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Test Method to Measure Fluid Permeation of Polymeric Materials by Speciation

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1. SCOPE

This test method described in this document covers a procedure to speciate that is, to determine the amounts of each different fuel constituent that permeates across sheets, films or slabs of plastic materials. One side of the sheet is meant to be in contact with either a liquid test fuel or a saturated test fuel vapor, the other side is meant to be exposed to an environment free of fuel. The test fuel can either be a mixture of a small (usually smaller than ten) number of hydrocarbon, alcohol and ether constituents or it can be a sample of a real automotive fuel, e.g., one that may contain hundreds of different constituents.

Furthermore, Appendix A contains guidelines to speciate evaporative emissions from finished fuel system components such as fuel lines, fuel filler pipes, fuel sender units, connectors and valves.

2. REFERENCES

2.1 Applicable Publications

The following publications form a part of this specification to the extent specified herein. Unless otherwise indicated, the latest version 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 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
SAE J1737	Test Procedure to Determine the Hydrocarbon Losses from Fuel Tubes, Hoses, Fittings, and Fuel Line Assemblies by Recirculation
SAE Paper 981360	Fuel Permeation Performance of Polymeric Materials Analyzed by Gas Chromatography and Sorption Technique
SAE Paper 981376	Speciation of Evaporative Emission from Plastic Fuel Tanks
SAE Paper 1999-01-0376	Fuel Permeation Analysis Method Correlation
SAE Paper 1999-01-0377	Vapor and Liquid Composition Differences Resulting from Fuel Evaporation
SAE Paper 1999-01-0380	A Comparison of Vapor and Liquid Fuel Permeation of Fuel Systems Polymers
SAE Paper 2001-01-1999	Fuel Permeation Performance of Polymeric Materials
SAE Paper 2002-01-0635	Comparison of Fuel Hose SHED Test Results and Predicted Values Using Fundamental Material Barrier Properties

2.1.2 ASTM Publications

Available from ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959, Tel: 610-832-9585, www.astm.org

ASTM E 96	Test Method for Water Transmission of Materials
ASTM D 5134	Standard Test Method for Detailed Analysis of Petroleum Naphtas through n-Nonane by Capillary Gas Chromatography

2.1.3 Federal Regulations

Available from the Document Automation and Production Service (DAPS), Building 4/D, 700 Robbins Avenue, Philadelphia, PA 19111-5094, Tel: 215-697-6257, <http://assist.daps.dla.mil/quicksearch/>.

U.S. Code of Federal Regulations, 40 CFR, Chapter 1, Subpart B - §86.101 to 86.157

2.1.4 General Scientific Literature Publication

J.T. Scanton and D.E. Willis, "Calculation of Flame Ionization Detector Relative Response Factors Using the Effective Carbon Number Concept", J.of Chromatographic Science. Volume 23, August, 1985

Jrn of Chromatographic Science P. O. Box 48312, Niles IL 60648

2.2 Related Publication

The following publication is provided for information purposes only and are not a required part of this SAE Technical Report.

2.2.1 SAE Publication

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

SAE J2260 Nonmetallic Fuel System Tubing with One or More Layers

3. DEFINITIONS

3.1 FLUX

The flux, $F_i(l)$, of a specific fuel constituent i (across a sheet, film or slab of thickness l) is defined as the rate of flow (mass per unit time) of the specified constituent in the direction normal to the face of the sheet, through a unit area of the sheet. An accepted measure of $F_i(l)$ is $\text{gm}/\text{m}^2\text{day}$. The test conditions (test fuel composition, temperature, pressure, etc.) must be stated. Eventually in the course of a permeation test a steady state value, $F_i^{(ss)}(l)$, of the flux, $F_i(l)$, should be reached for each constituent (see 3.3).

3.2 VAPOR TRANSMISSION RATE

The vapor transmission rate, VTR_i , of a specific fuel constituent (across a sheet, film or slab) is defined as the steady state rate of flow (mass per unit time) of the specified constituent in the direction normal to the face of sheet, through a unit area and for a unit sheet thickness under the conditions of the test. An accepted measure of VTR_i is $\text{gm mm}/\text{m}^2\text{day}$. The test conditions must be stated. The steady state flux, $F_i^{(ss)}(l)$, and the vapor transmission rate, VTR_i , are related by $VTR_i = l * F_i^{(ss)}(l)$, where l is the film thickness.

3.3 MEASURING STEADY STATE CONDITIONS

When permeation across a plastic film or sheet is measured both the amount of permeate and its composition depend on the time t that has elapsed since the film or sheet was first exposed to the fuel: in other words, each flux $F_i(l)$, is a different function of t . Eventually, a steady state rate of permeation should be attained for each fuel constituent. In other words, for sufficiently long t , each flux $F_i(l)$ should attain a constant (steady state) value, $F_i^{(ss)}(l)$. Of course, this can only be possible if the composition of the test fuel does not change significantly in the course of the experiment. The time required to arrive at steady state is inversely proportional to the square of the film (or sheet) thickness and it varies greatly depending on:

- The material tested
- The composition of the test fuel
- The temperature of the experiment

For the purposes of the present test it is crucial that the steady state permeation rates (as opposed to the transient permeation rates detected prior to the attainment of steady state) be reported.

3.3.1 Operational Criteria for Steady State

An operational definition of steady state permeation may be as follows:

- 3.3.1.1 In the case of a test fuel constituent i , for which the flux $F_i(l)$ has been determined to be non zero (e.g., above the experimental quantitation limit), $F_i(l)$ will be considered to have attained steady state at time t , provided that (within the precision of the experiment) it has remained constant for an interval of time Δt greater than or equal to $t/4$.
- 3.3.1.2 In the case of a test fuel constituent i , for which the flux $F_i(l)$ has remained below the experimental quantitation limit throughout the entire time t over which testing has been conducted, it is not possible to determine the steady state permeation value from the test alone. In this case a null result should be reported together with the time t over which it has been observed. Repeating the experiment with a thinner film of the same material may yield a non null result.

In using the previous operational definition of steady state it is important to keep in mind that, in general, when a plastic material is exposed to a permeant (such as a test fuel), permeant is taken up by that material and the material may undergo significant changes as a result. These changes may include deformations due to swelling, changes in the material elastic properties, plasticization, changes in crystallinity and depletion of additives. All these changes affect how steady state is reached: an understanding of the changes that are likely to occur for the material and the test fuel at hand is important in assessing the test results.

3.4 TEST CONDITIONS

In order for the permeation rates measured according to the present procedure to be reproducible—a necessary prerequisite for reliability and ultimate usefulness—the following conditions under which the test procedure takes place must be carefully controlled.

3.4.1 Temperature

Permeation rates are known to be greatly affected by temperature, therefore test results should state both the test temperature and its standard deviation. Permeation rates usually increase with temperature and a rule of thumb of a 10% increase in permeation per degree centigrade is often quoted. However, the rate of change of permeation with temperature depends on both the material and the test fuel. For example, if the temperature of the experiment is close to the glass transition temperature of the material being tested, a much larger rate of permeation increase with temperature is to be expected.

3.4.2 Test Fuel Composition

The value of the flux $F_i(l)$ of a given fuel constituent is a function of the composition of the test fuel. Therefore it is crucial to insure that the composition of the fuel does not change significantly in the course of the experiment. This can be achieved either by using a quantity of fuel much larger than the amount likely to be lost by permeation and evaporation in the course of the test or by periodically replenishing the test fuel. SAE procedure J1681 defines a number of “standard test fluids”.

3.4.3 Pressure

External pressure itself has a negligible effect on permeation, since it has a negligible effect on the chemical potential of the permeants. However, pressure differentials between the two sides of the test sheet may cause it to stretch and/or warp and thus influence the results of a permeation test. Furthermore, the saturated vapor pressure of the permeant strongly depends on temperature: this vapor pressure contributes to the total pressure at which the test fuel is held and may also cause deformations (stretching or warping) of the test sheet if the temperature is changed. In order to reduce deformations as much as possible, it is necessary to insure that the total pressure on the two sides of the film is maintained at comparable levels. For example, this can be achieved by venting the test fuel to the outside or by minimizing changes in temperature after the film has been mounted. Clearly, for a given material, the thinner the test film the greater the chances for potentially damaging elastic deformations. Conversely a sufficiently thick test sample will be able to withstand a higher pressure differential between the two sides.

3.4.4 Materials

It is important to keep in mind that both the processing conditions (e.g., cast versus blow molded film) and the additive package may have significant effects on permeation. Thus, it should not be surprising that films of the same polymer obtained from different manufacturers exhibit somewhat different permeation characteristics when exposed to the same test fuel.

3.4.5 Liquid versus Vapor Exposure

Standard thermodynamic considerations imply that the permeation rates for films in direct contact with liquid permeant or with its saturated vapor should be the same. While there have been a large number of experimental observations confirming this rule (see SAE papers 981360 and 2001-01-1999), one observation has also been reported where weight loss appears to depend on whether the plastic specimen is exposed to liquid or vapor (see: SAE Paper 1999-01-0380). In this case, it is possible that, the liquid in contact with the plastic material is more effective than its vapor in depleting certain additives from that material, since the presence of the liquid favors convective transport away from the surface of additives leached out of the material. This may hold in particular for additives having relatively high molecular weight that are soluble in the liquid.

Concerning the issue of liquid versus vapor exposure it should be noted that, in the case of liquid exposure, the test film has to support the weight of the fuel. As a result, if the film is very thin, e.g., below 50 microns, it may easily become stretched or warped. This in turn may invalidate the results of the test.

3.4.6 Moisture Content and Relative Humidity

Both the moisture content of the fuel and the relative humidity experienced by the test film (moisture content of the purge gas) may affect permeation rates. Such materials as EVOH and nylons are known to be particularly sensitive to the presence of water; however, for these materials the presence of alcohols in the "test fuels" may overwhelm any effect of this kind. To make consistent comparisons between the results of different tests, the moisture content of the fuel used should either be:

- a. Kept to a minimum
- b. Controlled to a pre-agreed level
- c. Measured and reported

(listed in order of preference).

3.4.7 Film Thickness

Theoretical considerations suggest that the steady state flux, $F_i^{(ss)}(l)$, across a film of thickness l is inversely proportional to the thickness: the definition of vapor transmission rate VTR_i is based on this rule. However, this is based on the assumption that the material is uniform through the thickness of the sample. Actual samples may deviate from this assumption. For example, processing conditions are often such that the two surfaces of a film have material properties (crystallinity, local concentration of additives) significantly different from those of the bulk. As a result, for very thin films (typically 50 microns or less) where the surface material accounts for a significant fraction of the overall thickness, the observed values of the steady state flux, $F_i^{(ss)}(l)$, may depart from the rule stated previously.

Furthermore, it should be kept in mind that an absolute variation in thickness (for example, thickness specified to within ± 2 microns) represents a much greater uncertainty in percentage terms for a thin film than for a thick one.