SAE International™

SURFACE VEHICLE RECOMMENDED PRACTICE

J2665

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Test Procedure to Measure the Fuel Permeability of Materials by the Cup Weight Loss Method

RATIONALE

Not applicable.

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1. SCOPE

This test standard covers the procedure for measuring the permeation of fuel or fuel surrogates through test samples of elastomeric, plastic or composite materials, up to about 3 mm thick.

The method involves filling a test cup with the test fluid (fuel or fuel surrogate), sealing test sample over the open end of the cup, and then placing the sealed container into an oven at the desired test temperature and measuring the weight loss over time. Permeation rates are calculated from the rate of weight loss and the exposed area of the test sample.

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Standard permeation test temperatures are 40 °C and 60 °C. Standard test fluids are Fuel C, Fuel CE10 and Fuel CM15. Other fluids, such as Fuel CMTBE15, and other volatile liquids may be tested according to this procedure as desired (SAE J1681). The method is not applicable for measuring permeation of higher boiling materials that will not completely evaporate from the exterior surface of the sample at the test temperature.

2. REFERENCES

2.1 Applicable Publication

The following publication forms 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, 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 J1681 Gasoline, Alcohol and Diesel Fuel Surrogates for Materials Testing

SAE J2659 Test Method to Measure Fluid Permeation of Polymeric Materials by Speciation

2.2 Related Publications

The following publications are provided for information purposes only and are not a required part of this document.

2.2.1 ASTM Publications

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

ASTM D 814-86 Standard Test Method for Rubber Property-Vapor Transmission of Volatile Liquids

ASTM E 96-95 Standard Test Methods for Water Vapor Transmission of Materials

3. USEFULNESS AND LIMITATIONS

The cup method, when used in accordance with the guidelines presented in the procedure sections of this method, can be an easy, effective, and relatively inexpensive screening technique for determining the relative permeability of plastics, elastomers and composites:

a. The method is useful to establish the permeation rate of a given fluid and material.

b. The cup method is a useful tool for distinguishing between materials that have a significant difference in their resistance to permeation.

c. It is most accurate when the test fluid is a pure liquid or a liquid mixture for which changes in composition do not significantly affect the measured permeation rate.

The method does have some limitations:

a. The permeation rate of individual components of a fuel mixture through the test material cannot be determined; if this is desired, see SAE J2659.

b. The method should be used with caution when the components of a fuel mixture have widely different permeation rates: the high permeating components can be depleted from the mixture before the test is completed.

c. For low permeation rates, the weight change is small so that accuracy can be a problem. In addition, the time required to obtain measurable weight changes can be very long.

d. For highly permeable materials, the rapid loss of fluid may prevent the attainment of steady state before all the fluid has permeated the test film. The fuel composition of a mixed fuel may also change significantly and possibly provide erroneous results.

e. The following is a list of potential sources of error that should be minimized as much as possible:

Weighing accuracy
Leakage around seals
Fuel depletion
Temperature control
Film thickness variation
Pin holes in films
Film distortion
Change in test fluid composition

These sources of error should be reviewed and good experimental techniques employed to minimize the sources of error. Examples of these techniques are described in the method.

4. SAFETY

This method is intended for measuring permeation of potentially toxic and/or flammable liquids at elevated temperatures. Each laboratory is responsible for assuring that this method is run in a safe manner according to its internal safety regulations and practices.

5. APPARATUS

5.1 Permeation cups should be Thwing-Albert permeation cups (Vapometer Model 68 available from Thwing-Albert Instrument Company, 10960 Dutton Road, Philadelphia, PA 19154, website: www.thwingalbert.com) (shown in Figures 1 and 2) or equivalent. The permeation cups used shall be leak-tight when assembled with a blank (see 5.5). The Model 68 Thwing-Albert cups, with depth of 50.8 mm, shall be referred to as “TA” cups in this document.

NOTE: If T-A cups are used, they should be modified as follows (a) the supplied neoprene gaskets replaced with FKM gaskets (see Section 5.4) and (b) the six supplied knurled head screws modified or replaced to allow for torque wrench tightening (see Sections 5.7 and 8.9).

FIGURE 1 - MODIFIED THWING-ALBERT CUPS: ON THE LEFT, IT IS SHOWN DISASSEMBLED WITH RETAINER RING, SCREWS, MESH, FKM GASKETS AND CUP; ON THE RIGHT IT IS SHOWN FULLY ASSEMBLED

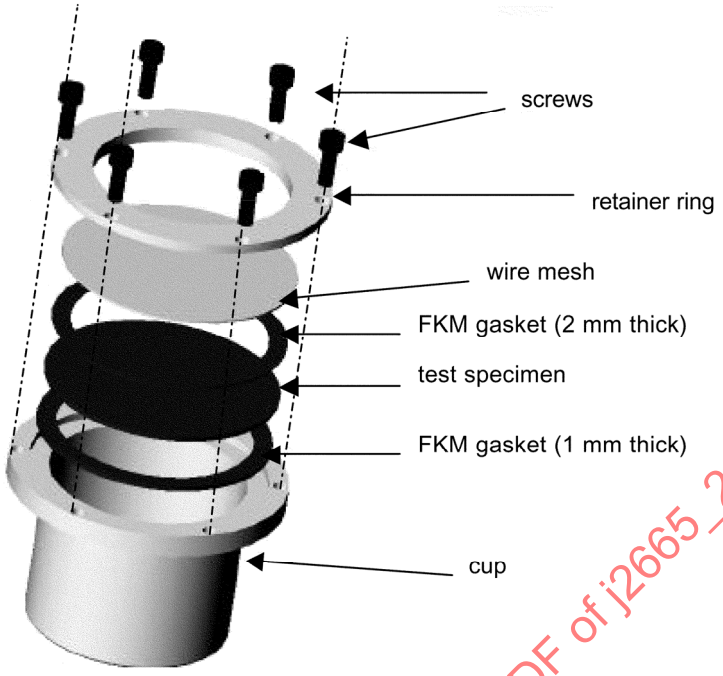

FIGURE 2 - EXPLODED VIEW OF THE MODIFIED THWING-ALBERT PERMEATION CUP, SHOWING THE ASSEMBLY SEQUENCE OF THE COMPONENTS

5.2 Analytical balance should be capable of measuring to 0.0001 gram (e.g., Mettler AT400 or equivalent). Balances with lower precision may be used, as long as the weight loss between measurements exceeds the precision of the balance by at least 10 times.

5.3 Explosion proof oven or similar constant temperature device capable of holding the filled cups and maintaining their temperature within ±1 °C of the desired test temperature. There shall be sufficient air circulation in the oven/constant temperature chamber to allow dilution and purging of the permeating fuel.

5.4 Two fluoroelastomer gaskets made from FKM. It is recommended that an FKM with a minimum fluorine content of 70% for TFE/VF_2/HFP polymers and a Shore A hardness of 55 to 70 be used. Nominal FKM gasket sizes for the Thwing-Albert cups are 63.5 mm by 76.2 mm by 1 and 2 mm (ID x OD x thickness) for the two gaskets.

5.5 Two metal blanks cut to an appropriate diameter to fit the permeation cups (76 mm in diameter for the Thwing-Albert cups) and about 0.5 mm thick. Aluminum or stainless steel should be used.

5.6 Micrometer capable of measuring sample thickness to 0.0025 mm (0.0001 in).

5.7 Torque wrench, for tightening cup screws, capable of measuring up to 1.1 N-m (10 lbf-in) torque.

5.8 Wire mesh for external sample support (should be 16 mesh, gauge of 1.5 mm (1/16 in)) cut to an appropriate diameter to fit the permeation cups (76 mm in diameter for the Thwing-Albert cup).

6. TEST FUELS

Examples of standard test fluids (fuels and fuel surrogates) and their method of preparation are given in SAE J1681. Other fuels, such as Fuel CM15, and other liquids may be tested according to this procedure as desired.

7. SAMPLE PREPARATION

With the cup method, as with all methods for measuring the permeability of a given material or composite, there are several issues that must be addressed with respect to the preparation of the sample to be tested:

7.1 Ideally, the sample should be fabricated by the same method as will be used to make the final part. This is to ensure the same morphology for the sample and the final part. The morphology is very important as it can effect the permeation rate.

7.2 For many plastic applications injection molded parts are used. The injection molding of very thin films may be a problem for some polymers. (1) The making of good thin films that are uniform and pinhole free by injection molding is difficult and (2) the thicker the film, the longer the required test time.

7.3 For materials that rely on crystallinity to provide the barrier, the sample thickness should be close to the intended use thickness. This is to ensure that the level of crystallinity is representative.

7.4 In composite constructions, the relative position of the layers should be representative of the final part.

8. PROCEDURE

8.1 Die cut the sample to fit the permeation cup (76.2 mm OD for T-A cups). If the sample is a composite material, label the sample, noting which side is to be the fuel contact side.

8.2 Measure the thickness of the sample to 0.0025 mm in 5 locations (once in each quadrant and once in the center of the sample). Average the measurements and record this value as the sample thickness (t) in mm. Measured sample thickness values must all be within 10% of the average thickness, t.

8.3 Weigh and record weight (C_1) of the empty cup, sample, gasket(s), etc., to the nearest 0.0001 g.

8.4 Fill the permeation cup approximately 7/8 full with the test fuel (about 150 ml for T-A cups).

8.5 Assemble two cups per material to be tested as follows:

8.5.1 Place a 1 mm FKM gasket in the groove of the cup (optional for materials with Shore A < 85).

8.5.2 Place the sample (fuel contact side down) on the gasket.

8.5.3 Place a 2 mm FKM gasket above the sample.

8.5.4 Position the wire mesh support above this gasket.

8.5.5 Put the cup retainer ring onto the cup.

8.5.6 Align the ring, put in the screws but do not tighten.

8.6 Record the weight (W_1) of the assembled cup to the nearest 0.0001g.

8.7 Repeat steps 8.3 to 8.5 for two blank cups where the metal plate is substituted for a sample.

8.8 Place the filled sample cups in the oven, right side up for one hour in order to come to equilibrium temperature. Further loosen the screws, if necessary, to relieve any built up pressure in the cup.

8.9 Finger tighten screws in a triangle form 1,3,5 then 2,4,6. Use a torque wrench to further tighten the screws in the same order to about 0.25 Nm (2 lbf -in), then firmly tighten using a torque wrench to 0.57 to 1.1 Nm (5 to 10 lbf -in). It is suggested that the screws be retighten after the first weighing and after 1 week. Note that if lower durometer gaskets are used than recommended in 5.4, torque levels may have to be lowered to avoid damaging the gaskets.

8.10 Weigh the samples and record weight (W_2) and time.

$$\frac{(W_2 - W_1)\times 100}{W_1 - C_1} \qquad \text{(Eq.1)}$$

8.11 Calculate the % weight change from Equation 1. If the weight change is greater than 5% then the weight loss on equilibration is too high and the cup assembly procedure must be restarted.

8.12 Place the samples back in the oven. If permeation is to be measured on the vapor phase, place the samples right side up. If it is to be measured on the liquid phase, place the samples upside down. (Safety note: it is strongly recommended that the samples be placed in a secondary container of sufficient size to contain all of the fuel in case of a leak or spill.)

8.13 Periodically remove the samples (and blanks) from the oven and record their weight and time. Frequency of weighing depends on permeation rate with more frequent weighing required for higher permeating materials. Frequency should be between once a day and twice a week.

9. PLOTTING THE WEIGHT LOSS WITH TIME

When rates of permeation are measured, the initial performance of a material may not be the same as the eventual equilibrium value. It takes a certain amount of time for the migration of the fuel to achieve its steady-state rate after first exposure to the fuel. This is particularly true if the material relies on crystallinity to provide its barrier properties.

The best way to judge whether the permeation rate has reached steady state is to plot the weight loss with time. The following outlines the steps:

9.1 Determine the average weight, B, of the blanks (B = average of B_1 and B_2) at each measurement time and subtract from the measured sample weights. These will be called the corrected sample weights. This will correct for losses through the gasket and for weighing errors due to buoyancy and other effects.

9.2 Plot the corrected sample weights (in grams) against the sampling time (days). The sampling time should be in days, where day = 0 for the first corrected sample weight.

9.3 A plot such as that shown in Figure 3 should be obtained. Note that not all experiments show the fuel depletion and equilibration parts of the plot. This depends on how fast equilibration takes place and duration of the experiment.

9.4 If the plot shows a steady state linear permeation consisting of at least 10 measured consecutive points, proceed to the next section to calculate the steady state flux of the material.

10. CALCULATION OF STEADY STATE FLUX (F)

The "steady state flux" of a sample is sometimes also refered to as "normalized permeation rate" or "GMD" for "grams per meter squared per day". To be consistent with other SAE documents (SAE J2659) and general scientific convention, the term "steady state flux" will be used in this document.

10.1 To calculate the sample steady state flux, perform a linear regression calculation using at least 10 consecutive steady state values from the data of corrected cup weights versus. time. The equilibration and fuel depletion data points must not be included (see Figure 3).