



# SURFACE VEHICLE RECOMMENDED PRACTICE

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Methods for Determining Physical Properties of Polymeric Materials Exposed to Hydrocarbon Fuels or Their Surrogates and Their Blends with Oxygenated Additives

## RATIONALE

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## 1. SCOPE

This SAE Recommended Practice applies to determining worst-case fuel or test fluid surrogate, conditioning test specimens in worst-case fuel(s)/surrogate(s) prior to testing, individual tests for properties of polymeric materials exposed to oxygenate fuel/surrogate mixtures with additives. The determination of equilibrium, as well as typical calculations are also covered.

### 1.1 Purpose

Polymeric materials are used in applications that require exposure to a variety of fluid environments. Tests to determine the effects of such exposure on material properties are well established. However, the determination of the effects on polymeric materials exposed to fuels of variable blends with assorted oxygenates poses new problems. This document seeks to address those concerns by detailing changes to standard tests that make them suitable for that purpose.

## 2. REFERENCES

### 2.1 Applicable Publications

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

### 2.1.1 SAE 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](http://www.sae.org).

|           |                                                                                                                                |
|-----------|--------------------------------------------------------------------------------------------------------------------------------|
| 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 J2659 | Test Method to Measure Fluid Permeation of Polymeric Materials by Speciation                                                   |
| SAE J2663 | Test Procedure to Measure Permeation of Elastomeric Hose or Tube by Weight Loss                                                |
| SAE J2665 | Test Procedure to Measure the Fuel Permeability of Materials by the Cup Weight Loss Method                                     |

### 2.1.2 ASTM Publications

Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, Tel: 610-832-9585, [www.astm.org](http://www.astm.org).

|             |                                                                                                                             |
|-------------|-----------------------------------------------------------------------------------------------------------------------------|
| ASTM D 412  | Test Method for Rubber Properties in Tension (ISO 37)                                                                       |
| ASTM D 413  | Test Method for Rubber Property—Adhesion to Flexible Substrates (Peel Test for Fabric Adhesion) (ISO 36)                    |
| ASTM D 429  | Test Methods for Rubber Property—Adhesion to Rigid Substrates (Metal adhesion, Using Method A, Button) (ISO 813 or ISO 814) |
| ASTM D 471  | Test Method for Rubber Property—Effect of Liquids (ISO 1817)                                                                |
| ASTM D 543  | Test Method for Resistance of Plastics to Chemical Reagents (No ISO Found)                                                  |
| ASTM D 618  | Practice for Conditioning Plastics and Electrical Insulating Materials for Testing, Procedure A (ISO 291)                   |
| ASTM D 624  | Test Method for Tear Strength of Conventional Vulcanized Rubber and Thermoplastic Elastomers (ISO 34 Part 1)                |
| ASTM D 638  | Test Method for Tensile Properties of Plastics (ISO 527 Parts 1 and 2)                                                      |
| ASTM D 790  | Test Method for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials (ISO 178)   |
| ASTM D 991  | Test Method for Rubber Property—Volume Resistivity of Electrically Conductive and Anti-Static Products (No ISO Found)       |
| ASTM D 1053 | Test Method for Rubber Property Stiffening at Low Temperatures; Flexible Polymers and Coated Fabrics (ISO 1432)             |
| ASTM D 1329 | Test Method for Evaluating Rubber Property—Retraction at Low Temperature (TR Test) (ISO 2921)                               |
| ASTM D 2240 | Test Method for Rubber Property—Durometer Hardness (ISO 868)                                                                |

### 3. REQUIREMENTS

#### 3.1 General

A problem arises in testing materials for use in vehicles that can run on fuels with oxygenate concentrations varying from 0 to 85% by volume. There is no single oxygenate-containing test fuel composition that can be used to evaluate the relative performance of different polymeric materials.

NOTE: Polymeric materials include plastics and elastomers.

Because polymeric materials differ in their molecular structure, the most damaging concentration of oxygenate for one material may not be the most damaging for another. This problem has necessitated the designation of a series of test fluids that can be used to compare the properties of one polymeric material in its "worst-case" fuel with those of another polymeric material in a different "worst-case" fuel. Nevertheless, it is desirable to reduce the total number of tests as much as possible. Therefore, a single test, using weight gain at equilibrium, has been chosen to determine which fuel composition (from Table 1) exerts the greatest effect on each polymeric material. Once a polymeric material's "worst-case" fuel has been determined, its properties will be measured after exposure to that fuel.

Since the rate of diffusion in plastics (glassy polymers) can be very slow, the time required for some plastics to reach equilibrium may be quite lengthy. In the simplest cases of diffusion with polymeric materials, the behavior may be described by Fick's Law. For Fickian diffusion, the time required to attain equilibrium sorption for a polymer slab or sheet exposed to a penetrant is proportional to the square of the sample thickness. Thus, the time does not increase linearly, but exponentially, as the thickness increases. However, for plastics exposed to organic liquids, the diffusion coefficient typically is not constant, may be because of non-uniformity, boundary conditions, physical effects (burrs, nicks, folds, etc.), and swelling of the sample periphery may also occur. This leads to non-Fickian or anomalous diffusion and the effect of sample thickness on the equilibrium time is not as straightforward as for the ideal Fickian case; but, in either case, as the sample thickness increases, so does the time required to achieve sorption equilibrium. Therefore, to shorten conditioning times, sample thickness should be as small as possible and still be consistent with the requirements of the test for a particular physical property.

This document consists of procedures that are designed to evaluate the performance of polymeric materials when they are exposed to variable oxygenate content fuels. The first part of this document addresses the determination of the worst-case fuel. The second part contains the procedure for conditioning actual test specimens in fuel, prior to measuring their physical properties. Finally, the third part details the test methods for evaluating polymeric materials for use in oxygenate fuels.

The methodology presented is general enough that it can be extended to determine the physical properties of polymeric materials in gasoline mixtures with most oxygenate additives such as methanol, ethanol, MTBE, ETBE, TAME, EAME, etc., or Diesel fuel with bio-diesel additives (Fatty Acid Methyl Esters or FAME). These additives are not to be used above the maximum regulated percent of the specific additive.

### 4. DETERMINING WORST-CASE FUEL

#### 4.1 Equilibrium Weight Increase

To compare the extent of polymer swelling by the test fluids given in SAE J1681 paragraph 7.1.4, the polymeric material must be allowed to reach equilibrium with each fuel. Thus, the samples will be immersed in the test fuels until they attain a constant weight, as set forth in Appendix A.

#### 4.1.1 Sample Preparation

To reduce the amount of time required to complete the test, plastic samples used for this procedure will be thin films produced under a nitrogen blanket by hot-plate forming or other similar method. Note should be taken that the effect of fuel on very thin hot-plate formed films can vary significantly from the effect of fuel mixture on injection molded, or extruded specimens. Samples will have a minimum surface area of 12 cm<sup>2</sup> and will be made as thin as possible. Thicker specimens can be used but they will require much longer equilibration times. After thirty test specimens have been marked for identification with an engraver or punch, precondition them at 23 °C ± 2 °C and 50% ± 5% relative humidity for a minimum of 40 hours according to ASTM D 618, Procedure A. For rubber, mark twenty-five ASTM D 471 volume change test specimens with a punch, for identification.

#### 4.1.2 Conditioning the Samples

Weigh the specimens individually, in grams, to 4 decimal places. Place 6 plastic specimens (only 5 for elastomers) into each of 6 glass vessels furnished with a reflux apparatus, as specified in ASTM D 543, and equipped with a calcium sulfate drying tube. The sixth, specially marked plastic sample is to be used to evaluate long-term chemical effects on the plastic and should not be removed from the hot fuel when the other 5 samples are weighed. Samples should be separated using stainless steel wire and glass beads according to ASTM D 471. Fill each vessel with a different oxygenate blend test fuel such that all specimens are completely submerged. Fuel formulations are provided in SAE J1681. Allow at least 1 cm of fuel above the plastic samples to compensate for any evaporation. For elastomers, allow at least 3 cm fuel above the samples to compensate for swelling as well as evaporation. Using a water or sand bath, heat the test fuel uniformly in the vessel to 55 °C ± 2 °C. Do not use heating mantles or hot plates. For elastomers, replace the test solution daily for the first three days and weekly thereafter. For plastics containing plasticizer, replace the solution twice each week for the duration of the exposure. For other plastics, replace the fuel mixtures weekly. Continue sample exposure until constant weight is attained, or for a minimum of 500 hours. Observe safety precautions applicable to the handling of flammable and toxic mixtures (refer to OSHA and local laboratory procedures and standards).

#### 4.1.3 Weighing the Samples During Conditioning

Except for special situations, applicable to the second part as follows (conditioning in the worst-case fuel), weigh each sample at one week intervals to determine if absorption of the fuel has stabilized. Remove the samples to be weighed from the vessel containing the hot fuel and place them in a container of the same fuel at room temperature. Let the samples stand in the fuel for a period of 0.5 to 1 hour. Weigh a weighing bottle, in grams, to 4 decimal places. Remove one sample from the cool fuel, blot it, drop it into the weighing bottle and seal it within 10 seconds to prevent any further weight loss due to evaporation. Weigh the bottle containing the sample and record the weight of the sample (obtained by difference from the tared bottle). Repeat this procedure for the other five samples (and for each fuel blend). If there is no change in Apparent Percent Weight Increase for each sample in a particular fuel (as defined in Appendix A) for three consecutive weighings, the five samples are to be removed for drying. The exposure of the sixth plastic sample in each set is to continue until the worst-case fuel has been determined, at which time exposure will be continued only for the sample in that fuel. (See Equation 1.)

$$\text{Apparent Percent Weight Increase} = \frac{[(\text{Wt of bottle} + \text{sample}) - \text{wt of bottle} - \text{Original wt}]}{\text{Original weight}} \times 100 \quad (\text{Eq. 1})$$

Once it has been determined that equilibrium has been reached, the weights for each sample for the last three weighing periods will be averaged. This value will be termed the Equilibrium Swollen Weight for that sample.

NOTE: The time necessary for the samples to reach equilibrium may vary from one test fuel to another. Therefore, exposure to a test fuel should continue until the statistical treatment of successive Apparent Percent Weight Increase determinations indicate that equilibrium has been attained in that fuel. It might be expected that samples in the worst-case fuel will reach equilibrium more rapidly than in any other. However, this assumes that there is only one mechanism involved in the absorption process, which might not be the case.