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# **A Rational Approach to Qualifying Materials for Use in Fuel Systems**

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## ABSTRACT

About 10 years ago in the US, an automotive OEM consortium formed the Oxygenated Fuels Task Force which in turn created the SAE Cooperative Research Project Group 2 to develop a simple rational method for qualifying materials. At that time the focus was Methanol/Gasoline blends. This work resulted in SAE J1681, Gasoline/Methanol Mixtures for Materials Testing. Recently this document was rewritten to make it the single, worldwide, generic source for fuel system test fluids. The paper will describe the rationale for selecting the fuel surrogate fluids and why this new SAE standard should replace all existing test fuel or test fluid standards for fuel system materials testing.

## INTRODUCTION

Fuel system engineers have always struggled with the task of assuring that materials used for fuel system components will survive in the fuel environment. The uncontrolled variability of fuels in the marketplace has been a major obstacle to the engineer's quest for certainty that his design will work. The situation today is made worse as environmental pressure drives the development of a wider variety of "clean" fuels. Added to the engineers task is the public's growing desire for vehicles that will last 10, 15, perhaps 20 years. Furthermore, during that vehicle lifetime, newly implemented evaporative emission regulations dictate that it is unacceptable for the fuel system to develop

leaks or for emissions critical the components to stop working.

Over the past 30 years, commercial and governmental standards groups, automotive OEMs, fuel system component manufacturers and others have developed test fuel recipes they used to determine if a given material would be acceptable for use in the fuel system. Some were based on actual fuels, others were simple mixtures of the most aggressive components of fuel. In this state of affairs a material or component manufacturer might have to test in 10 or 20 similar but different test fluids in order to satisfy all customers. Creating or responding to materials specifications was a nightmare. This is illustrated in Appendix A, which presents a comparison of many current test fuels and laboratory test fluids used in the automotive industry.

While the situation portrayed in Appendix A may appear bleak, that table is organized to show the main categories of chemicals in fuels. In this way one may see how similar the fuels actually are as well as where the differences exist.

## DEVELOPING RATIONAL FUELS

The task of rationalizing test fuels began within the SAE Fuel System Forum Committee with the formation of the SAE J1681 Task Force. Its assignment was to take the existing document that dealt exclusively with gasoline/methanol mixtures and convert it to a document that would use the same rationale for test fluids and test fuel recipes but be inclusive of all available types of fuels.

**THEORETICAL BASIS** – Automotive fuels are made from many compounds that are derived from petroleum, natural gas and biomass. Petroleum derived fuels consist of all hydrocarbons compounds that fall within certain broad ranges of boiling point limits that can be derived from any given crude oil feedstock. For example, gasoline is typically C<sub>4</sub> through C<sub>10</sub> hydrocarbons having a boiling range between –40°C to +180°C and diesel fuel is typically C<sub>12</sub> through C<sub>25</sub> hydrocarbons having a boiling range between +35°C to +250°C. There can be up to 400 specific molecules that occur in commercial fuels and the exact makeup of a given liter of fuel can vary substantially between manufacturers, batches, seasons and geographic location.(1, 2)

While this situation may seem chaotic, as the table headings in Appendix A suggest, the multitude of fuel chemicals fall into a few broad classes namely: Alkanes, Olefins, Aromatics, Oxygenates and aggressive impurities. By knowing the characteristics of each class and selecting individual members whose properties are either representative of the mean of the population or worst case, one can construct a simplified test fluid which can create the effects typical of the complex commercial fuel.

This is exactly what is done to determine the “octane rating” for an engine. Blends of Iso-Octane (100 octane) and normal Heptane (zero octane) tested in the engine to establish the “octane” value at which detonation is first detected.(3) This rating, using only two specific chemical species, accurately predicts the engine performance of commercial fuels with all their variability in chemical make up. The same can be done for the effect of fuels on materials.

The steps in this process are:

- Understand the potential effects of each class of fuel chemicals or impurities on materials
- Select compounds from each class that are representative of fuels in the marketplace and create worst-case effects on materials.
- Create specific mixtures of these compounds to evaluate specific effects on material.

## UNDERSTANDING FUEL CHEMICALS

**Alkanes** – The alkane component group includes straight chain, branched chain and cyclic aliphatic hydrocarbons. The alkanes are often referred to as paraffins and are derived from the naphtha fractions from a refinery or gas processing facility. This class of compounds makes up about 40% to 70% of gasoline and about 60% to 70% of diesel fuel (2).

Alkanes are non-polar solvents. Polymeric materials, especially non-polar polymers, can sorb alkanes, which can in turn cause swelling. Extensive earlier work on rubber materials was performed using the alkane isooctane (2,2,4 - trimethyl pentane)(4)(5).

**Olefins** – This component group is very similar to the alkanes except that they contain unsaturated sites (double bonds between carbon atoms). They comprise about 5% to 30% of gasoline but less than 3% of diesel fuel (2).

The sorption effects of olefins on polymeric materials are also similar to the alkanes. Their main adverse effect on fuel systems and materials is the ease with which they can degrade by oxidation at the double bond sites. The products of this degradation are gums, varnishes and peroxides. The effects of the olefins in fuel are represented by the addition of peroxide contaminant in aggressive test fluid recipes, which will be discussed more fully later.

**Aromatics** – The aromatics group includes molecules based on the six-member ring compound benzene, and substituted benzenes, such as toluene, and xylenes. The aromatics also include molecules containing additional carbons in various configurations and positions on a benzene molecule. Aromatic compounds have high blending octane value in gasolines. They comprise about 20% to 50% of gasoline and 30% to 40% of diesel fuel (2).

Polymeric materials can undergo swelling and decomposition when exposed to high concentrations of aromatics. Extensive earlier work on rubber materials was performed using the aromatic toluene, generally in a mixture with the alkane isooctane (6)(7).

**Oxygenates** – Oxygenates in fuel blends include lower molecular weight alcohols, such as methanol (MeOH) and ethanol (EtOH). Oxygenates also include ethers such as methyl tertiary - butyl ether (MTBE), ethyl tertiary - butyl ether (ETBE), and tertiary-amyl methyl ether (TAME). Oxygenated compounds are manufactured to create useful fuel components starting with natural gas, certain natural gas liquids, certain refinery and petrochemical intermediates, and alcohols generated from biomass feeds that are readily available to the fuel industry.

MTBE is the most common oxygenate in the world because it has good blending octane, can be made from oil field by-product gases and it is easy to blend with hydrocarbon fuels. Mixtures of up to 15% MTBE with gasoline are typical.

Worldwide production of ethanol for fuel is confined to that produced from corn in the US and sugar cane in Brazil. In the US, ethanol fuel mixtures consist of either 10% fuel ethanol splash blended with gasoline (Gasohol) or E85 (70% to 85% fuel ethanol with the balance being gasoline of sufficiently high volatility to facilitate cold starting). Two ethanol fuels are used in Brazil; E22 (20% to 25% anhydrous ethanol splash blended with gasoline) or straight hydrated ethanol (approximately 93% ethanol plus 7% water).

Oxygenates in diesel fuel are currently limited to fatty acid methyl esters. These products are currently being blended into commercial diesel fuels; however, volumes of these products in the market are still small.

In Europe, methyl esters are made from the transesterification of rapeseed oil with methanol predominate. These are referred to as rapeseed methyl esters (RME). RME fuels are generally used as 5% blends in regular diesel fuel primarily in France. German regulations permit the use of 100% RME and some is available in that marketplace.

In the US, vegetable oils (predominantly soybean oil), animal fats and used cooking oils are reacted with either methanol or ethanol to form fatty acid alkyl esters. The US product is referred to as BioDiesel. It generally appears in the US marketplace as a blend of 5% to 10% ester in standard diesel fuel.

Oxygenates can affect polymers (including elastomers, and plastics) and polymer systems (including laminates and multi-layered components). Alcohols are strong polar solvents; as such they have the greatest effect on polar polymers (6)(7).

Oxygenates and some compounds derived from these oxygenates can also affect metals, especially when moisture is present in the fuel. In particular, alcohols are hygroscopic and can attract large amounts of water into the fuel system. This water can remain in solution in fuel/alcohol mixtures unless the temperature of the mixture is below its water solubility limit, at which point a large water/alcohol separated phase will form. This water phase can cause metal corrosion (8).

Impurities – During the manufacturing, transfer, storage and use of fuels, many chemical reactions can occur to fuels and fuel components. The products of these reactions can in turn affect the properties of the materials that are in contact with the fuels.

Contaminated fuels can represent especially severe environments for polymer, elastomer, plastic, and metal fuel system parts. Several different types of contaminants commonly found in commercial fuels can be added to the test fluids to increase the severity of the test fluids for more rapid materials testing.

Acids – Organic acids such as formic acid and acetic acid are present in certain fuels along with its corresponding alcohol (MeOH and EtOH, respectively). The acid is formed either in the alcohol production process or due to oxidation of the alcohol during handling, transfer or storage. Ethanol may also contain trace amounts of sulfuric acid, a strong mineral acid that also originates in the production process.

Organic sulfur-containing acids and corresponding sulfur-containing esters can be formed by sulfur containing gasoline components that react with alcohols, notably ethanol and acetic acid / ethanol mixtures. These acids affect materials that are in contact with the fuel (9).

Peroxides – Fuel hydrocarbons can undergo natural oxidation in the presence of heat and oxygen. Unsaturated hydrocarbons, such as olefins, degrade more easily. Organic peroxides form as a result of fuel oxidation (often referred to as auto-oxidation). These peroxides decompose to form free radicals that can chemically attack reactive sites (primarily at carbon double bonds but also hydrogen bonds to the carbon backbone) on polymers and elastomers in the fuel system. Other by-products of these degradation processes are acids that cause corrosion of metal components in the fuel system and gums and varnish that can coat the fuel system.

Although time and temperature alone are capable of decomposing peroxides, transition metals capable of a one electron transition, such as copper, will catalyze the decomposition of organic peroxides thereby accelerating this reaction (10, 11, 12).

Ionic Compounds – Sodium chloride or salt, is a compound that often makes its way into fuel systems. In fuels that contain moisture, the salt can adversely affect materials, especially metals (9).

Water – Water has been mentioned in conjunction with several of the components. It is ubiquitous in the fuel environment, occurring as part of the manufacturing, transport and distribution of fuels. Water allows and contributes to many reactions concerning fuel components. Many of these reactions adversely affect materials in fuel systems.

Sulfur – Commercial gasolines contain varying amounts of sulfur in several forms. Prior to the introduction of reformulated gasoline, the average 1990 sulfur content in U. S. gasolines was 338 ppm. The ASTM D 4814 standard for gasoline allows up to 1000 ppm of total sulfur, the U. S. Federal Reformulated Gasoline (RFG) specification currently has no limit for total sulfur, and the current California CARB Phase 2 RFG regulations allow an average of 30 ppm of total sulfur. Similar restrictions on sulfur are becoming common around the world. In Europe, gasoline sulfur levels will be limited to 50 ppm after 2005.

One process in the refining and gasoline processing industry is the “sweetening” of gasoline components by converting mercaptans ( $R-S-H$ ) to disulfides. Disulfide compounds are in the form of  $R_1-S-S-R_2$ , where the groups  $R_1$  and  $R_2$  are generally  $C_1$  to  $C_4$  alkyl radicals. Disulfides can be converted to sulfonic and sulfinic acids in the presence of atmospheric oxygen and water.

Disulfides and their related sulfonic and sulfinic acid oxidation products have significant effects on metals, some elastomers, and some plastics. The stability of fuels and test fluids containing these sulfur compounds is not well understood. Further research is required before sulfur impurities can be recommended for addition to material testing fluid formulations.

Commercial diesel fuels also contain varying amounts of sulfur. The 1996 average sulfur content in U. S. and European diesel fuels was 310 ppm while the application specifications limit sulfur to 500 ppm max. The trend to lower sulfur diesel is underway, with the 2005 European sulfur content target set at 50 ppm. Similar reductions on diesel fuel sulfur are becoming common around the world.

**SELECTING TEST FLUID CHEMICALS** – As was stated before, due to the complexity and variability of commercial automotive fuels, they are not suitable for repeatable and reproducible scientific study of materials or material qualification testing. The next problem then is to develop a set of controlled test fluids to simulate commercial fuels. Selection criteria include:

- Representative of marketplace fuels.
- Creates a severe, reproducible level of a particular effect.
- Safe and easy to handle in a laboratory setting.
- Safe and easy to use at temperatures between  $-40^{\circ}\text{C}$  and  $+60^{\circ}\text{C}$ .
- Globally available to scientists and engineers.
- Available with no potentially active impurities or contaminants.

Base Fluid – Because fuels consist primarily of alkane or aromatic hydrocarbons, selecting one species from each class should create representative test fluids. However, with real fuels consisting of such a wide range of alkanes and aromatics, why select one of the class over another? One criterion has been to identify the compound that creates the greatest effect. In the case of fuel system materials volume swell is used as a reliable indicator of the general effect a solvent will have on polymers and elastomers. The mechanisms that create swell in various polymer–solvent systems are complex (13)(14)(15)(16). While it is an inexact, way to think of this, in many cases the lowest molecular weight species in a class tends to have the greatest effect. For example, isobutane is the smallest alkane in gasoline, toluene is the smallest aromatic, methanol the smallest alcohol, dimethyl ether is the smallest ether, etc. The problem with this approach is that the boiling point of many of these choices is below a practical laboratory range. This also makes them unlikely to appear in large quantities in real world fuels that are handled in open containers at atmospheric pressure.

For at least 20 years the issue of correlation between test fluids and marketplace fuels has been addressed by creating test fluids from the main classes of fuel chemicals. The ASTM reference fuel series, described in ASTM D 471/ISO 1817, consists of mixtures of the alkane, isooctane (BP =  $99.3^{\circ}\text{C}$ ) and the aromatic, toluene (BP =  $110.6^{\circ}\text{C}$ ). Their boiling points make them reasonably safe and easy to use even at elevated test temperatures. Furthermore, toluene is the lowest

molecular weight, safe to handle aromatic hydrocarbon and it has a significant effect on polymers and elastomers.

During the discussions involved in rewriting SAE J1681 task force members pointed out that the cost and availability of isooctane might pose an impediment to more universal acceptance of this document. It was suggested that isoparaffin solvents were widely available and were controlled to sufficiently narrow chemistries to permit repeatable test results. Furthermore it was argued that the alkanes have the least effect of all the fuel hydrocarbons on polymers and elastomers. For example, in permeation experiments (17)(18), researchers have found that when testing with ASTM fluid C the speciated permeant consists of 98% toluene and 2% isooctane. For mixtures of Fluid C and methanol the speciated permeant consists of 65% methanol, 33% toluene and 2% isooctane.

Before adopting a change of this type, members of the committee conducted experiments to compare the effect of isooctane and the isoparaffin solvent both alone and in test fluid mixtures. Two isoparaffin surrogates, Soltrol 10 and Isopar C, were tested on materials and compared with isooctane. Solvent uptake tests on loosely crosslinked isoprene were performed to determine polymer swelling after exposure to these three hydrocarbons.

Swelling tests were performed on crosslinked isoprene with exposures of 42.5 and 66.5 hours, at  $40^{\circ}\text{C}$ . Additional testing was done on High Density Polyethylene (HDPE) with exposure of 450 hours, at  $40^{\circ}\text{C}$  and on Natural Rubber with exposure of 450 hours, at  $40^{\circ}\text{C}$ .

The results of these tests are presented in table 1.

These results confirmed that there was no statistically significant difference in the swell caused by the isoparaffin solvent or isooctane. The results, however, hint that there may be a tendency for the isoparaffin solvents to be slightly more severe. This is reasonable to expect because the isoparaffin solvent contains small amounts of alkanes of a lower molecular weight than isooctane. Therefore, use of the isoparaffin solvent may result in a conservative evaluation of the suitability of materials for use in fuel systems. SAE J1681 designates isoparaffin solvent test fluid "surrogate fluid C" (termed fluid SC).

Those using the new SAE J1681 must consider the purpose of their testing when choosing the right base fluid. The use of isoparaffin solvent based fluids may be the right choice for routine material specification testing because of its cost and availability. If the test purpose is to study detailed physical or chemical phenomena it may be better to use fluids based on isooctane. For example, speciation of permeant is repeatable and easy to interpret when using isooctane but not so with isoparaffin solvent.

**Table 1.**

	<b>Solvent Uptake, Weight fraction (1)</b>	<b>Solvent Uptake, Percent Weight increase (2)</b>	<b>Solvent Uptake, Percent Weight increase (2)</b>
<b>Material</b>	Crosslinked Polyisoprene	High Density Polyethylene	Natural Rubber
<b>Solvent</b>			
Isooctane	2.07	5.96	72.7
Soltrol 10	2.23	6.64	89.0
Isopar C	2.15	6.21	79.4
ASTM Fluid C	4.00	---	---
Surrogate C1 (50% Soltrol 10 + 50% toluene)	4.09	---	---
Surrogate C2 ( 50% Isopar C + 50% toluene)	4.04	---	---

Notes:

(1) Weight fraction is defined as the difference in weight before and after exposure of the polymer to the solvent, divided by the original weight of the polymer tested. Polymer preparation is described by Mazich, Rossi and Smith in *Macromolecules* **25**, 6929 (1992). Testing courtesy of Ford Motor Co.

(2) Testing performed on 1 x 2 inch samples suspended in the test fluid. Testing courtesy of Solvay Automotive.

Additives to Create Other Effects – The main effects of fuel on materials can be increased or changed to additional effects by the presence of major additives such as oxygenates or due to contaminants such as water, auto-oxidation by-products, acids, and sulfur. To simplify the laboratory practice, the SAE J1681 task force decided to include all the typically severe contaminants in either the oxygenates or water so that the appropriate test fluid could be simply produced by mixing standardized stock solutions.

Ethanol – The most common source for fuel ethanol is from biomass (corn, grain, sugar cane, etc.) fermentation. Since there are multiple feed sources and several fermentation/purification processes, the minor components of biomass derived ethanols vary greatly. SAE J1681 now recommends the use of synthetic ethanol that has a recognized set of specifications. The ethanol and its associated denaturant specified in J1681 will help to minimize some of the variables in the use of ethanol as a test fluid component. For example, many denaturants use methyl-ethyl ketone (MEK) as a major ingredient. The presence of MEK would add an unnecessary degree of severity because it is not present in real fuels.

The denaturant specified by SAE J1681 is approved by the U. S. Bureau of Alcohol, Tobacco & Firearms (BATF), and will render the ethanol as “completely denatured”. This denaturant is made primarily of heptane isomers, and was chosen so that the denaturant would have a

minimal effect on the test fluid. At the same time, the chosen denaturant will allow the testing laboratory to avoid the expenditure of resources on the BATF record keeping requirements associated with non denatured ethanol or “specially denatured” ethanols.

Ethanol derived from biomass contains a complex set of acid(s) and buffers associated with the acid(s). The buffering process is not fully understood at this time, but acetic acid that is found in biomass derived ethanol has been found to act as a buffer, tending to control the pH of the alcohol when strong acids such as sulfuric acid are present.(6)

A new method analogous to pH, called  $pH_e$ , has been developed for use with ethanol. The method for measuring  $pH_e$  is ASTM D 6423-99, Determination of  $pH_e$  of Ethanol. Denatured Fuel Ethanol and Fuel Ethanol (Ed75-d85). This method uses special pH electrodes and an ISE meter to rank ethanols for acid strength on an arbitrary scale. The method was developed to overcome the shortcomings of ASTM D 1613 Acidity method which only measures buffering capacity and cannot distinguish between weak and strong acids. Many ethanol producers are currently using the D 6423 method to monitor and control strong acids in biomass derived ethanol. ASTM is presently balloting  $pH_e$  specification limits of 6.5 to 9.0 for inclusion into ASTM D 4806, Standard Specification for Denatured Fuel Alcohol for Blending with Gasoline for Use as Automotive Spark Ignition Engine Fuel.

SAE J1681 now recommends that a specific synthetic ethanol be purchased for materials testing. Reagent Water is added to purchased alcohols prior to their use in materials testing fluid blends simulate the water content commonly found in fuel grade methanol and ethanol. The amount of water required is determined by difference between the total water content required and the water content of the product as received, determined by Karl Fisher titration. Ethanol for use in materials testing fluids should be adjusted by adding Reagent Water to the ethanol, as received, to obtain a test fluid component that contains 1.0 wt.% total water content.

Most materials test fluids containing ethanol should be what is termed "Aggressive Ethanol" in SAE J1681. An "Aggressive" alcohol contains the alcohol, salts, water and a corresponding organic acid adjusted to represent the aggressive aspects of fuel grade ethanol produced from biomass. Aggressive ethanol also contains sulfuric acid, a contaminant found in some commercial bio mass derived ethanols. The recipe is shown in Table 2.

Methanol – The methanol specified is synthetic commercial methanol derived from natural gas and as such has few production induced impurities compared to ethanol from biomass. The specifications for methanol

found in ASTM D 1152 are recommended by SAE J1681. Methanol used in materials testing fluids should be adjusted by adding Reagent Water to the methanol, as received, to obtain a test fluid component that contains 0.3 wt.% total water content.

Methanol for materials testing should be aggressive methanol. The recipe appears in table 3.

Methyl-Tertiary-Butyl Ether (MTBE) – MTBE is the most prevalent oxygenate / octane booster found in current gasoline formulations. MTBE is used worldwide as a replacement for tetraethyl lead, which is legally prohibited from many automotive fuels. Suppliers and specifications for the oxygenated components are presented in SAE J1681. There are no known production related impurities in MTBE which have been shown to adversely effect materials so none are added to purchased MTBE.

Corrosive Water for Metals Testing – Corrosive water for hydrocarbons is composed of Reagent Water and salt. Fluids C and SC (or C and SC with MTBE) are the only fluids that can contain Corrosive Water and are used for metals testing only. The formulation used in making corrosive water is also found in Table 4.

**Table 2. Recipe for Aggressive Ethanol**

Component	Units	1.0 Liter	1.0 Gallon
Ethanol, Synthetic	gm.	816.0	3084.5
Deionized Water	gm.	8.103	30.631
Sodium Chloride	gm.	0.004	0.014
Sulfuric Acid	gm.	0.021	0.080
Glacial Acetic Acid	gm.	0.061	0.230

Note: to minimize esterification reactions, the sulfuric acid should be blended into the Reagent Water prior to mixing with ethanol.

**Table 3. Recipe for Aggressive Methanol**

Component	Units	1.0 Liter	1.0 Gallon
Methanol, Commercial Grade	gm.	792.4	2995.3
Reagent Water	gm.	2.330	8.808
Sodium Chloride	gm.	0.004	0.014
Formic Acid	gm.	0.046	0.173

**Table 4. Recipe for Corrosive Water**

Component	Units	1.0 Liter	1.0 Gallon
Sodium Chloride	gm.	0.165	0.624
Reagent Water	gm.	1000	3780

Peroxides – Tertiary-butyl hydro-peroxide (TBHP) is the recommended organic peroxide to be added into test fluids to simulate and create an oxidized fluid. TBHP is normally sold either as a 70% aqueous solution or as part of a hydrocarbon solution. In order to control the amount of TBHP added, the peroxide content of the TBHP solution must be known. This determination can be performed by the supplier and provided with the product, or the testing laboratory can perform it prior to use per ASTM E 298, "Test Methods for Assay of Organic Peroxides".

In the past a great deal of confusion existed because of the "Peroxide Number" system used to report and/or specify the strength of peroxide solutions. Peroxide Number (PN) is an indication of the quantity of oxidizing constituent present in solution. The active species, i.e. oxidizing agents, are free radicals generated as a result of hydro-peroxide decomposition. This process may occur by thermal decomposition, resulting in two free radicals or by metal catalyzed decomposition, resulting in one free radical. Therefore, for a given same starting quantity of hydro-peroxide, e.g. 50 mMol/liter, thermal decomposition will produce a PN of 100 whereas metal catalyzed decomposition produces a PN of 50 (19)

The SAE J1681 task force decided that the point of this document was to provide direction to testing labs that it would focus on how to make the solutions rather than rating the effectiveness of any given solution. Therefore, the J1681 task force has adopted the practice of specifying the strength of the solution in millimoles of hydro-peroxide per liter.

In order to simulate an oxidized fuel, 50 millimoles (mMol) of TBHP are added per liter of the test fluid. The amount of TBHP solution required to obtain a concentration of 50 mMol TBHP per liter of test fluid is calculated using the following equation:

Grams of TBHP solution to make 1.0 liter of test fluid =:

$$\frac{(50\text{mMol TBHP}) \times (90 \text{ mg / mMol TBHP})}{(\text{wt. fraction of TBHP in solution}) \times (1000 \text{ mg/g.})}$$

According to this equation, 6.43 grams of 70% TBHP solution is required per liter of test fluid.

Small amounts of copper, especially cuprous, (Cu<sup>+1</sup>) compounds are often added to a test fluid that contains organic peroxides to accelerate the decomposition of peroxides into free radicals. Copper compounds are documented in the literature as promoters of free radicals, and their use offers an acceptable method to reduce testing time in auto-oxidized fluid mixtures (10, 11, 12). Copper is often found in fuel systems in the form of electrical circuit components, fuel pump motor windings and armatures and in associated wiring.

Copper addition to test fluids containing TBHP in prior work was accomplished using a cupric (Cu<sup>+2</sup>) salt such as copper naphthenate. In the Cupric form, heat and time were required to convert some Cu<sup>+2</sup> into Cu<sup>+1</sup> in order to make it available as a catalyst in peroxide decomposition

reactions. Results from current and prior testing using cupric salts as a promoter are considered valid. If a suitable cuprous (Cu<sup>+1</sup>) compound which is both soluble and active can be found, its use will accelerate the formation of free radicals in the test fluid, which in turn accelerates the oxidation of the test fluid and generates quicker results.

The use of copper compounds to create copper ions in solution is optional for SAE J1681. If required, the final test fluid should contain 0.01 mg of Cu per liter. The amount of copper compound required to give 0.01 gm copper per liter of test fluid can be calculated from the following equation where the Wt% copper is obtained from the suppliers specification for the copper compound or it is determined by testing:

$$(0.01 \text{ mg Cu}) \div [(\text{Wt\% Cu in copper compound}) \times (0.01)] = \text{mg Cu compound to be used}$$

The addition of copper should be made with adequate mixing immediately AFTER blending the TBHP into the test fluid. Mixing the cuprous ions into the concentrated TBHP solution is NOT recommended.

It should be noted that adjustments in test time and/or temperature should be made if changing from a test fluid that either contained or did not contain the copper ion. For example, to duplicate tests conducted with the copper ion using a test fluid without the copper ion might require raising the test temperature 10°C to 20°C or increasing the exposure time by a factor of 2 to 4 at the same temperature.

#### NAMING CONVENTION FOR TEST FLUIDS –

A recommended naming convention for test fluids and fuels was evolved based on the system published in the first issue of SAE 1681. Using these abbreviations uniquely identifies the composition of each fluid. Uniform adaptation of this flexible nomenclature for other test fluids is encouraged.

The general formula for a material test fluid is:

$$X (Y_1 \%) (Y_2 \%) (Y_n \%) S$$

where:

X = Hydrocarbon fluid

Y<sub>1</sub> = First optional oxygenate

Y<sub>2</sub> = Second optional oxygenate

Y<sub>n</sub> = the n<sup>th</sup> optional oxygenate

% = Volume percent of oxygenate

S = optional minor constituent such as:

P = the presence of an organic peroxide contaminant which can be added to any fluid mixture.

A = a subscripted "A" following the alcohol component indicating the presence of an aggressive alcohol.

w = a lower case "w" component indicating the presence of Corrosive Water added to a hydrocarbon fluid or MTBE containing hydrocarbon fluid.

Hydrocarbon Nomenclature – Letter designations for the recommended hydrocarbon fluids in the nomenclature are:

C = ASTM Fluid C (50 % Toluene and 50% Isooctane)

SC = ASTM Surrogate Fluid C (50 % Toluene and 50% substitute isoparaffin solvent)

D<sub>EPA2</sub> = EPA Certification No. 2 low sulfur reference diesel fuel

Oxygenate Nomenclature – Letter designations for the more common oxygenates in the nomenclature are:

M = methanol

E = ethanol

ME = methyl tertiary-butyl ether

EE = ethyl tertiary-butyl ether

TE = tertiary-amyl methyl ether

RME = rapeseed methyl esters

SME = soybean methyl esters (this is the recommended version of BioDiesel).

Examples – Examples utilizing this naming convention are presented below:

Example 1: C(E10) = 90 volume % Fluid C  
+ 10 volume % ethanol.

Example 2: SC(M25)<sub>A</sub> = 75 vol.% Surrogate Fluid C  
+ 25 vol.% aggressive methanol.

Example 3: C(E25)(ME15) = 60 volume % Fluid C  
+ 25 volume % ethanol  
+ 15 vol.% methyl tertiary butyl ether.

Example 4: C(ME15)P = 85 volume % Fluid C  
+ 15 volume % methyl tertiary butyl ether  
+ tertiary butyl hydro-peroxide (an auto-oxidized fluid).

Example 5: C(E10)<sub>A</sub>P = 90 volume % Fluid C  
+ 10 volume % aggressive ethanol  
+ tertiary-butyl hydro-peroxide (an auto-oxidized fluid).

Example 6: D<sub>EPA2</sub> (RME20) = 80 vol.% Certification diesel + 20 volume % rapeseed methyl esters.

Example 7: SC<sub>w</sub> = Surrogate Fluid C with Corrosive Water (for metals testing).

**RECOMMENDED FLUIDS FOR TESTING FUEL SYSTEM MATERIALS** – While SAE J1681 establishes a system of methods and nomenclature capable of creating and naming an infinite variety of potential test fluid recipes it is not the intention that materials should be tested in all potential fluids. SAE J1681 recommends a minimum number of test fluids to evaluate all candidate materials.

The recommended fluids are designed to simulate typical, severe, real world conditions that can be encountered. The selected fluids are intended to:

- Minimize the testing required to rigorously evaluate fuel system materials,
- Reduce variability in test fluids listed in materials specifications, and
- Standardize testing of fuel system materials.

The final choice of materials to be selected and the test fluids to be used to qualify these materials are dependent on the application as well as the specific design of the component. It will likely involve the cooperative efforts of the component engineer, materials engineer and the suppliers.

The test fluid compositions specified in Section 7 of SAE J1681 are recommended solely for evaluating materials. They are not intended for other activities, such as engine development, design verification or process validation unless agreed upon by the contracting parties. SAE J1681 now also lists marketplace test fuels that can be used for engine, vehicle or component testing.

For the purposes of this discussion the term FLUID is applied to mixtures of specific controlled components used to simulate the effects of fuels. The term FUEL is used in conjunction with fully blended hydrocarbon or hydrocarbon/oxygenate mixtures for use in commercial automotive engines.

Fluids to Qualify Materials for Worldwide, Basic, Gasoline and Diesel Fuel System Applications – The four test fluids that are representative of severe general gasoline fuels in marketplaces, worldwide include:

C(M15)<sub>A</sub> or SC(M15)<sub>A</sub>,  
C(ME15) or SC(ME15), and  
CP or SCP  
C<sub>w</sub> or SC<sub>w</sub> for metals testing only

Testing in these fluids alone is sufficient for qualifying materials for consideration in general fuel system applications.

Note that a fuel mixture that contains methanol is recommended (CM15) whereas methanol is rarely found in commercial fuels. This fuel has been used for years in the automotive industry and continues to be a valid test fluid because methanol creates effects on materials which are representative of worst case marketplace fuels whether they contain methanol or not. Ethanol and higher alcohols, which may be more common in fuels, usually create significantly smaller effects on fuel system materials.

Diesel fuel is usually less chemically aggressive; therefore, materials qualified in these four test fluids can also be used in diesel fuel applications without being tested in additional fluids.

Fluids to Qualify Materials for Dedicated, Alcohol Based Fuel System Applications – Materials to be qualified for use in fuel systems intended for dedicated, alcohol fueled vehicles (E100 to E85 or M100 to M85 fuels) must be tested in the following test fluids:

All fluids designated for World Wide Basic qualification testing listed above and either: C(M85)<sub>A</sub>, SC(M85)<sub>A</sub>, C(E85)<sub>A</sub>, or SC(E85)<sub>A</sub> (depending on the intended marketplace alcohol fuel: ethanol or methanol).

Fluids to Qualify Materials for Alcohol Based, Flexible Fueled Vehicle Fuel System Applications – Materials to be qualified for use in fuel systems intended for flexible fueled vehicles (gasoline, E85, or M85) should be tested in the intended marketplace alcohol fuel (ethanol or methanol) as follows:

All fluids designated for World Wide Basic qualification testing listed above and

C(E15) <sub>A</sub>	or	SC(E15) <sub>A</sub> (if applicable) and	
C(M30) <sub>A</sub>		or	SC(M30) <sub>A</sub>
C(E30) <sub>A</sub>	or	SC(E30) <sub>A</sub>	and
C(M50) <sub>A</sub>		or	SC(M50) <sub>A</sub>
C(E50) <sub>A</sub>	or	SC(E50) <sub>A</sub>	and
C(M85) <sub>A</sub>		or	SC(M85) <sub>A</sub>
C(E85) <sub>A</sub>	or	SC(E85) <sub>A</sub>	and
C(M20) <sub>A</sub> (ME15)	or	SC(M20) <sub>A</sub> (ME15)	or
C(E20) <sub>A</sub> (ME15)	or	SC(E20) <sub>A</sub> (ME15).	

The purpose for testing with the six additional fluids indicated in this section is to identify the fluid composition that causes the greatest effect on the material being evaluated. The worst case fluid should then be used exclusively for all subsequent testing of that material. This allows all materials to be compared at their individual worst to indicate suitability for use in a particular application.

Fluids to Qualify Materials for Diesel Fuel Only Applications – Materials previously qualified in gasoline systems and tested in fluids designated in Section 7.1.2 will qualify for diesel fuel applications. To qualify for diesel fuel only applications, candidate materials need only be tested in D<sub>EPA2</sub> (EPA certification No. 2 diesel fuel). Testing in RME or BioDiesel and their mixtures with diesel fuel may also be required.

**FURTHER QUALIFICATION OF MATERIALS IN MARKETPLACE FUELS** – In addition to the basic fuel compatibility testing specified above, component or automotive original equipment manufacturers may require further testing in marketplace representative fluids or fuels. These include:

1. test fluids that simulate marketplace fuels which are all fluids designated for World Wide Basic qualification testing listed above, and
2. test fuels that represent typical marketplace fuels:

- a. EPA certification gasoline
- b. California Phase II reformulated gasoline
- c. GM test fuel 1(TF-1)
- d. GM test fuel 2(TF-2)
- e. Brazilian Gasohol(with 25 % Biomass Ethanol)
- f. D<sub>EPA2</sub>(EPA certification No. 2 diesel fuel)
- g. D<sub>EPA2</sub> (RME20)(EPA certification No. 2 diesel fuel plus 20% RME)
- h. D<sub>EPA2</sub> (SME20)(EPA certification No. 2 diesel fuel plus 20% SME)

**OTHER REQUIREMENTS** – A material's properties, including its fuel compatibility, can be strongly influenced by the part design and the manufacturing processes used to convert the raw material into a finished component. The task of assessing fuel compatibility and suitability of a material for use in a fuel system is not complete unless the material is re-evaluated after conversion into the intended component using the test fluids or test fuels identified as worst case. However, as emphasized earlier in this report, it is not intended that the TEST FLUIDS described here be used for product or process validation of devices (e.g., fuel pumps, injectors, etc.) assembled from materials so qualified. Product validation should only be conducted using the TEST FUELS specified in this document.

**COMPARISON OF THE NEW SAE J1681 SYSTEM WITH THE OLD SYSTEMS** – Using the background provided by this paper, an inspection of Appendix A reveals that most of the test fluid recipes involve an ASTM Ref Fuel, Iso octane, toluene or alcohols. Each of these could be described in terms of the system of naming test fluid described in this paper. The differences in the specific test fluid compositions are small compared to those recommended by SAE J1681.

Furthermore, during the research involved in preparing the revised SAE J1681, it was found that each of the chemical compounds identified for use in a test fluid recipe could be procured to a wide range of specifications. This presented yet another source of potential variation in testing practice. Therefore, in addition to standardizing the nomenclature and test fluid composition SAE J1681 now presents exact specifications for obtaining all of the test chemicals. Specific measures were taken to assure that allowed impurities would be similar to fuel constituents and represent an uncontrolled aggressive factor to potential fuel system materials.

## **CONCLUSION**

1. Commercial fuels can vary substantially between manufacturers, batches, seasons and geographic location. Using those fuels for materials testing will lead to uncontrollable variation in test results. Therefore, use of the standardized test fluid and test

fuels listed in SAE J1681 will permit consistent results from materials testing and more uniform specification of materials.

2. The new version of SAE 1681 presents recommendations for test fluids that can be used to simulate most commonly available real world fuels. It defines the nomenclature and composition for standardized surrogate gasoline and diesel test fluids and test fuels to be used in materials testing.
3. The new standardized nomenclature and fluid recipes can easily replace the many systems now in use around the world.
4. Formulations in SAE J1681 have been designed and developed to:
  - Be representative of marketplace fuels.
  - Create reproducible levels of particular effects that are intended to exaggerate the effects of typical severe fuel on materials.
  - Allow tests to be conducted in a reasonable amount of time.
  - Be safe and easy to handle in a laboratory setting.
  - Be safe and easy to use at temperatures between  $-40^{\circ}\text{C}$  and  $+60^{\circ}\text{C}$ .
  - Be globally available to scientists and engineers.
  - Be available with no potentially active impurities or contaminants.

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**APPENDIX A**

**Worldwide Test Fluids and Test Fuels**

<b>IDENTIFICATION</b>	<b>NAME</b>	<b>MAJOR INGREDIENT</b>	<b>ALKANE (vol. %)</b>	<b>OLEFINIC (vol. %)</b>	<b>AROMATIC (vol. %)</b>	<b>OXYGENATE (vol. %)</b>	<b>OTHER (vol. %)</b>
<b>AUTO-OIL GASOLINE</b>	U.S. Industry Avg. (Summer)	Full range distillate mixed C <sub>4</sub> - C <sub>10</sub> Hydrocarbon	58	9.2	32	none	1.5 Benzene, 8.7 psig RVP 330°F T <sub>90</sub>
<b>U.S. EPA CERTIFICATION GASOLINE</b>	Specified Fuels & Chem. EEE; Indolene Clear	Full range distillate mixed C <sub>4</sub> - C <sub>10</sub> Hydrocarbon	Balance	10.0 max.	35.0 max.	none	.5 Benzene, 9.0 psig RVP 309°F T <sub>90</sub>
<b>CALIFORNIA PHASE II REFORMULATED</b>		Full range distillate mixed C <sub>4</sub> - C <sub>10</sub> HC + Oxygenate	74	4.0	22	15 MTBE or 5 Methanol or 7 Ethanol	.8 max. Benzene, 7.8 psig RVP, 290°F T <sub>90</sub>
<b>US EPA CERTIFICATION #2 DIESEL FUEL</b>		Full range distillate mixed C <sub>9</sub> - C <sub>25</sub> Hydrocarbon	Balance	2.5 Max.	28.0 - 35.0	none	
<b>ASTM D 471 ('79) / ISO 14469</b>	Ref. Fluid A	Iso-Octane	100				
	Ref. Fluid B	Iso-Octane	70		30 Toluene		
	Ref. Fluid C		50 Iso-Octane		50 Toluene		
	Ref. Fluid D	Iso-Octane	60		40 Toluene		
	Ref. Fluid E	Toluene			100 Toluene		
	Ref. Fluid F	Grade #2 Diesel Fuel	Balance mixed	2.5 max.	28 - 35 mixed		
	Ref. Fluid G	Ref. Fluid D				15 anhydrous denatured Ethanol	
	Ref. Fluid H	Ref. Fluid C				15 anhydrous denatured Ethanol	
	Ref. Fluid I	Ref. Fluid C				15 anhydrous denatured Methanol	
	Ref. Fluid K	Ref. Fluid C				85 anhydrous denatured Methanol	
<b>DIN 51 604</b>	FAM A (11/82)	Toluene	30 Iso-Octane	15 Di-Isobutylene	50 Toluene	5 Ethanol	
	FAM B (03/84)	84.5 FAM A				15 Methanol	0.5 Water
	FAM C (03/84)	40 FAM A				58 Methanol	2.0 Water

## Worldwide Test Fluids and Test Fuels

IDENTIFICATION	NAME	MAJOR INGREDIENT	PARAFFINIC (vol. %)	OLEFINIC (vol. %)	AROMATIC (vol. %)	OXYGENATE (vol. %)	OTHER (vol. %)
ISO 1817 (03/85)	Liquid A	ASTM Fluid A					
	Liquid B	ASTM Fluid B					
	Liquid C	ASTM Fluid C					
	Liquid D	ASTM Fluid D					
	Liquid E	Toluene			100 Toluene		
	Liquid F	Mixed paraffinic	80 C <sub>12</sub> to C <sub>18</sub>		20 1-Methylnaphthalene		
	Liquid 1	DIN EAM A					
	Liquid 2	DIN EAM B					
	Liquid 3	90 ASTM Fluid C				7 Ethanol 3 Methanol	
	Liquid 4	85 ASTM Fluid C				15 Methanol	
	CM(As specified)	ASTM Ref Fluid C				As specified Methanol	.5 Aggressive water
	CM(As specified)P	ASTM Ref Fluid C				As specified Methanol	50 mmol/liter t-Butylhydro-peroxide plus .01 mg Cu <sup>+</sup> ion
	CM0/CM0P	ASTM Ref Fluid C					"P" index = adding 50 mmol/liter t-Butylhydro-peroxide plus .01 mg Cu <sup>+</sup> ion
	CM15/CM15P	ASTM Ref Fluid C				15 Methanol	
	CM30/CM30P	ASTM Ref Fluid C				30 Methanol	Aggressive Methanol = 995 ml Anhydrous Methanol + 5 ml Aggressive water = Add to one liter of Distilled Water:
CM50/CM50P		25 Iso-Octane			50 Methanol	.028 ml organic acid normally in chosen alcohol + 990 mg NaCl + 888 mg Na <sub>2</sub> SO <sub>4</sub> + 828 mg NaHCO <sub>3</sub> .	
CM85/CM85P	Aggressive Methanol	7.5 Iso-Octane			7.5 Toluene	85 Methanol	

## Worldwide Test Fluids and Test Fuels

IDENTIFICATION	NAME	MAJOR INGREDIENT	PARAFFINIC (vol. %)	OLEFINIC (vol. %)	AROMATIC (vol. %)	OXYGENATE (vol. %)	OTHER (vol. %)
<b>CHRYSLER</b> Flex Fuel Testing (03/91)	MS-8004 UnLeaded Fuel M15, M30, M85	Full range distillate gasoline 85, 70, 15 MS-8004				15, 30, 85 Aggressive Methanol	Specified Fuels & Chem. Hydrocarbon Aggressive Methanol = 995 ml Anhydrous Methanol + 5 ml Aggressive water (990 ml Distilled Water + 10 ml Formic acid + 1-gm NaCl + .8 gm Na <sub>2</sub> SO <sub>4</sub> )
	Gasohol	90 MS-8004				10 Ethanol 15 MTBE	2.6 ml t-Butylhydroperoxide + 10.4 ml Laurylperoxide per liter of fuel.
<b>FIAT</b> 9.02137/01(04/95)	Oxygenated Gasoline  Oxidized Stale Gasoline	85 MS-8004  MS-8004					
	Gasoline + Alcohol (benzina alcolata)	90 Octane Super (Norm 55511)				3 Methanol + 5 Ethanol + 2 Iso-Propanol	
<b>FORD</b> - CURRENT	Auto-Oxidized gasoline FLTM AZ-105-01 Fuel Compatibility FLTM AZ-105-02 (similar to SAE J1681)	ASTM Ref. Fluid C				As specified Methanol, Ethanol, MTBE or other	mmol/liter Peroxide as specified by adding t-Butyl hydroperoxide + .01 mg Cu ions per liter. .5 Aggressive Water (Add to one liter of Distilled Water: .028 ml organic acid normally in chosen alcohol + 990 mg NaCl + 888 mg Na <sub>2</sub> SO <sub>4</sub> + 828 mg NaHCO <sub>3</sub> ) plus 5 mg NaCl + .05 mL Formic acid per liter of fluid.
	Permeation Test FLTM BP J14-02	85 ASTM Ref. Fluid C				15 Methanol	
<b>GENERAL MOTORS</b> (U.S.)	TF-1  TF-2  GM6264M (03/90) Sour Gasoline Brazil Worst Case	U.S. EPA Certification Fuel U.S. EPA Certification Fuel ASTM Ref. Fluid B  ASTM Fluid C	39.5 Iso-Octane	15 max.  15 max.	adjusted to 45 - 50 adjusted to 45 - 50  39.5 Toluene	10 Ethanol + 5 Methanol + 2.5 Ethanol  20 Ethanol	Specified Fuels & Chem. Hydrocarbons Specified Fuels & Chem. Hydrocarbons 50 Peroxide number by adding t-Butylhydroperoxide 1 Corrosive Water: Into 1 L Distilled Water, dissolve: • 198 mg/L NaCl • 148 mg/L Na <sub>2</sub> SO <sub>4</sub> • 1.67 mL/L Ethyl Acetate • 1.33 mL/L Acetaldehyde • 1.50 mL/L Acetic Acid

## Worldwide Test Fluids and Test Fuels

IDENTIFICATION	NAME	MAJOR INGREDIENT	PARAFFINIC (vol. %)	OLEFINIC (vol. %)	AROMATIC (vol. %)	OXYGENATE (vol. %)	OTHER (vol. %)
OPEL (GMEurope)	GME L 0001 (06/80)	FAM A					20 ppm Formic acid
	GME L 0003 (07/80)	FAM B				22 Ethanol	To each Liter of fuel add: <ul style="list-style-type: none"> <li>• 250 mg Peroxide</li> <li>• 2000 ppm Water</li> <li>• 14.2 mg Acetic Acid</li> </ul>
NIPPONDENSO	Brazil Compatibility Test Fuel - Pumps & Injectors	Gasoline (?)					
RENAULT	03-50-000/-A (11/91) Fluid A	ASTM Fluid A					
	03-50-000/-A (11/91) Fluid B	ASTM Fluid B					
	03-50-000/-A (11/91) Fluid C	ASTM Fluid C					
	03-50-000/-A (11/91) Fluid D	ASTM Fluid D					
	03-50-000/-A (11/91) Fluid E	ASTM Fluid E		100			
	03-50-000/-A (11/91) Fluid L	CEC-RF-08-A-85					DKA Premium, Unleaded Gasoline acc. German stVZO
	03-50-000/-A (11/91) Fluid N	ISO 1817/Liquid 3					
	03-50-000/-A (11/91) Fluid O	ISO 1817/Liquid 4					
	03-50-000/-A (11/91) Fluid T	CEC-RF-01-A-80					Premium leaded gasoline for European emission tests (supplier: Haltermann)
	03-50-000/-A (11/91) Fluid Y	90 ISO 1817, Liquid 3		10 p-Xylene			
VOLVO	STD 1027.6131 (09/91) Test Liquid I	ASTM Ref. Fluid C					
	50 TL-VW 703 Prüfkraftstoff	Iso-Octane		30 Toluene	20 Ethanol		
VOLKSWAGEN	PV 3006 Testkraftstoff	CEC-RF-08-A-85					DKA Premium, Unleaded Gasoline acc. German stVZO (supplier: Haltermann)
	TL-VW 782 R-OK-N UnLeaded	55 mixed C <sub>4</sub> -C <sub>10</sub>	? 10	? 35			4 ppm Cu(II) ion as Cu(CH <sub>3</sub> COO) <sub>2</sub>