry (such as a tube), the permeation rate can be expressed measured in terms of grams per unit of area per day. By including the "unit area" aspect, the actual size of the part is eliminated from the measurement. It's important to note that the area referred to is that part of the material or component actually "wetted" by the fuel.

3.5.2 CAUTIONS ABOUT UNITS OF MEASUREMENT—When using these units of measurement to compare the performance of one part to another, one material to another, one fuel to another, etc., there are a number of aspects that must be kept in mind:

Basis of comparison must be well-defined and carefully documented (see Section 9). The two components, configurations, etc., that are being examined must be compared as much the same basis as possible.

For a specific tube, care must be taken to ensure that grams/meter²/day is not confused with grams/day per meter of tubing length. For example, a tube with an O.D. of 8 mm and a wall thickness of 1 mm would require more than 53 m of length to equal a square meter of surface area.

When comparing different materials from which a part is made, the most direct comparison in performance is for a tube or other part to have the same wall thickness. If thickness differs significantly between two parts when the measurements for permeation are taken, the thickness of the wall of the part should be recorded to make a more complete set of information for comparison of the two sets of materials that comprise the part. In some cases, the performance of a material can even be expressed in terms of permeation per unit of thickness.

If a particular dimension of a part is significantly different when compared with another part, the results could be misleading unless the difference is carefully noted (an example of this, is reporting permeation in grams per meter²/day for two hoses that differ significantly in wall thickness). When comparing two parts that may be used in a particular application, it is probably least confusing to test the parts in as close to the actual configuration and size to be used in that application. The permeation would then be best expressed in grams/day or grams/meter^2 day.

4. Apparatus and Equipment—Figure 1 shows a typical schematic of components of the apparatus used to measure permeation by the recirculation technique. Details in the figure are meant to give a general indication of how the apparatus is set up. Modifications are acceptable (such as for equipment availability reasons or for meeting specific end user needs) as long as the general testing procedures, parameters, and other important criteria described in this document are adhered to.

4.1 Elements of Apparatus

NOTE—The last digit of the paragraph numbers that follow correspond to the labeling in Figure 1.

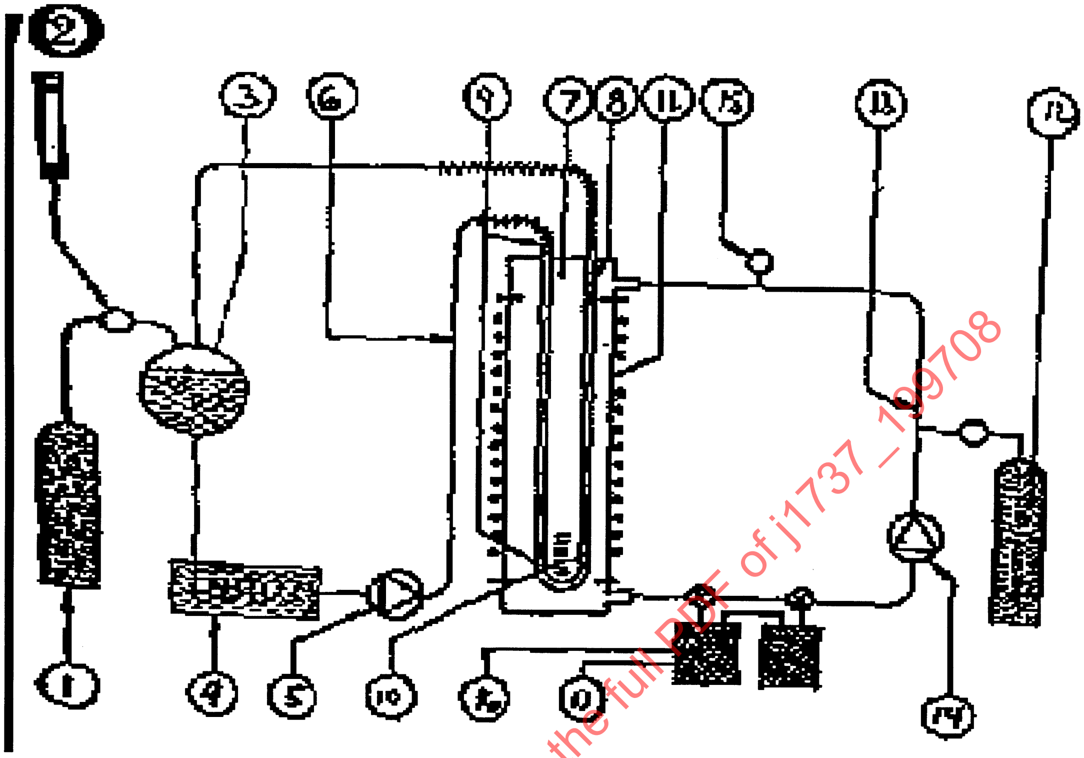
4.1.1 INERT GAS FOR PRESSURIZING FUEL—Supply of gas used to pressurize the test fuel (usually Nitrogen). Must be dry to prevent moisture being introduced into the fuel.

4.1.2 PRESSURE REGULATOR AND GAUGE—Used to set the specific pressure level needed for the test fuel (range typically used is 0 to 4 bar). For safety considerations, it is recommended that pressures should not be allowed to exceed 5 bar (unless the entire unit has been specifically. designed for a higher pressure level).

4.1.3 FUEL CONTAINER—The size of the reservoir for holding the fuel used in the test will depend on the specimen being tested. Minimum size of the reservoir shall be 800 times the internal volume of the part being tested. For a typical test procedure with an 8 x 1 mm tube that is 0.5 m long, the volume of the reservoir should be about 3 L (for each specimen that is in the test).

4.1.3.1 When fuel is first introduced in the system, there must be a means to allow the air that had been in the system to be vented. This can be accomplished with a purge valve which is located at or near the highest point of the fuel circulation system.

4.1.3.2 The operator should closely monitor the volume of the reservoir. The maximum allowable weight loss of the total fuel volume during the full duration of test should not exceed 2%. If the 2% loss level is exceeded, the fuel in the reservoir should be renewed totally, because the measured permeation rate will be significantly affected if a loss of such magnitude is experienced. If the 2% level of fuel loss is ever achieved during a test, the length of time during which that loss occurred shall be noted. From that point on, until the permeation test is complete, the fuel shall be fully renewed at that same time interval.



NOTE—The seventeen numbered elements illustrated in this schematic are described in 4.1.1 through 4.1.17.

FIGURE 1—SCHEMATIC—RECIRCULATION TECHNIQUE OF PERMEATION MEASUREMENT

4.1.3.3 For specimens with high permeation rates, the test fuel may need to be renewed to keep it within acceptable limits.

NOTE—Replenishing only a part of the fuel or adjusting composition may lead to errors.

The reservoir should be constructed to allow access to the test fuel while conducting the test when the fuel must be replenished. The permeation test can be stopped briefly (maximum of 2 hours) by following these general steps:

a. Turn off fuel heat sources
b. Turn off fuel pump
c. Carefully reduce pressure on fuel down to ambient level
d. Carefully open purge valve to release any residual pressure
e. Open reservoir and carefully insert additional fuel
f. Reseal reservoir
g. Turn pump on again
h. Bring pressure slowly back up to test levels
i. Open purge valve carefully to vent any excess air or vapor
j. Turn heat sources back on
k. Continue with test

4.1.4 HEATER FOR THE TEST FUEL—This is usually a bath heater. It must have a range up to 80 °C. Included with this component are the necessary controls to keep fuel at the desired temperature ±1 °C. The fuel may be heated at roughly the point shown in Figure 1, but the temperature measurement should be close to the test specimen to ensure the fuel in the test specimen is at the proper temperature.

4.1.5 RECIRCULATION PUMP AND REGULATOR FOR FUEL FLOW RATE—The fuel recirculation system must be able to keep flow of fuel at reasonably constant rate, depending on fuel pressure, temperature, and size of specimen being tested. For 8 x 1 mm test specimen, a typical flow rate is between 10 L/h and 20 L/h. (Higher flow rates are possible, but this may lead to a problem in controlling fuel temperature because fuel will heat up by the flow resistance in the various components).

4.1.6 TUBING TO ROUTE TEST FUEL—Must be able to contain the fuel being tested while allowing minimal loss through permeation or leaks. Typical materials used are metal and fluorocarbon hose constructions. If metal is used, it must be a type that is not corroded by the test fuel. Minimum inner diameter of this line is 4 mm.

4.1.7 TEST CELL—This is a cylinder (usually glass) that contains the specimen being tested and allows the flow of Nitrogen around the test specimen to carry the permeate to the adsorption canisters. Volume of the test cell will depend on the size of the specimens being tested or their configuration in the test cell (see 4.1.9). In general, the cell should be kept as small as possible so the hydrocarbons that do escape from the test specimen will be as high a concentration as is practical in the carrier gas that flows through the cell.

4.1.8 TEST SPECIMEN-GENERAL—This usually involves a tubular-shaped component that is completely inside the cell and through which the test fuel flows at the required temperature and pressure. Individual components or even small assemblies (such as a tube/connector combination) can be tested. The only limitation is its ability to fit within the cell. Even when components or small assemblies are tested, there is usually an amount of tubing involved. The configuration of the specimen will determine the units of permeation that are most likely to be used (see 3.5).

4.1.9 MOUNTING TEST SPECIMEN—This recirculation procedure is ideally suited for testing tubular shaped parts or small assemblies involving mostly tubing. The configuration of the test specimen and the connections that are used are important considerations. It is also important that the specimen mounting technique allows for length changes which the specimen may undergo due to thermal expansion and chemical volume swell.

4.1.9.1 Four test specimen configurations are commonly used. Others may be possible, but must meet the criteria described in this document.

4.1.9.1.1 Two test specimens can be used in the cell. They would be straight, attached to the inlet and outlet that direct the test fuel into and out of the cell, and are joined at the other end by a U-shaped metal tube that completes the fuel circuit (this is the general nature of the drawing that is Figure 1).

4.1.9.1.2 A single test specimen can be used that is bent into a "U" shape and attached to the test fuel inlet and outlet. Care must be taken that the bend is not so severe that the fuel flow is restricted in any way. For thermoplastic tubing, it is recommended that the bend be done by thermo forming.

4.1.9.1.3 A single test specimen can be suspended inside the test cell; it would be straight and would be completely blocked at the end opposite the fuel inlet. The fuel would be inserted into the tube through a hollow needle probe that extends nearly to the very end (blocked) of the test specimen. The fuel flows from the needle probe out the other end of the test specimen, resulting in a fuel recirculation with a single tube. With this technique, there is a need for only one "fuel port" attachment associated with the cell.

4.1.9.1.4 A single test specimen that extends through the length of the test cell, with fittings outside of the housing. These types of chambers can be fabricated from chromatography columns. Because the component is fixed at both ends, length change might lead to bending and thus it might touch the wall of the cell. This has to be avoided by using a suitable diameter cell, to assure proper permeate transport.

4.1.9.2 Care must be taken that connectors which attach the test specimen to the test cell do so safely and have no leaks which contribute to the hydrocarbons that are measured. If possible, all connections should be made outside the test cell. Compression fittings are recommended when attaching a flexible tube test specimen to metal tubes.

4.1.9.2.1 For the test configuration #1 in previous section, there are 4 connections required, at least 2 of which must be inside the test cell.

4.1.9.2.2 For the test configuration #2 in previous section, there are 2 connections. They can be external to the cell.

4.1.9.2.3 For the test configuration #3 in previous section, there is 1 connection (needle probe and tube combined). It can be external to the cell, and the plugged end will be in the chamber.

4.1.9.2.4 For test configuration #4 in previous section, there are no connections in the chamber, as the tubing passes through it, with the connections outside the chamber.

4.1.10 LENGTH CHANGE MEASUREMENT—This data can be helpful in determining if steady state has been achieved in the measurement of permeation. Refer to 3.3 and 7.2. When a tubing is exposed to fuel, there can be dimensional changes as elements of the fuel are absorbed into the wall. These dimensional changes are most easily measured as length change of the tubing and will continue until an equilibrium state is achieved. Care must be taken that the length measure is for the part of the specimen that is inside the cell (see 7.1).

How it is done depends on the configuration described in 4.1.9.1:

1. Measured by comparing the length of the 2 specimens to a linear gauge inside the cell. A pointer is attached to the test piece inside the chamber.
2. For U-shaped specimen, length change may be determined by indirect measurement, then additional calculations.
3. For the single - straight - tube configuration, length change can be measured by use of a linear scale or by a magnet attached to the end of the tube connected to a gauge or other measuring device.
4. Accurate length change measurement for this technique of mounting can only be done by removal from the cell (after the test).

4.1.11 HEATING MANTLE FOR TEST CELL—A heating element for the test cell is a separate means to maintain the heat of the test cell environment at the given test temperature. This is typically done by a jacket that surrounds the cell. Keeping all elements of the test cell at the required temperature will yield more consistent results.

4.1.12 CARRIER GAS FOR FUEL VAPORS—This is typically Nitrogen. Activated charcoal will adsorb water vapor as well as fuel hydrocarbon vapors. Therefore, it is absolutely necessary that gas be dry so moisture is not introduced into the permeate or into the charcoal canisters. This carrier gas could also be heated to the test temperature to produce the most consistent results.

4.1.13 TUBING FOR CARRIER GAS—Fuel hydrocarbon vapors are heavier than air. Thus the carrier gas must be routed so that its outlet is at the lowest part of the test chamber. This location of the outlet along with the temperature and pressure of the carrier gas will help keep the fuel vapor from condensing before it is picked up by the carrier gas and transported to the canisters.

4.1.14 PUMP AND FLOW REGULATOR FOR CARRIER GAS—The flow rate of the carrier gas should be checked routinely. The actual rate of flow is a compromise between two factors:

a. High flow rate carries all vapors that have permeated,
b. Low flow rate is needed to ensure optimum adsorption of all vapors by the activated charcoal.

The volume of gas flowing through the cell within 1 h should be 10 to 15 times the volume of the cell so that a sufficient rate of gas exchange can occur.

A flow rate for the rates of permeation typically encountered for smaller cells is 100 mL/min.

There are two types of systems for handling the Nitrogen carrier gas. One is a closed-loop system (shown in Figure 1). In this system, the Nitrogen is used repeatedly, circulating in the loop between the test cell, the canisters, and the pump. The second system is an "open" design. This means the Nitrogen flows through the system only once and is then exhausted to the atmosphere.

The lower the permeation rate, the lower the optimum gas flow rate. However, care has to be taken that no condensation of the permeate happens on the wall of the cell. This can be avoided by keeping all elements of the cell at the required temperature by heating the Nitrogen to the test temperature to decrease any cooling effect that the circulation of the Nitrogen might cause as described under 4.1.11.

4.1.15 PRESSURE REGULATOR—Pressure regulator for Nitrogen flow keeping a pressure slightly above atmospheric is recommended (0.25 bar above normal is typical).

4.1.16 CHARCOAL CANISTERS—Charcoal canisters are containers that hold the activated charcoal. They are mounted in the line in such manner that the carrier gas is routed through them after picking up the permeate in the test cell. There are usually 2, but sometimes 3 that are arranged in series. They must be easily removed for regular weighing since it is the weight gain of each of these canisters that result in the calculation of permeation of the test specimen. Note that the weight of the canisters should be as low as possible to increase the accuracy of the weight increase measurements that are made. The canisters must be equipped with valves for "sealing up" after filling with charcoal and during the weighing process. This avoids moisture pick-up when the canister is not connected to the measuring device. Adding cotton swab in front and behind the charcoal helps to avoid charcoal dust being taken out by the nitrogen flow (could lead to errors). It is possible the use of a cotton swab may restrict flow excessively. If that's the case, coarse steel wool is a reasonable substitute for blockage of charcoal dust.

4.1.17 ACTIVATED CHARCOAL—A suitable grade of activated charcoal must be used, such as activated carbon beads (6 to 14 mesh, Fisher catalog or equivalent). A typical specific surface needed for the adsorptive capability is 1000 to 1500 m^2/gram.

The amount of charcoal used should be as small as possible as long as it is sufficient for the adsorption of the amount of permeate that will be measured. In order to estimate a volume suitable for running the test over an acceptable period of time, it can be taken as baseline, that charcoal can absorb about 40% of it's own weight in hydrocarbons before absorbency is exhausted.

For efficiency of adsorption, the length to width (or diameter) ratio of the charcoal bed should be 6 to 1 or greater. Refer also to 5.3 and 7.4.3. See the attachment for suggested procedure for measurement of the adsorptive capacity of the charcoal.

4.2 Other Equipment—Besides the apparatus used in the specific test procedure, there are other items needed as this test procedure is carried out.

4.2.1 BALANCE—A suitable device is used to measure the weight increase of the canisters as the permeate is adsorbed into the charcoal. An analytical balance can be used manually to periodically weight each canister or it can be used for automatic weighing. In the "automatic" technique, each canister rests on a balance and is not moved. The readings of the balance are noted periodically or they can be fed directly into a recording device. Devices that are used for automatic weighing must be monitored for drift. Accuracy of balance must be such that weight can be made within ±0.01 grams or ±0.1%, whichever is "better".

4.2.2 FUEL HANDLING ITEMS—Fuel of a specific composition must be mixed for a test. All the necessary fuel and additive storage containers, mixing tanks, and means to transfer it into the reservoir before the permeation test begins are part of the equipment needed for this test.

Care must also be taken for proper disposal of the fuel when the test is completed. The steps and equipment needed must comply with local health and safety laws.

4.2.3 TOOLS—This is the variety of wrenches, screwdrivers, pliers, and other items needed to manipulate the various connectors, clamps, seals, etc.

4.2.4 SAFETY ITEMS—Safety items are necessary for use with the apparatus (see Section 6).

***5. Chemicals*—**For conducting this test procedure, a number of chemicals are needed. Their use is referred to in the detailed discussion of the apparatus (Section 4) and the procedure itself (Section 7). When using these various chemicals, refer to the Material Safety Data Sheets available from the supplier of the materials for guidelines on how to properly handle each one and the safety precautions that must be taken.

5.1 Fuel Mixtures—This will be determined by the specific needs of the test to be performed; the end user should provide guidance on how to proceed and the specific test fuel to use (refer to 3.4.2 and 7.5). The basic test fuel that should be used for comparison of performance of recirculation units or test specimens is CM15 (refer to SAE J1681).

5.2 Nitrogen (Dry)—This is recommended for use as the carrier gas for removal of the permeate and can also be used to pressurize the fuel flow. If Nitrogen is not used, the carrier gas must be inert to the chemicals and components that are part of this test. The gas must be dry to eliminate the effects of the moisture on the accuracy of the test (refer to 4.1.1 and 4.1.12).

5.3 Activated Charcoal—Use activated charcoal beads (6 to 14 mesh, Fisher catalog or equivalent). The charcoal must not be reused in the canisters after it has been exposed to fuel in this permeation test or similar procedures unless it is properly reconditioned.

Charcoal also can be reconditioned and thus be used several times. First tests showed that by heating the charcoal for 45 h at 200 °C in a hot air oven with air exhaust tube, the adsorbed hydrocarbons are sufficiently driven off. Reconditioning at 175 °C takes approximately 100 h. The reconditioning that is done should be in a dry nitrogen atmosphere. Ambient air should not be used inside the oven; it can be a source of unwanted moisture because of the humidity.

The means for determining if the charcoal has been regenerated is by comparison of weights.

a. Weight at beginning before test begins (see 7.4.4)
b. Weight at end of test (see 7.10.2 and 8.1)
c. Weight after the heating step described previously and compare to the weight measured in 7.4.4

Charcoal that will no longer be used in this test should be disposed of properly. Refer also to 4.1.17, 7.4.1, 7.4.2, and 7.4.3.

6. Safety Equipment and Facilities—With fuel being used at elevated temperatures and pressures, special safety precautions must be taken when conducting this permeation test. The operator must follow local laws, regulations, and recommended industry guidelines when setting up the apparatus and conducting all parts of the procedure. Certain equipment is suggested here, but it is meant as an initial guideline. The operator must take responsibility to meet all safety requirements that pertain. Equipment other than what's listed here may be necessary.

6.1 Vapor Control Device—Fumehood or other means of removal of fuel vapors that may emanate from the test apparatus.

6.2 Gloves

6.3 Safety Glasses

6.4 Temperature Controls—Care must be taken to not exceed the recommended maximum temperatures. Measurement, feedback, and control devices must be part of the means by which temperature is kept safely at the desired level.

6.5 Pressure Controls—Care must also be taken to not exceed the recommended maximum pressure levels. Measurement, feedback, and control devices must be part of the means by which pressure is kept safety at the desired levels.

7. Test Procedure—The following are the steps to conduct the permeation measurement of the test specimen. Since there are a variety of acceptable configurations of the apparatus, the steps described here are of a more general nature. Slight modifications will be acceptable if the intent of this document is met and the modifications are necessary to properly utilize the specific test apparatus.

The steps described here are written as if the specimen being tested is a fuel tube. Similar steps would be taken if other elements were being tested (fuel filter, tube/connector assembly, individual connector, etc.

As different steps are described, various parts of the apparatus are occasionally referenced. Refer to Figure 1 for any specific components indicated by item number.

7.1 Test Specimen Preparation—For testing a tube, cut it to the appropriate length. Measure and record the length, width, wall thickness, inside diameter, and any other dimensions that may be useful. The length is the "free length" of the tube that is inside the cell and not covered in any way by elements that are inserted in the ends. Dimensions should be taken as accurately as possible (±0.1% is recommended). Weigh the sample within ± 0.1%. Refer to 8.3 and 9.3. For testing a part with a complex geometry or a small system of more than one part, the dimensions of the components are not as important.

For all specimen tests, the general construction of the parts should also be noted. Such aspects as materials used, layer configuration, layer thickness, types of connections, etc., can all be important (see 9.3).

7.2 Preconditioning Test Specimen—It can take a very long time to achieve the required steady-state permeation level. Even at 60 °C and utilizing methanol content fuels, the time to reach equilibrium can approach 1000 h for tubes with very low permeability (refer also to 3.3).

If it is not necessary to study the phenomena that occur as the material or part reaches equilibrium, the part to be tested can be preconditioned in a recirculation system separate from the permeation tester. The following criteria must be followed during the preconditioning step.

a. Fuel used should be same type of fuel used to measure permeation.
b. Preconditioning should take place with the fuel inside the component only, total immersion of the part is not recommended because it could cause erroneous results and may actually take longer to reach equilibrium.
c. Preconditioning temperature should be the same as the temperature that will be used in the permeation measurement.

Test specimens can be kept in preconditioning until it is estimated that equilibrium has been reached. Some research suggests that length change can be useful for the purpose (refer to 4.1.10). Record the test specimen length during preconditioning. When length changes cease, permeation equilibrium is established and preconditioning can be terminated; leave the specimen in the preconditioning set up until the specimen can be connected to the permeation tester.

For an indication of time involved in preconditioning and permeation testing, refer to 3.3.

7.3 Mounting Test Specimen—Attach test specimen to the inlet and outlet ports and to other connection points that cause the routing of the fuel circulation system to be complete (refer to 4.1.9).

With all connections secure and any appropriate other routing tubes attached, insert the test specimen into the test chamber. Cover of chamber should then be secured so a complete and uniform seal is obtained.

7.4 Preparing Charcoal Canisters—(Refer to 4.1.16 and 4.1.17.)

7.4.1 CHARCOAL ADSORPTIVE CAPACITY—It can be important to determine the adsorptive capacity of the charcoal used in the canisters and the performance of the specific canisters that are used. The adsorptive capacity is a measure of how efficiently the charcoal can adsorb the emissions that come from the test specimens. If less than 90% of the emissions are adsorbed by the charcoal, then there will be inaccuracies in test results. Either the charcoal will need to be replaced by "better" charcoal and the test re-done or the inaccuracy caused by inadequate adsorption of emissions components will have to be accounted for mathematically. Refer to Appendix A for details on this procedure.

7.4.2 DRY THE CHARCOAL—The charcoal should be dried to drive off unwanted moisture that may be present. Typically this takes 36 h at 150 °C, followed by cooling the charcoal in a desiccant box.

7.4.3 CHARCOAL INTO THE CANISTERS—When drying is complete and the charcoal has cooled, place the charcoal into the canisters and close them. A typical amount used is 125 g into a canister with a capacity of 250 mL.

NOTE—Charcoal must not be reused in the canister unless it has been properly regenerated (see 5.3). If it has been previously utilized and not regenerated, it should be disposed of properly (refer also to 4.1.17).

7.4.4 WEIGH THE CANISTERS—Measure and record the weight of each canister. Each canister must be weighed as accurately as the balance will permit. This is a critical starting point for the weight gain measurements that will be made later and that will be used to calculate permeation. Canister must be weighed in the exact configuration in which it will be weighed in subsequent steps.

7.4.5 ATTACH CANISTERS TO APPARATUS—Ensure that all joints are secure, seals are uniform and complete, and appropriate routing of carrier gas is achieved. Canisters should not be exposed to any of the carrier gas or permeate until Steps 7.4 through 7.8 are completed (Refer to 7.9).

7.5 Preparing Fuel Mixture—Add necessary ingredients to the base fuel to make the test fuel to be used that has the required composition. Basic test fuel should be CM15 (refer to SAE J1681). Any other test fuel shall be agreed upon between all parties involved. Use the test fuel to fill the fuel reservoir (refer to 3.4.2 and 5.1).

7.6 Fuel Flow—Begin flow of fuel by activating the pump that is part of the system. As the various components of the fuel circuit fill with fuel, some purging of air from the system may be necessary. This is done at the reservoir. Flow of fuel should be adjusted to be at required rate (10 to 20 L/h is suggested).

7.7 Set Test Temperature—The temperature of the test is defined by the temperature of the fuel that flows through the apparatus. That fuel temperature should be controlled at the inlet and outlet ports of the test cell. If possible, other parts of the apparatus such as the wall of the test cell and even the carrier gas can be at the desired temperature level as well.

NOTE—The carrier gas temperature may be at a higher temperature.

7.7.1 TYPICAL TEST TEMPERATURES—Typical temperatures to be used in this procedure are 60 °C for liquid applications and 40 °C for vapor/emissions system components. For safety considerations, the temperature of the fuel should not exceed 60 °C. For comparison among recirculation units or between specific parts, the "baseline" test temperature should be 60 °C. If other temperatures are used, they should be agreed upon by the producer and end user involved.

7.7.2 RECORD THE TEST TEMPERATURE—Actual temperature used must be recorded. Measure and record the temperature throughout the test to confirm that it is being maintained appropriately.

7.7.3 SUGGESTED PROCEDURE FOR USING TEMPERATURE LESS THAN 60 °C—Permeation rate is influenced significantly by the temperatures involved. Running the test at less than 60 °C can have a dramatic effect on the length of time to reach the final steady-state value (see Table 1, 3.3.1). For conducting tests at temperatures less than 60 °C, the following procedure should be followed to keep the time of the test in the same realm as the times shown on Table 1:

a. Run the test at 60 °C until steady-state permeation levels are achieved (refer to 3.3.2).
b. After steady state has been achieved at a temperature of 60 °C, then drop the test temperature to the lower level (40 °C), for example).
c. Continue the test at the new temperature until a new steady-state level of permeation is achieved. Experience has shown this additional step to typically take about 1 week after temperature was lowered.

7.8 Pressurize the Test Fuel—Using the pressurizing gas (Figure 1, #1) turn on the flow and carefully pressurize the fuel to the desired level (Figure 1, #2). Examine all parts and connections carefully to ensure there are no fuel leaks. A typical pressure used for the fuel in this test is 2 bar. Care should be taken that pressure does not become too high; a typical upper limit used is 5 bar.

It is recommended that the pressure used in this test procedure matches the maximum pressure of the system in which the part being tested will be used. This should be agreed upon by the end user and all other parties involved.

For purposes of comparison among recirculation units or between test specimens, pressure of the used in the test shall be 2 bar for liquid system application and 0.1 bar above the vapor pressure of the test fuel.

7.9 Starting Test—Carrier gas should be introduced into the test cell to purge it. After this is done, the adsorption canisters is attached to the system or the necessary valve should be opened so the Nitrogen flow circuit is complete (a typical flow rate for the Nitrogen carrier gas is 100 mL/min). Once the carrier gas can enter the canisters and routine flow has started, the test has begun.

7.10 Conducting the Test—All fuel and gas flows and the temperature required must be maintained at constant levels throughout the entire duration of the test. All temperature pressures and flow must be monitored continuously to assure they remain within specified limits. They must be recorded whenever weight measurements are made or daily whichever is more frequent.

7.10.1 First 8 Hours—Determine fuel loss, pressure levels, and any other pertinent items to make sure there are no leaks. If leaks of any kind are detected, follow the shut down procedure of 7.11, correct the leak, and then repeat all of Section 7.

7.10.2 Weigh the Charcoal Canisters—This should be done at regular intervals. Initially, the canisters should be weighed every 24 h. When using smaller canisters, this period may be shorter. For longer duration tests, the weigh interval can be less frequent. As steady state is approached, the interval between weighing of canisters should again be every 24 h (refer to 7.4.1 and Appendix A).

This process should continue until it can be confirmed that equilibrium state has been achieved (see 3.3).

To ensure that the permeate is adsorbed in the canisters in a proper way, it has to be guaranteed that no interval is longer than the time to reach the maximum capacity of the canisters.

Having two canisters in line, the weight gain of the second one must not be more than 20% of the total weight gain of both canisters. If weight gain of the second one is higher than 20%, this indicates that the maximum adsorbance capacity of the first one has been exceeded.

7.11 Shutting Down the Test Apparatus

a. Turn off the sources of heat (allow system to cool down before other steps are followed).
b. Turn off source of pressure for test fuel.
c. Turn off fuel pump.
d. Stop flow of carrier gas.
e. Disconnect tube to inlet of test chamber (Figure 1, #6). Use that tube to drain fuel from the tubing in the circulation system. Use 1 to 2 bar Nitrogen to empty the test specimen of fuel. The fuel can be directed into the reservoir or into a separate container.
f. Disconnect the canisters.
g. If test is complete, then dispose of all exposed charcoal according to environmental and safety guidelines or regenerate it as described in Section 5.3. The test fuel must also be disposed of according to environmental and safety guidelines.

8. Calculations

8.1 Weight Gain—Determine weight gain (daily or at other periodic intervals). To calculate the weight gain, subtract the weight value from the previous measurement from newly recorded value for each collection bottle. Add the values together for the summation (of the 2 or 3 weights) of that time period. Refer to 7.1.

8.2 Confirm Dimensions at End of Test—Measure the effective length at the end of the test. If two test specimens are used, add the length of the two together. Refer to 7.1 for the initial dimensions that were measured.

8.3 Calculate Permeation Rate at End of Test—Use Equation 1 for measurement of permeation of tubing once the rate has achieved a steady-state value.

$$P = \frac{A}{ID \cdot \Pi \cdot L \cdot \frac{B}{24}} \cdot \frac{1}{E} \qquad \text{(Eq. 1)}$$

where:

P = Permeability in g/m^2/24 h
A = Weight gain in grams between two points in time
ID = Inside diameter of tubing in m (measure prior to testing)
P = 3.1416