

# A New Fuel Permeation Test Set-up for SAE J1737 to Determine Hydrocarbon Liquid Losses Through Tubes, Hoses and Fittings

**Abraham Pannikottu, Mark Centea**  
ARDL Engineering Group  
Akron Rubber Development Laboratory, Inc.

## ABSTRACT

This ARDL proposed measurement practice is intended for the determination of the losses of hydrocarbon fluids, by permeation through component walls as well as through "micro leaks" at interfaces of assembled components while controlling temperature and pressure independently of each other. This is achieved by a new permeation test set-up using 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. This technique will be effective for measuring permeation of fuel components with permeation values above eight (8) grams per square meter per day. This experimental set-up is a low-cost fuel permeation measurement system compared to a measurement system with flame ionization detectors (FID).

## INTRODUCTION

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. Currently, the SAE fuel tube standard, SAE J2260, recommends determining permeation performance based solely on resistance liquid permeation. The recommended practice for making the assessment, SAE J1737, does not address vapor

permeation. This paper examines the development of a low-cost analytical equipment set-up and procedure, enabling ARDL Engineering Group to quantitatively determine the total level of permeation of a specific fuel as well as the permeation of the majority of the automotive fuel components.

## BACKGROUND INFORMATION

### 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 are normally used to measure permeability of materials or smaller components and the performance of specific interfaces.

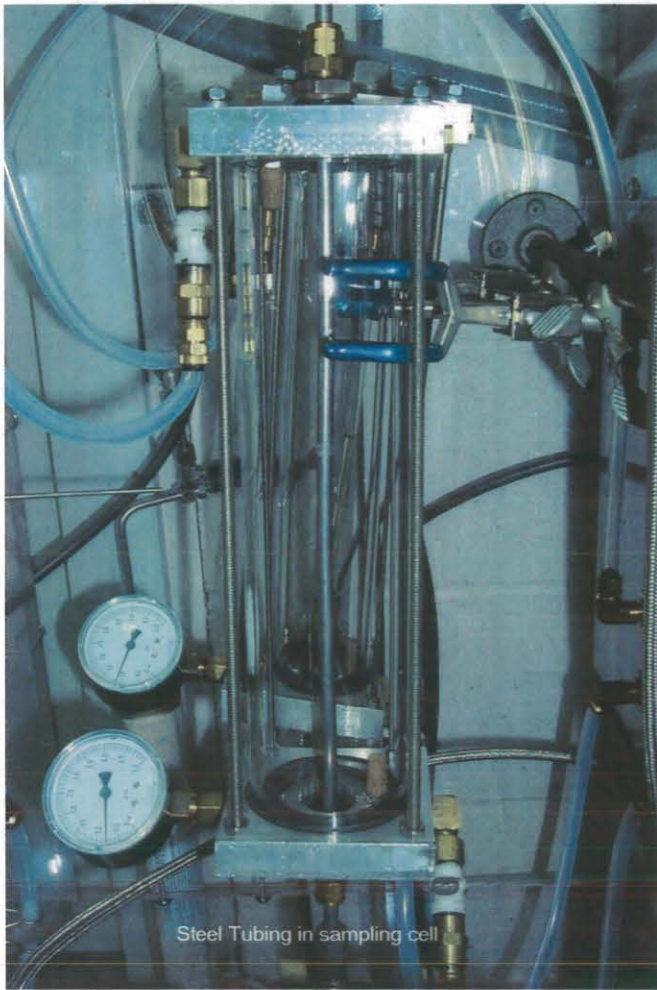


Figure 1 – Sample Cell

### OTHER MEASUREMENT TECHNIQUES

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: rate of permeation of constituents of fuel or fuel vapor through the walls of a component into the external environment, 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 micro leaks).

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 the SAE documents 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 paper describes a technique that has the required level of sensitivity for measurement of very low rates of permeation.

### MEASURING STEADY STATE CONDITIONS

When rates of permeation are measured, the initial performance of a component or materials 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.

### 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 alcohol-blend test fuels and 60°C permeation test temperatures.

TABLE 1		
STEADY-STATE PERMEATION RESISTANCE MEASUREMENT (GRAMS/METER <sup>2</sup> ·DAY )		MINIMUM TIME NECESSARY TO ACHIEVE STEADY-STATE PERMEATION MEASUREMENT (HOURS)
METHANOL BLEND TEST FUELS	ETHANOL BLEND TEST FUELS	
0 - 25	0 - 10	over 1000
25 - 50	10 - 20	1000
50 - 100	20 - 40	800
100 - 200	40 - 80	600
200 - 400	80 - 160	500
over 400	160 & 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. 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.



## DETERMINING STEADY STATE

Equilibrium can be determined by three different techniques. Each of them are described below 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.

## LENGTH CHANGE MEASUREMENT

Specimen being tested for permeation will simultaneously experience changes in length when exposed to the test fuel. When all length changes have ceased, 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 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.

## 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.

## 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.

## 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

## 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•day, etc. 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...average of next to

last and last days measurements is least data point). Once the moving 2-day averages of rate of permeation have been calculated for at least 3 to 4 weeks of test results, the following steps should be followed:

- Plot 2-day moving average as a function of time (days).
- As the rate of permeation nears steady state, that plot will approach horizontal.
- Calculate a least – squares line for the 5 most recent points used to make the plot.
- Steady state can be defined as the time when that least squares line is horizontal within  $\pm 5\%$  of the absolute value of the rate of permeation indicated as the steady state value. (An example of this is an absolute rate of 20 grams/meter<sup>2</sup>•day would have to show a 5-day least squares plot of the five 2-day moving average points within  $\pm 1$  g/m<sup>2</sup>•day of being horizontal over the whole 5-day period).

## 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.

## 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 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 temp is  $\approx 55^\circ\text{C}$ , the material changes structure during the transition to 60°C; the result is a greater change in rate of permeation. For precise permeation measurement, temperature must be controlled not only for the fuel used in the test, but also for the environment that surrounds the component. As a general rule of thumb, there is a 10% increase in permeation for every degree.

## EXTERNAL 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 effect 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 micro leakage 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. The vapor pressure of the fuel has a major influence on the permeation of the material. The external system pressure influences the permeation only by mechanical deformation of the test component or material.

## MATERIAL TYPE

The characteristics of the material being tested can have an effect. An example of this is the type of additives (glass, heat stabilizer, UV stabilizer, etc.) and their concentration. Sometimes, one or more of the additives (such as plasticizer) can emanate during the permeation test. This could affect the apparent permeation rate that is measured.

## 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 resistance 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.

## 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.

## 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 (such a materials like EVOH or Nylon 6). To make consistent comparisons of one material to another, the moisture content of the fuel used should either be kept to a minimum, controlled to a pre-agreed level or be specified.

## TYPES OF UNITS OF MEASUREMENT

For permeation resistance, the units of measurement are expressed in on of three ways:

### 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

### 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.

### GRAMS/METER<sup>2</sup>•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.

## 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. The two components, configurations, etc. that are being examined must be compared on as much the same basis as possible.

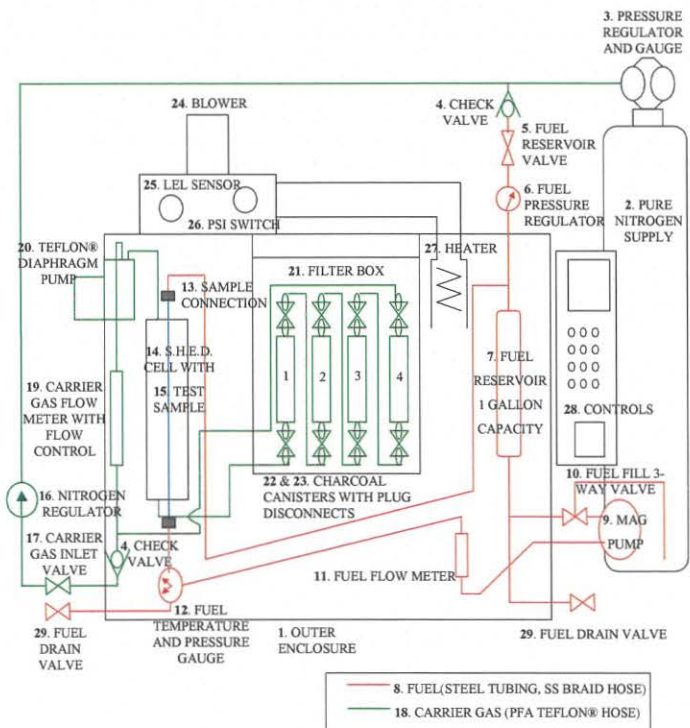


- For a specific tube, care must be taken to ensure that grams/meter<sup>2</sup>•day is not confused with grams/day per meter of tubing length. For example, a tube with an O.D. of 8mm and a wall thickness of 1mm would require more than 53 meters of length to equal a square meter of surface area.
- When 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 meter<sup>2</sup>•day for two hoses that differ significantly in wall thickness). When comparing two parts that may be used in 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•day.

**FIGURE 2**

**SCHEMATIC**

**RECIRCULATION TECHNIQUE OF PERMEATION MEASUREMENT**



**APPARATUS & EQUIPMENT**

Figures 1 thru 4 show 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.

**ELEMENTS OF APPARATUS**

**Note:** The last digit of the paragraph numbers that follow, correspond to the labeling in Figure 1.

**EXTERIOR ENCLOSURE**

External cabinet used to house all components carrying fuel and nitrogen. Enclosure is heated to control environmental temperature of components and fuel. Carrier gas tubing is routed through enclosure to prevent any condensation of fuel vapor. Enclosure is LEL monitored to prevent any build up of volatile vapors.

**INERT GAS FOR PRESSURIZING FUEL AND S.H.E.D.**

Supply of gas used to pressurize the test fuel (usually pure Nitrogen) must be dry and free of hydrocarbons to prevent contamination of fuel and/or sampling cells.

**PRESSURE REGULATOR & GAUGE**

Used to set the specific pressure level needed for the test fuel (range typically used is 0 to 4 bar, 0 to 58 psi). For safety considerations, it is recommended that pressures should not be allowed to exceed 5 bar (72.5 psi) unless the entire unit has been specifically designed for a higher pressure level.

**CHECK VALVES**

Used to prevent any back flow of vapors into nitrogen line. Isolates fuel reservoirs and sampling cells from each other.

**FUEL RESERVOIR BALL VALVE**

Allows nitrogen supply to pressurize fuel reservoir. If fuel system is free of leaks, ball valve can be closed during operation and checked during daily filter weighing. Ball valve can be left open if small leak causes system pressure to drop between daily filter weighing. Typically, fuel pressure will be maintained with ball valve closed.

**FUEL PRESSURE REGULATOR**

Mainly used to relieve excess pressure from fuel reservoir during initial heating. With open reservoir, ball valve can be used to maintain pressure due to loss of temperature or small system leak.

**FUEL CONTAINER**

The size of the reservoir for holding the fuel is one gallon. SAE J1737 recommends using a minimum reservoir size

800 times the internal volume of the part being tested. For a typical test procedure with an 8 x 1 mm tube that 0.5 meters long, the volume of the reservoir should be about 3 liters for each specimen. This reservoir size is recommended to reduce the need to change fuel mixtures during the extensive time period involved in aging as well as testing samples. By aging samples before permeation measurements, the amount of fuel required is reduced, but must be monitored.

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 by setting the fuel pressure regulator to 0 psi during fill.

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. Fuel loss can be evaluated by the total weight gain of the filters.

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 1 hour) by following these general steps:

- turn off fuel pump,
- close fuel reservoir valve,
- reduce fuel regulator to 0 psi,
- place containers at fuel drain valves,
- slowly open drain valves,
- allow fuel to drain from system,
- close drain valves,
- place dip tube from 3-way fill valve into fresh fuel supply,
- turn on fuel pump,
- allow pump to refill reservoir and plumbing,
- turn 3-way fill valve to recirculate position,
- open fuel reservoir valve
- set fuel pressure regulator fuel pump flow to desired level
- resume test.

## TUBING TO ROUTE TEST FUEL

The tubing 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.

## FUEL RECIRCULATION PUMP

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 liters/hr and 20 liters/hr. (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). This test apparatus uses externally mounted variable speed magnetically coupled gear pumps. The external mounting keeps the drive motors out of the enclosure and eliminates the pump shaft seal as a possible leak point. The variable speed drive is used to set the desired flow rate.

## WAY FILL VALVE

The 3-way fill valve allows fast filling of the reservoir without operator contact with fuel. A flexible dip tube from the fill valve allows the pump to fill the reservoir without pouring fuel or opening the reservoir.

## FUEL FLOW METER

The fuel flow meter is used to monitor the fuel flow rate though the test sample(s).

## FUEL TEMPERATURE AND PRESSURE GAUGE

Located just prior to sampling cell, this gauge provides a visual check of fuel temperature and pressure entering the test sample. Using a thermocouple and pressure transducer, the fuel pressure and temperature may be automatically recorded.

## SAMPLE CONNECTION

For material testing, the fuel line to sample connection is made externally of the sampling cell to prevent fitting leaks from influencing measurements. The connection can be made in the test cell when the fitting is tested for leakage.

## 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. 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. Multiple passes



in series through the test cell can be made to increase the effective length of the sample being tested. Compression fittings are on the cell ends.

### 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  $\pm 1^\circ\text{C}$ . 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 it is at the proper temperature.



Figure 3: Complete Permeation Set-up

### ELEMENTS OF APPARATUS

#### 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.

#### 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.

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

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.

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 tubings, it is recommended that the bend be done by thermo forming.

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.

A single test specimen that extends through the length of the test cell, with fittings outside of the housing. These typed of chambers can be fabricated from chromatography columns.

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. Compressions fittings are recommended when attaching a flexible tube test specimen to metal tubes.

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

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

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

#### LENGTH CHANGE MEASUREMENT

This data can be helpful in determining if steady state has been achieved in the measurement of permeation. 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 measured is for the part of the specimen that is inside the cell.

#1 Measured by comparing the length of the 2 specimens to a linear gauge inside the cell.

#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).

#### *HEATING MANTLE FOR TEST CELL*

An optional, but recommended 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 water jacket that surrounds the cell. Keeping all elements of the test cell at the required temperature will yield more consistent results.

#### *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 should also be heated to the test temperature to produce the most consistent results.

#### *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.

#### *PUMP & 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:

- high flow rate carries all vapors that have permeated,
- 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 one hour 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/minute.

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.

#### *PRESSURE REGULATOR*

A pressure regulator for Nitrogen flow keeping a pressure slightly above atmospheric is recommended (.25 bar above normal is typical).

#### *CHARCOAL CANISTERS*

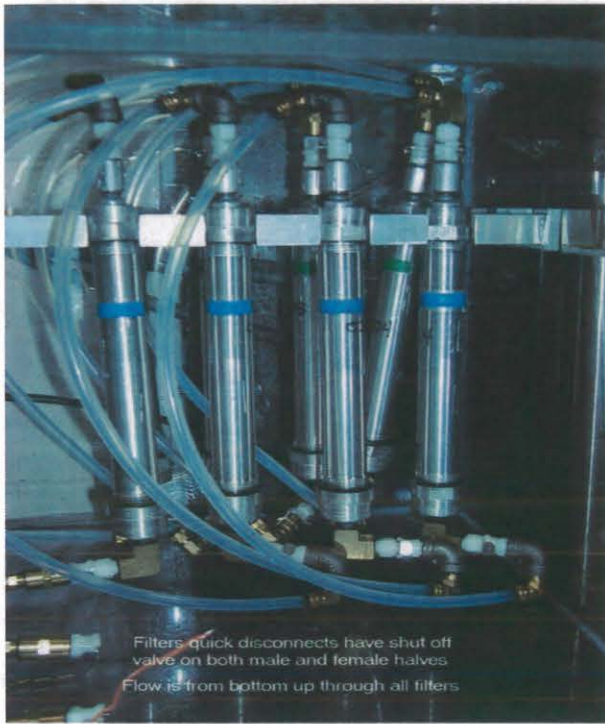
Charcoal canisters are containers that hold the activated charcoal. They are mounted in the line in such manner that the carrier gas is routed through them after picking up the permeate in the test cell. There are usually 2, but sometimes 3 that are arranged in series. They must be easily removed for 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 that 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.

#### *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<sup>2</sup>/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. For efficiency of adsorption, the length to width (or diameter) ratio of the charcoal bed should be 6 to 1 or greater.





**Figure 4: Charcoal Canisters**

#### OTHER EQUIPMENT

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

#### 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 weigh 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  $\pm 0.01$  grams or  $\pm 0.1\%$ , whichever is "better".

#### 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. The basic test fuel that should be used for comparison of performance of recirculation units or test specimens is CE-10 (refer to SAE J1681).

#### 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.

#### 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 45h at 200°C in hot air oven with air exhaust tube, the adsorbed hydrocarbons are sufficiently driven off. Reconditioning at 175°C takes approximately 100h. 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.

- weight at beginning before test begins.
- weight at end of test.

#### 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 recommended practice 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.

#### 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 ( $\pm 0.1\%$  is recommended). Weigh the sample within  $\pm 0.1\%$ . 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.

## 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 hours for tubes with very low permeability.

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

- fuel used should be same type of fuel used to measure permeation,
- 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,
- preconditioning temperature should be the same as the temperature that will be used in the permeation measurement.

## 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.

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.

## 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 used in the test shall be 2 bar for liquid system application and 0.1 bar for vapor system applications.

## STARTING TEST

Carrier gas should be introduced into the test cell to purge it. After this is done, the adsorption canisters are 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/minute). Once the carrier gas

can enter the canisters and routine flow has started, the test has begun.

## 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.

## WEIGH THE CHARCOAL CANISTERS

This should be done at regular intervals. Initially, the canisters should be weighted every 24 hours. When using smaller canisters, this period may be shorter. For longer duration tests, the weigh interval can be less frequent. Once per week should be the longest interval considered. As steady state is approached, the interval between weighing of canisters should again be every 24 hours.

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

## SHUTTING DOWN THE TEST APPARATUS

- Turn off the sources of heat (allow system to cool down before other steps are followed).
- Turn off source of pressure for test fuel.
- Turn off fuel pump.
- Stop flow of carrier gas.
- 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.
- Disconnect the canisters.
- If test is complete, then dispose of all exposed charcoal and test fuel according to environmental and safety guidelines or regenerate it. The test fuel must also be disposed of according to environmental and safety guidelines.



## EXPERIMENTAL

### WEIGHT GAIN

Typical data from the proposed set-up is showed in tables 2 thru 6.

**Table 2**

DATE	TIME	WEIGHT 1A g	WEIGHT 2A g	WEIGHT 3A g	WEIGHT 4A g	Δ1	Δ2	Δ3	Δ4	ΔT
4/27	11:00 AM	442.785	436.622	444.381	443.366					
4/28	11:00 AM	443.378	436.656	444.378	443.376	0.593	0.034	-0.003	0.010	0.634
4/29	11:00 AM	443.634	436.947	444.377	443.410	0.256	0.291	-0.001	0.034	0.580
4/30	11:00 AM	443.791	437.323	444.386	443.424	0.157	0.376	0.009	0.014	0.556
5/1	11:00 AM	443.956	437.445	444.635	443.419	0.165	0.122	0.249	-0.005	0.531
5/2	11:00 AM	444.192	437.465	445.034	443.453	0.236	0.020	0.399	0.034	0.689
5/3	11:00 AM	444.419	437.484	445.207	443.684	0.227	0.019	0.173	0.231	0.650
5/4	11:00 AM	444.598	437.523	445.241	444.056	0.179	0.039	0.034	0.372	0.624
5/5	11:00 AM	444.939	437.548	445.270	444.324	0.341	0.025	0.029	0.268	0.663
5/6	11:00 AM	445.484	437.585	445.287	444.326	0.545	0.037	0.017	0.002	0.601

**Table 3**

TUBE DIMENSIONS	TEST START	TEST END
DIA (m)	0.0061	0.0063
L (m)	0.635	0.743

### CALCULATE PERMEATION RATE AT END OF TEST

Use the following equation for measurement of permeation of tubing once the rate has achieved a steady state value.

$$P = \frac{A}{ID \cdot \pi \cdot L \cdot (B)} \cdot \frac{1}{E} \quad (24)$$

where:

P = Permeability in g/m<sup>2</sup>/24 h

A = Weight gain in grams between two points in time

ID = Inside diameter of tubing in m (measure prior to testing)

π = 3.1416

L = Total length of tubing tested in m (measure prior to testing)  
(subtract lengths of any metal inserts that are used)

B = Time (hours) weight gain accrued (difference between the two points)

E = Adsorptive factor

For parts or small systems that have complex geometries, the permeation rate is calculated by the formula  $24 \cdot A/B$ .

**Table 4**

DATE	TIME	TOTAL WEIGHT GAIN (g)	PERMEATION CALCULATION	PERMEATION (g/m <sup>2</sup> /day)
4/27	11:00 AM	0.182	C1/(0.0063*3.1416*0.743)	12
4/28	11:00 AM	0.634	C2/(0.0063*3.1416*0.743)	43
4/29	11:00 AM	0.580	C3/(0.0063*3.1416*0.743)	39
4/30	11:00 AM	0.556	C4/(0.0063*3.1416*0.743)	38
5/1	11:00 AM	0.531	C5/(0.0063*3.1416*0.743)	36
5/2	11:00 AM	0.689	C6/(0.0063*3.1416*0.743)	47
5/3	11:00 AM	0.650	C7/(0.0063*3.1416*0.743)	44
5/4	11:00 AM	0.624	C8/(0.0063*3.1416*0.743)	42
5/5	11:00 AM	0.663	C9/(0.0063*3.1416*0.743)	45
5/6	11:00 AM	0.601	C10/(0.0063*3.1416*0.743)	41

### CALCULATE PERMEATION RATE AT END OF TEST

When the steady state value of permeation rate measured by this procedure is reported, the result should be rounded off in the manner shown in the following table:

**Table 5**

RANGE OF PERMEATION RATE	RESULT SHOULD BE ROUNDED OFF
0 – 50	to nearest full unit
50 – 100	to nearest even number of units
100 – 200	to nearest 5 units
over 200	to nearest 10 units

**Table 6**

VALUE	DAY 1 THRU 6	DAY 2 THRU 7	DAY 3 THRU 8	DAY 4 THRU 9	DAY 5 THRU 10
1	27.745	41.277	38.625	36.959	41.481
2	41.277	38.625	36.959	41.481	45.527
3	38.625	36.959	41.481	45.527	43.317
4	36.959	41.481	45.527	43.317	43.759
5	41.481	45.527	43.317	43.759	42.977
AVG PERM (g/m <sup>2</sup> /day)	37.217	40.774	41.182	42.209	43.412
MAX VALUE	39.078	42.813	43.241	44.319	45.583
MIN VALUE	35.356	38.735	39.123	40.098	41.242
SLOPE	2.315	1.136	1.795	1.544	0.122
INTERCEPT	30.271	37.367	35.796	37.578	43.045
LINE START	32.586	38.503	37.591	39.121	43.167
LINE END	41.848	43.045	44.772	45.296	43.657

Average permeation during 5 days when least-squares line within ±5% of the indicated permeation = 43.412  
Permeation value reported = 43 g/m<sup>2</sup>/day

### CONCLUSION

The proposed new technique is useful for determining liquid permeation of fuel system components with a permeation value above eight (8) grams per square meter per day. This proposed practice is very cost effective compared to fuel permeation techniques utilizing a flow-through system with flame ionization detectors (FID).

## REFERENCES

1. Non Metallic Multilayer Fuel System Tubing. SAE Test procedure **J2260**
2. Permeation and other losses from fuel tubes, hoses, fittings, and fuel line assemblies by fuel recirculation. Proposed SAE test **J1737**.
3. Testing of Plastics – Determination of the Percentage of Heat-Extractable Matter. **DIN 53 738**
4. Diffusion In and Through Polymers edited by Wolf R. Vieth; Hanser Publishers (1991)
5. **T McNally, GM McNally, SB Byrne and WR Murphy**. “Transport and Sorption Properties of Various Barrier Materials used in Multi-layer Fuel Line Tubing on Immersion in Fuel Components”. ANTEC, 3, 24662-2464 (1998).
6. **T McNally, GM McNally, Mn Ahmad and WR Murphy**. “Sorption and Diffusion of Fuel Components in various Nylons”. ANTEC, 3, 2794-2799 (1997).
7. HL Frisch. “Sorption and Transport in Glassy Polymers – A Review”, J. Polym. Eng. And Sci., **20**, 1-13 (1980)
8. **J Koszinowski**. “Diffusion and Solubility of n-Alkanes in Polyolefins”, J. Polym. Sci., **32**, 4765-4686 (1986).
9. **M Matsuguchi, Y Sadaoka and K Umeda**. “Solvatochromic Study of Water Sorption in Polymer Films”. J. Appl. Polym. Sci., **63**, 1681-1691 (1997).
10. **MA Samus and G Rossi**. “Methanol Absorption in Ethylene-Vinyl Copolymers: Relationship between Solvent Diffusion and Changes in Glass transition Temperature in Glassy Polymeric Materials”. *Macromolecuels.*, **29**, 2275-2288, (1996).
11. **G Rossi and KA Mazich**. “Macroscopic Description of the Kinetics of Swelling for a Cross-linked Elastomer or a Gel, American Physical Society, Physical Review., **48**, 1182-1192, (1993)
12. **TM Aminabhavi and HTS Phayde**. “Sorption, Desorption and Permeation of Aliphatic Alkanes into Santoprene Thermoplastic Rubber”. *J. Appl. Polym. Sci.*, **55**, 17-37 (1995).
13. **RS Khinnavar and TM Aminabhavi**. “Resistance of Barrier Elastomers to Hazardous Organic Liquids”. *J. Appl. Polym. Sci.*, **45**, 1107-1125, (1992).
14. **TM Aminabhavi and J Donaldson**. “Chemical Compatibility of Geomembranes- Sorption, Diffusion and Swelling Phenomena”. *Antec '97*, 1649 (1997).
15. **SB Harogoppad and TM Aminabhavi**. “Interactions of substituted Benzenes with Elastomers”. *Polymer.*, **32**, 870-876 (1991).
16. **TM Aminabhavi and RS Munnolli**. “Sorption and Diffusion of Aldehydes and Ketones into Elastomers, *Polymer International*, **32**, 61-70 (1993).
17. **Giuseppina Ceccorulli, Maria Pizzoli and Mariastella Scandola**. “Dynamic Mechanical Relaxation's of Poly(vinlidene floride)- Poly(vinylpyrrolidone) Blends, *Polymer.*, **32**, 1251-1256 (1989).