



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

RATIONALE

Five-Year Review updates several spelling and grammar mistakes, and updates Figure 1 for readability and future editing if necessary. The procedures and equations were not substantively changed.

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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 elements of a test fuel that permeate through the walls of a test specimen and migrate through the interfaces are transported by a controlled flow of dry nitrogen to a point where they are measured. That measurement point is a device, such as a canister containing activated charcoal or other means of collection or accumulation, where the hydrocarbon losses are then measured by weight change or analyzed by some other suitable means.

2. REFERENCES

2.1 Applicable Documents

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

2.1.1 SAE Publications

Available from SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001, Tel: 877-606-7323 (inside USA and Canada) or +1 724-776-4970 (outside USA), www.sae.org.

SAE J30 Fuel and Oil Hoses

SAE J1527 Marine Fuel Hoses

SAE J1681 Gasoline, Alcohol, and Diesel Fuel Surrogates for Materials Testing

3. BACKGROUND INFORMATION

Government regulations continue to evolve and establish more stringent standards for limiting 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 is 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. Breathing losses from the engine's air intake system and from the fuel system vent through canisters
- 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 General Comment - This Procedure

This document describes a procedure that has several advantages over the other measurement techniques that are commonly used:

- It has a better level of sensitivity needed for measuring very low rates of permeation.
- It's better suited to be run at higher temperatures (up to 60 °C). This allows steady state to be achieved more quickly and yields higher measured rates of permeation that are easier to use for comparison among components.
- When alcohol blend fuels are used, this procedure can account for all the alcohol that's present in the permeate. (Doesn't account for all if you use a FID [Flame Ionization Detector] to measure.)
- This procedure provides an easier means to maintain the proper composition levels in alcohol blend fuels.
- Fuel is being circulated through the system/component being tested as the permeation measurements are being made.

3.2 Other Measurement Techniques

3.2.1 Weight Loss Techniques

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 SAE J1527; they can be used to measure very small permeation rates, but they have limitations when rates are higher, or when temperatures are higher, because test fluid chemistry changes significantly during the test due to selective permeation of constituents. Also, these two techniques do not involve circulating fuel as the permeation is measured.

3.2.2 Sealed Housing Evaporative Determination (SHED) Units

One of the primary techniques for measurement of vehicle hydrocarbon emissions is the Sealed Housing (for) Evaporative Determination (SHED) test. This test uses an enclosed chamber that contains the item being tested (vehicle, system, or component). The environment inside the chamber 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). It is 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. Smaller SHED units (miniSHED and microSHED) are normally used to measure permeability of materials or smaller components and the performance of specific interfaces.

The SHED unit has some limitations when measuring permeation rates for components, especially when alcohol-blend fuels are involved.

- Average temperature for the typical diurnal cycles for the SHED procedures are actually very low (30 to 35 °C) compared to what is typically used in the procedure described in this document (up to 60 °C). Therefore, if the SHED detection technique is used, the test shall be conducted using a 24-hour isothermal temperature of either 40 °C or 60 °C.
- If alcohol-blend test fluids are used, the SHED detection technique (FID) does not account for the entire amount of alcohol that's present in the permeate. This is because the FID is calibrated using propane to give the response factor of 1.0 for propane. Other constituents in the SHED then have response factors less than or greater than 1.0. Most fuel hydrocarbons have a response factor that is close enough to 1.0 that this is a small error. However, alcohols such as ethanol have a response factor that is significantly less than 1.0. To compensate for this reduced response, a multiplication factor of 1.08 shall be used.

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 use test conditions that cause high permeation rates. The higher the permeation rate, the sooner equilibrium is achieved. The amount of time spent in the expensive permeation measurement unit can be shortened by preconditioning the specimen in a low-cost preconditioning unit. See 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 hundred hours for the equilibrium permeation rate to be achieved at the test temperature of 60 °C using alcohol-blend test fluids. If temperatures are lower than 60 °C, or if test fuels are "less active" than those containing alcohol, the overall time will be longer.

3.3.1.1 Testing at 60 °C

There has been a broad base of experience with measuring permeation rates at 60 °C using both ethanol-blend and methanol-blend fuels. How long it takes to reach true steady state depends on the rate of permeation of the specimen being tested. Table 1 was developed from experience of testing tubing.

**Table 1 - Estimate of time to achieve steady-state permeation
for tubing at 60 °C exposure
(g/m²/day)**

Measured Result for Methanol-Blend Test Fuels	Measured Result for Ethanol-Blend Test Fuels	Approximate Time Necessary to Achieve Steady-State Permeation Rate (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 is 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.

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 is not always a practical procedure because permeation rate can be at a steady-state level well before dimensional changes of the component being tested have ceased.

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 five successive points establish a straight line, steady state has been achieved. The slope of that line ($\text{g}/\text{m}^2/\text{day}$) 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 five successive measurements yield the same weight change, then the steady-state weight change per unit time is the equilibrium rate.

3.3.2.1.4 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 (see 5.3, 7.4, and Appendix A).

3.3.2.2 Three Practical Means to Determine Steady State

3.3.2.2.1 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 g/day , $\text{g}/\text{m}^2/\text{day}$, etc. (see 3.5 and 8.3). The calculated rate on two successive days should be used to determine a 2-day moving average (average of Day 1 and Day 2 measurements is first data point, the average of Day 2 and Day 3 measurements is second data point, and the average of next-to-last and last day's measurements is last data point). Once the moving 2-day averages 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 five 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.

EXAMPLE: An example of this is an absolute rate of $20 \text{ g}/\text{m}^2/\text{day}$ would have to show a 5-day least-squares plot of the five 2-day moving average points within $\pm 1 \text{ g}/\text{m}^2/\text{day}$ of being horizontal over the whole 5-day period.

3.3.2.2.2 An alternative means to measuring a steady state weight change value for permeation rate recognizes that there is an inherent scattering of test results that make it more difficult to get an indication of steady state. This involves the following steps:

- a. Precondition the part to be tested in the test fluid at 60 °C for 1000 hours.
- b. Keeping the part in contact with the test fluid, allow it to equilibrate at the test temperature for 1 week.
- c. Measure permeation rate in the designated test fluid and at the designated test temperature each day for a 5-day period.
- d. Value to be reported is an average of those five data points.

3.3.2.2.3 An alternative to measuring using either weight change method identified previously is the use of an Isothermal Mini or MicroSHED test. This involves the following steps:

- a. Precondition the part to be tested in the test fluid at 60 °C for 1000 hours.
- b. At the end of the preconditioning, remove the test sample from the recirculation apparatus and attach one end of the sample to a test vessel similar in design to what is specified in SAE J30. The other end of the sample should be sealed with a non-permeable cap or plug.
- c. This test vessel should then be filled with the test fluid to a minimum volume of 40%.
- d. Keeping the part in contact with the test fluid, allow it to equilibrate at the test temperature for 1 week.
- e. Conduct a 24-hour Isothermal Mini or MicroSHED test to measure the permeation rate in the designated test fluid and at the designated test temperature.
- f. The value to be reported is the result of one 24-hour SHED test.

NOTE: Testing using this method may also allow for the option of speciation of the permeate.

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 °C or 15 °C. The rate can increase even more when multilayer wall constructions are involved. For precise measurements of permeation, the temperatures of all parts of the apparatus and procedure must be controlled very closely throughout the full duration of the test. Close control of the fuel temperature is the most important aspect. Less variability will occur if there are other temperature controls in place as well. For example, a water jacket around the test cell to maintain its temperature and controlling the temperature of the carrier gas are two steps that also contribute to making the measurements more consistent and accurate.