

The Effects of E20 on Metals Used in Automotive Fuel System Components

Bruce Jones, Gary Mead, Paul Steevens, and
Mike Timanus

Minnesota Center for Automotive
Research at Minnesota State University, Mankato

ABSTRACT

The focus of this study was to compare the effects of E20 versus E10 and gasoline on metal materials found in automotive, marine, and small engine fuel system components. Metal samples were prepared using SAE and ASTM standards and exposed to blends of Fuel C; Fuel C and 10% aggressive ethanol; and Fuel C with 20% aggressive ethanol at an elevated temperature of 45 °C for 2016 hours. The fuel was changed in weekly intervals with photo images and mass loss/ gain data recorded at the 1st, 3rd, 6th, and 12th week.

INTRODUCTION

Minnesota Governor Tim Pawlenty signed a bill on May 10, 2005 that requires by volume 20% of the fuel sold in Minnesota to be ethanol. Currently, gasoline sold in Minnesota contains 10% ethanol (E10) by volume. Ethanol, C₂H₅OH, is an alcohol that can be derived from starches such as corn or materials containing sugars such as sugar cane. Ethanol can also be made from cellulosic materials such as grasses by converting them into sugars. However, this process is much more cost intensive and has not yet reached commercial production levels. Ethanol is considered a renewable fuel and is also classified as an alternative fuel since it can be used as a substitute for gasoline.

The passage of the law is only the first step. The fuel may not be used on public roadways until a federal section 211f waiver is obtained from the United States Environmental Protection Agency (EPA). Extensive testing in five areas: vehicle driveability, vehicle emission control system effectiveness and durability, vehicle tailpipe and evaporative emissions, fuel system material compatibility, and public health must be conducted in order to obtain the waiver (C. Jewitt, personal communication, July 6, 2005). This will be a costly and timely process requiring many different studies.

This paper represents one in a series of four papers that focus on the effects of 20% ethanol-blended fuel (E20) on fuel system components. This paper provides some of the background information on E20 and the various laws that affect its use as an on-road fuel. Next, it contains a brief review of literature on E20 with a particular focus on material compatibility issues. Finally,

it investigates the issues of designing the material compatibility study in terms of standards, procedures, and equipment needed to carry out the testing.

MINNESOTA ETHANOL LEGISLATION

On May 10, 2005, Minnesota Governor Tim Pawlenty signed into law a bill requiring ethanol to constitute 20% of the gasoline sold in the State of Minnesota. The bill allows for two methods of achieving this. First, if by December 31, 2010 the volume of ethanol sold in the State through the combination of E10 and E85 reaches 20% of the total gasoline sold in the State, then the goal will be met and there will not be any changes in the fuel sold. If the combination of the two fuels' ethanol content does not reach at least 20% of the total fuel sold, then by August 30, 2013 the ethanol content of gasoline will be increased from 10% to 20% by volume (Eisenthal, 2005).

The second method, 20% ethanol in all gasoline, requires the EPA to approve a waiver for the use of E20. EPA waivers may be granted one of two ways. The EPA can review the application and supporting data and grant the waiver. Or, if the EPA fails to provide a decision on the waiver within 180 days, the waiver is automatically granted. This clause is of particular concern because the original fuel waiver for E10 was granted because the EPA failed to make a decision in 180 days. The Minnesota law explicitly states that the failure of the EPA to act shall not be deemed an approval.

LITERATURE REVIEW

The passage of the Minnesota E20 legislation raised many questions about the effects of E20 on non-flex fuel vehicles. All vehicles sold in the United States since the early 1980s are compatible with E10, but whether or not they are compatible with E20 is not known. Before E20 can be sold in Minnesota, a section 211f waiver needs to be obtained from the United States Environmental Protection Agency (EPA). In order to obtain a waiver, extensive research and testing will need to be conducted in five areas: driveability, fuel system material compatibility, tailpipe and evaporative emissions, emission control system effectiveness and durability, and health effects. This research and testing will be conducted to ensure that the fuel does not cause any more problems than gasoline in the five categories.

The fuel systems on vehicles produced today are seeing an ever-increasing amount of ethanol-blended fuels and are expected to have a usable lifespan of 15 to 20 years. Currently, all fuel systems are compatible with E10, but as increased ethanol blends become more prevalent, the need to quantify the effects of higher blends on materials is necessary. Unfortunately, there is little information on the non-linear effects of increased ethanol blends on fuel system materials. This means that as the concentration of ethanol increases from 0 to 100% there is no model that accurately predicts the effects on materials. In fact, neat ethanol and neat gasoline often have a smaller negative impact on materials than gasoline-ethanol blends. To compound this problem, mid-range blends of 15 to 50%, often have the largest negative impact on materials.

The MSU material compatibility study started out like most research with a comprehensive review of the literature. To guide this review, a few questions were asked: (a) What literature exists on E20 and materials? (b) What materials are in a fuel system? (c) What standard test procedures are used to validate a material for compatibility with a fuel? (d) By what criteria is a material deemed compatible or incompatible with a fuel? With these questions in mind, a thorough review of the Society of Automotive Engineers' (SAE) technical paper library was conducted along with an extensive internet search. Also, Original Equipment Manufacturers (OEMs), Tier I and II suppliers (supply parts to the OEMs), and material testing laboratories were contacted for information.

During the search for information on E20, it became apparent that there was very little information publicly available. Most OEMs have tested with a fuel close to E20, such as E25 because of its extensive use in Brazil, but retain this information as proprietary. Several small material studies mentioned E20 or tested a blend close to E20 such as E25, but these studies offered only a small portion of the information that would be necessary for a waiver. In fact, only one major study on E20 was found, the Orbital Engine Company's study for the Commonwealth Government of Australia conducted from October 2002 through March 2003 (Orbital, March 2003, May 2003).

The Orbital study was extremely comprehensive, covering both automobiles and small engines in areas of emissions, driveability, material compatibility, durability, and a well-to-wheel study. With regards to material compatibility, the Orbital study tested actual components from vehicles. The study found that E20 caused significant problems with many metal, plastic, and rubber components that gasoline did not. The Orbital study was carefully reviewed at MSU. The study identified significantly higher levels of discoloration and tarnishing on components exposed to the E20 than the gasoline

reference fuels in the study. However, it should be noted that E10 was not included in this study and other research studies have noted similar discoloration and tarnishing associated with the use of E10. Also, it is believed that corrosive water was added to the E20 blends at levels high enough to cause phase separation but was not added to the gasoline reference fuels. According to SAE recommended practice J1681 *Gasoline, alcohol and diesel fuel surrogates for materials testing*, corrosive water is only added to gasoline or ASTM Fuel C, not ethanol (SAE, 2000). This test method could be a reason for the extreme negative results that the Orbital study reported.

FORMULATING THE EXPERIMENTS

With a lack of available literature on ethanol compatibility, it became apparent that actual material compatibility experiments would be necessary to answer some of the questions about the effects of E20. It was also determined that E10 should be included in any material compatibility tests as a reference to the changes caused by ethanol. Ethanol does cause different changes to materials than gasoline. But, if E20 does not cause a larger negative impact on materials than E10, an accepted motor fuel, then E20 would be acceptable. The inclusion of E10 in the testing for the purpose of differentiating acceptable changes caused by ethanol is a significant component of the MSU material studies.

PROCESS OVERVIEW

Four standard practices from Society of Automotive Engineers (SAE) and the American Society for Testing and Materials (ASTM) were used to develop the specific testing procedures. The procedure developed was a combination of SAE J1747, *Recommended methods for conducting corrosion tests in gasoline/methanol fuel mixtures*, and ASTM G31, *Standard practice for laboratory immersion corrosion testing of metals*. The test fuels were blended as per SAE J1681, *Gasoline alcohol and diesel fuel surrogates for materials testing*. SAE J1747 modifies ASTM G31 to make it fuel-testing specific. A decision was made to use the exposure method of ASTM G31 over SAE J1747 because it allowed more data to be collected. Originally, SAE J1747 called out for all of the samples to be completely immersed. Due to the fact that corrosion can occur at the liquid/vapor level and in the vapor level itself, a decision was made to use ASTM G31's suggested exposure where as one sample is completely immersed, the second sample is halfway immersed, and the third sample is exposed to vapors only. This allowed data to be collected from all three types of exposures as the material would be in use.

All testing and data collection took place in a room equipped with a ventilation system designed to handle hazardous fumes. All samples were prepared as specified by ASTM G1, *Standard practice for preparing, cleaning, and evaluating corrosion test specimens*. After the samples were prepared, the weights and dimensions were measured to provide a baseline for comparison. Photographs of the color and surface texture were also taken.

Three bottles were used for each material. Each contained three coupons of a specific material and one of the three test fuels. Next, 510 ml of the appropriate test solution was added to each of the bottles along with the three test samples on the sample stands. The bottles were placed in the oven unsealed until a temperature of 45 ± 2 °C was reached. Upon temperature stabilization, the bottles were sealed. The samples were exposed to the test fluid for a period of 2016 hours. Each week, the test solution was changed to minimize bulk solution composition changes, oxygen depletion, and to replenish ionic contaminants. Finally, the samples were photographed and weighed after the 1st, 3rd, 6th, and 12th week. Refer to Appendix A for a detailed step-by-step procedure.

MATERIALS

Based on the review of numerous material compatibility studies, only the raw materials used in the construction of fuel system components were tested, rather than actual components. Testing raw materials allows for much broader coverage than testing components. For instance, the results from a test on mild steel could cover fuel lines, fuel rails, tanks, and injectors of any manufacturer using that material. This was determined to be more practical than testing each of the components individually. Also, many industry-accepted standard tests require specimens of specific dimensions that would be difficult to obtain from actual components.

The list of materials used in the fuel systems of automobiles from the 1970's forward, marine engines, non-road engines, and fuel dispensing equipment is immense. It would be virtually impossible for one study to test every material and combination of materials used. The materials list for this study was created from various sources such as literature reviews, manuals, and recommendations from fuel system and engine manufacturers. After the list of metals was assembled, it was forwarded to a list of fuel system engineers from several OEMs and Tier I and II suppliers for peer review. Finally, materials that are commonly used in flex-fuel vehicle fuel systems were removed from the list because they have already been proven compatible with any blend of ethanol from 0 to 85%. The 19 materials included in the study are listed below.

- brass 260
- brass 360
- cast iron
- copper 110
- 6061 aluminum
- 3003 aluminum
- cast aluminum mic 6
- 60/40 tin/lead solder
- 1018 steel
- 1018 steel tin plated
- 1018 steel nickel plated
- 1018 steel zinc plated
- 1018 steel zinc tri-chromate plated (hexavalent)
- 1018 steel zinc di-chromate plated (hexavalent free)
- 1018 steel zinc-nickel plated
- terne plate
- Zamak 5
- magnesium AZ91D
- lead

After the testing had begun, magnesium AZ91D was identified by small engines manufacturers as an alloy used in many fuel systems. Because the addition of another sample was not possible due to size constraints of the oven, the Magnesium samples were tested at a later date with the materials from another study at a slightly higher temperature of 55 ± 2 °C.

SAE J1747 suggests a sample size of 1 x 4 x 0.125 in. strips for testing because three will fit in the test bottle and maintain a minimum surface area to fuel volume ratio of 0.2 cm²/ml. Due to the decision to test samples in three states, immersed, liquid/vapor, and vapor, this size would not allow sufficient space for the vapor sample. A new sample size of 1.5 x 1.5 x 0.125 in. was chosen instead. This allowed ample space in the test bottle for the vapor sample, while maintaining the minimum surface area to fuel volume ratio of 0.2 cm²/ml.

The raw materials for the samples were obtained in either 1.5 x 0.125 in. strips or 12 x 12 x 0.125 in. sheets. The test samples were cut from the strips or sheets using a horizontal band saw and then milled to the final dimension to ensure squareness and accuracy. After milling, a 9/32 in. hole was drilled to provide a means of suspending the sample. Next, an identification number was stamped on each sample to identify the material, the state in which it was to be tested, and the fuel it was to be tested in. All of the preparation work was done before sending out the samples that needed plating to ensure that the entire surface was covered. Finally, before testing, all samples were prepared in accordance with ASTM G1, *Standard practice for preparing, cleaning, and evaluating corrosion test specimens*.

TEST FUEL

The test fuel selection for this research was a major focus in the test plan development. In the review of the literature, several studies were identified in which it was difficult to determine the specific composition of the base gasoline or ethanol components. The test fuels used in this study were based on the test-fuel standard specified in SAE J1681, *Gasoline alcohol and diesel fuel surrogates for materials testing*. This paper was the result of a task force formed in the early 1990's for testing materials with methanol. Since then, the standard has been altered to include many fuels. The task force adopted ASTM Fuel C to use as a reference for gasoline in material compatibility testing. ASTM Fuel C represents a worst-case-scenario gasoline due to its composition of 50% iso-octane and 50% toluene. The iso-octane represents the alkane group that makes up 40 to 70% of gasoline (Harrigan, Banda, Bonazza, Graham, Slimp, 2000, p. 2). It is important that the alkane group is represented for two reasons. First, they make up a large percentage of gasoline and second, they can cause swelling in polymers (plastics and elastomers). The toluene represents the aromatic group that makes up 20 to 50% of gasoline (p.2). Aromatics can cause swelling in polymers, but they also help suspend alcohols within the fuel mixture.

Synthetic ethanol, not fuel-grade ethanol, should be used for materials testing because its known composition "will help to minimize some of the variables in the use of ethanol as a fuel test component" (Harrigan et al., p. 5). Many impurities can be found in fuel-grade ethanol including sulfuric acid, acetic acid, water, and sodium chloride. "The acid is formed either in the alcohol production process or due to oxidation of the alcohol during handling, transfer or storage" (p. 3). Water also is a by-product of production and can cause metal corrosion, especially when combined with sodium chloride. It is important that these are represented in the test fuel because they can cause material compatibility issues. As per SAE J1681, these impurities were added to the synthetic ethanol to form aggressive ethanol. Aggressive ethanol is a worst-case-scenario fuel that would still be acceptable under ASTM D4806, *Standard specification for denatured fuel ethanol for blending with gasoline for use as automotive spark-ignition engine fuel* (ASTM, 2006).

All fuels used in this study met SAE Standard J1681's criteria. The three test fuels used included

Surrogate gasoline [C] - ASTM Fuel C, 50/50 toluene iso-octane mixture (500 ml toluene and 500 ml iso-octane)

E10 fuel [C(E10)_A] - 90% Fuel C + 10% aggressive ethanol (450 ml toluene, 450 ml iso-octane, 100 ml aggressive ethanol)

E20 fuel [C(E20)_A] - 80% Fuel C + 20% aggressive ethanol (400 ml toluene, 400 ml iso-octane, 200 ml aggressive ethanol)

Aggressive ethanol consists of synthetic ethanol 816.00 g, de-ionized water 8.103 g, sodium chloride 0.004 g, sulfuric acid 0.021 g, and glacial acetic acid 0.061 g (SAE J1681 Appendix E.1.2).

APPARATUS

The samples were placed in 1 L, high-density polyethylene (HDPE) bottles for the immersion testing. A wide mouth design was selected to allow easy access to the test samples. A test stand to support the three samples, one immersed, one halfway immersed, and one suspended above the liquid in the vapors, was designed and fabricated due to the lack of a suitable commercially available unit (see Figure 1). These stands were constructed from HDPE due to its insulating properties and resistance to chemical attack. The test stands resembled a ladder with three, ¼ in. horizontal rungs running between two, ½ in. vertical rods. Samples were hung on the rungs by a 9/32 in. diameter hole and the entire assembly extended up to the neck of the bottle to facilitate easy removal, yet allowed the bottles to be sealed tightly.



Figure 1. Samples on test stand and bottle

An explosion-proof friction air oven was used to maintain the samples at 45 ± 2 °C. It uses the heat generated by circulating air to maintain the temperature instead of an element or a flame. This is very important when heating combustible liquids in the presence of oxygen due to the potential for an explosion if the vapors were to come into contact with an ignition source.

DATA COLLECTION

The samples weights and dimensions were recorded before being exposed to the test fluids. Along with weight, a photo was taken of each sample to provide a reference of the original color and surface texture. The color of the solution was also noted before each weekly fuel change. Throughout the 2016 hours of the test, data was collected after the 1st, 3rd, 6th, and 12th week. After a sample was removed from the test fuel, it was dried and weighed to determine any mass gains or losses. Next, the samples were cleaned by scrubbing with a bleach-free scouring powder and a soft bristle brush. This was done to remove any corrosion that occurred. After the corrosion was removed, the samples were weighed again to determine any mass losses.

Three primary pieces of measuring equipment were used to collect the data. A micrometer with a resolution

of 0.0001 of an inch was used to measure the dimensions of the test samples. A special micrometer with pointed anvils was used to measure corrosion pit depth and a Radwag WAX 220 analytical balance was used to measure the mass of the test samples. The balance has a linearity of $\nabla 0.0002$ g and a repeatability of 0.00015 g as outlined in the user's manual. Based on the resolution and linearity of the scale, a weight change in a specimen less than 0.0008 g could be due to scale error and should not be considered a measurable change.

DATA ANALYSIS

Two methods of determining the effects of the three blends of fuels were incorporated into this study. The first was visual examination of the samples for pitting, surface texture change, and discoloration. The test fuel was examined for color change and loose by-products each week. The second was the mass loss analysis as described in ASTM G1 Section 8.

VISUAL EXAMINATION

The first method of determining the effects of the different fuel blends was through a visual examination of each sample. When examining the samples visually, corrosion and its different states need to be defined.

According to the *1989 ASM International Handbook of Corrosion Data*, edited by Bruce D. Craig of Metallurgical Consultants Inc, uniform / general corrosion is defined as, "A form of attack that produced overall uniform wastage of the metal" (p.1). Pitting corrosion is defined as, "A high localized attack of the metal creating pits of varying depth, width, and number. Pitting may often lead to complete perforation of the metal with little or no general corrosion on the surface" (p.1).

The pictures of the samples before, during, and after were used to determine any discoloration, change in surface texture, or pitting. Pitting deeper than 0.025 mm was measured using a micrometer with pointed anvils. For the purpose of this study, discoloration was considered acceptable because fuel system components are generally not aesthetic parts. Also, light pitting (less than 0.025 mm) that did not result in loose by-products was considered acceptable. However, deep pitting (greater than 0.025 mm) or perforation was considered unacceptable. And, "heavy corrosion resulting in the production of loose corrosion by-products" was considered unacceptable, because these could potentially become lodged in fuel pumps, filters, injectors, etc... causing a failure (anonymous OEM fuel and corrosion engineer, personal communication, February 10, 2006).

MASS LOSS

To understand the effects of the different fuel blends in relation to time, the mass loss can be changed to a corrosion rate. The corrosion rate is a prediction of the amount of the material in millimeters per year (mm/yr), that a component would lose after being exposed to the fuel. This can be used to project the amount of time a component made of a particular material will last before failure.

The data gathered from the original dimensions and weights were used along with the data collected from weighing the samples throughout the test to calculate percent mass lost or gained. If uniform corrosion had occurred, the corrosion rate was calculated in millimeters per year. Only corrosion rates higher than 0.0025 mm/yr are reported in this study. Corrosion rates less than this require much longer immersion periods to determine. ASTM G31 recommends the following formula: $[\text{Hours} = 50 / (\text{corrosion rate in mm/yr})]$ to determine the length of time needed to accurately predict extremely low corrosion rates (ASTM G31, 8.11.4). Corrosion rates less than 0.0025 mm/yr represent an insignificant loss of material over a 20-year time span. If pitting had occurred, then the corrosion rate was not calculated due to the inconstant nature of pitting, which would result in an inaccurate corrosion rate (ASTM G1, 8.2). Instead, the pit depths were measured using a micrometer with pointed anvils and recorded.

Using the corrosion rate per year, perforation potential was calculated to determine if a leak would likely develop during a normal service life of 20 years. A material is considered acceptable if the corrosion rate allows a component to provide at least 20 years of service.

The mass-loss analysis involves calculating the corrosion rate from the data gathered on each sample. This data includes the time of exposure in hours, the surface area of each sample, the density of the material, and a constant to convert units. The corrosion rate formula as per ASTM G1 Section 8.1 is as follows.

$$\text{Corrosion Rate} = (K \times W) / (A \times T \times D)$$

K = a constant in ASTM G1 Section 8.1 (8.76×10^4)

T = time of exposure in hours

A = area in cm^2

W = mass loss in grams

D = density in g/cm^3

An example of the corrosion rate calculation on a sample of magnesium AZ91D exposed to test Fuel C(E10)_A at the liquid/vapor interface is shown below. The sample had a surface area of 28.326 cm^2 and weighed 4.0476 g before the soak process and 4.0194 g at the end of 2016 hours.

$$\text{Corrosion Rate} = (87600 \times -0.0282 \text{ g}) / (28.326 \text{ cm}^2 \times 2016 \text{ Hours} \times 1.77 \text{ g/cm}^3)$$

$$\text{Corrosion Rate} = -0.0244 \text{ mm/year}$$

The measure of mm/yr was chosen so that the time to corrode through materials of varying thickness could be determined. For example, if a 2.00 mm (0.080 in.) thick carburetor bowl made of magnesium AZ91D was exposed to the liquid/vapor interface of E10, it would take $(2.00 \text{ mm} / 0.0244 \text{ mm/yr})$ or 81.9 years to corrode through. This is well in excess of the expected 20-year service life.

RESULTS

Visual appearance and mass change data was used to verify if a material was compatible with the fuels. Also, the data from the E20 samples were compared to that of the E10 and Fuel C samples because the latter two represent approved fuels. If E20 did not cause any more significant changes than E10 or Fuel C, and met the criteria stated in the data analysis section of this paper, then a material was deemed compatible.

VISUAL CHANGE

Many of the materials showed discoloration after being exposed to the ethanol-blended fuels. In general, the samples exposed to E20 exhibited a greater degree of discoloration than the samples exposed to E10. Also, in all cases the sample that was completely immersed showed a greater degree of discoloration than the sample exposed only to the vapor. This could easily be seen on the liquid/vapor interface samples with the bottom half that was immersed being darker than the top half that was exposed to the vapor (see Figure 2). However, it should be noted that discoloration does not necessarily indicate a potential fuel system reliability problem. Also, because fuel system components are not aesthetic parts of the vehicle, discoloration is not a reason to deem a material incompatible.

Only one material, lead, showed discoloration after being exposed to Fuel C. Eleven materials, listed below, showed discoloration after being exposed to E10.

- 1018 steel
- copper
- lead
- 1018 nickel plated steel
- 1018 zinc-tri-chromate plated steel
- brass 260
- terne plate
- solder (60/40 tin/lead)
- Zamak 5
- cast iron
- magnesium AZ91D

Finally, fourteen materials showed discoloration after being exposed to E20. This included the same eleven materials that were discolored by E10 along with cast aluminum and brass 360. No discoloration of the test fuel was noted throughout the study.

Only Zamak 5, an alloy used in some carburetors, showed pitting when it was exposed to E10 and E20, but not Fuel C. The pitting was apparent on the liquid and liquid/vapor samples but not the vapor samples. Although the pitting could be seen with the naked eye, it was not easily measurable with a micrometer. Finally, there was a noticeable amount of loose corrosion by-product in the E10 and E20 Zamak 5 containers when the fuel was changed. This is unacceptable because the loose corrosion by-product could clog components in the fuel system. It should be noted that Zamak 5 is often plated to make it more corrosion resistant for fuel applications and the samples used in this study were not plated.

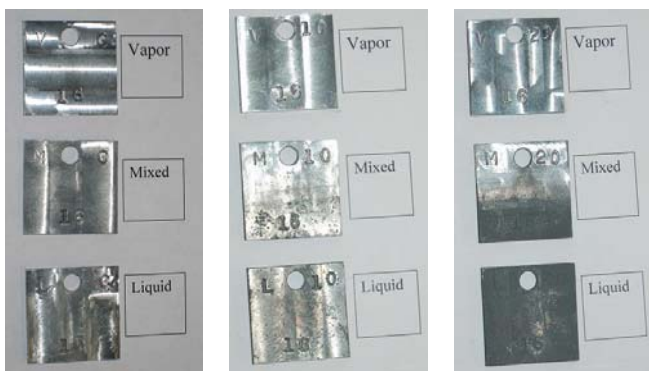


Figure 2. Zamak 5 samples after week 12: Fuel C (left), E10 (center), E20 (right)

MASS LOSS ANALYSIS

Several materials displayed measurable mass losses, 0.0008 g or greater, in one or all of the test fuels by the end of the study. Only two of these materials,

magnesium AZ91D and Zamak 5, displayed a mass loss large enough to potentially calculate an accurate corrosion rate or discuss further. The mass losses on the other materials were so small that they did not represent a concern, nor could an accurate corrosion rate be established as mentioned in the data analysis section of this paper. Refer to Appendix B for mass-loss data and corrosion-rate data for the individual samples.

Magnesium AZ91D is a die casting alloy that is commonly used in carburetors and diaphragm pumps. The magnesium AZ91D samples exhibited a mass loss in all three test fuels, with the largest loss occurring on the liquid/vapor sample immersed in Fuel C. This particular sample had a corrosion rate of -0.0248 mm/year. The samples exposed to E10 (liquid/vapor) and E20 (vapor) had similar, but slightly lower, corrosion rates of -0.0244 and -0.0224 mm/year, respectively. Even though these samples had a large enough mass loss to calculate a corrosion rate, the rate was small enough that potential problems in a 20-year life cycle would be unlikely and therefore was deemed acceptable.

Zamak 5, a material used in some early OEM carburetors and aftermarket carburetors, demonstrated significant mass loss, pitting, and loose corrosion by-products after being exposed to E10 and E20 during the 2016-hour study. The samples exposed to Fuel C did show a mass loss, but it was too small for an accurate corrosion rate to be calculated and therefore did not represent a potential problem. The Zamak 5 samples that were exposed to only vapor demonstrated little or no changes in all three fuels. However, the samples that were exposed to the liquid/vapor mix and liquid only showed significantly higher levels of corrosion as the ethanol concentration was increased. There was also pitting evident on the samples placed in the E10 and E20. The E20 samples also darkened to a greater extent than the E10 samples and had a greater mass loss. The mass losses of the samples exposed to E10 were 0.0393 g and 0.0568 g for the liquid/vapor and liquid samples, respectively. The samples exposed to E20 showed mass losses of 0.1318 g and 0.3384 g for the liquid/vapor and liquid samples, respectively. As the ethanol concentration increased from 10 to 20 %, the mass losses increased 3.5 and 6 times for the liquid/vapor sample and liquid sample, respectively (see Figure 3). The corrosion rate in mm/yr was not calculated for the E10 and E20 samples, as per ASTM G1 protocol, due to the fact that pitting was observed.

Overall, Zamak 5 was found to be incompatible with both E10 and E20 due to pitting and loose corrosion by-product that could potentially clog fuel system components. Zamak 5 also exhibited an unacceptable mass loss in E20. It should be noted that Zamak 5 is

often plated to make it more corrosion resistant and the samples used in this study were not plated.

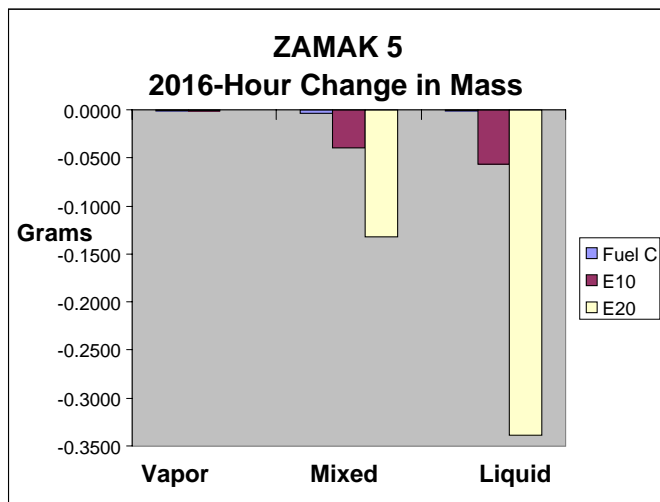


Figure 3. Zamak 5 mass loss comparison

CONCLUSIONS

This study tested and compared the effects of E20 to that of E10 and Fuel C on 19 different metals used in automotive, marine, and small engine fuel systems and fuel dispensing equipment. Eighteen of the nineteen metals were found to be compatible. One metal, Zamak 5, exhibited unacceptable levels of corrosion in both E10 and E20. It was deemed unacceptable in both fuels because of pitting and the formation of loose corrosion by-products that could clog fuel system components. Zamak 5 also exhibited an excessive mass loss when exposed to E20. Again, it should be noted that the Zamak 5 samples used in this study were not plated, which could be the reason that the corrosion problems found in this study with E10 are not seen on automobiles currently being used with E10.

Different degrees of discoloration were observed in many of the other materials. While many of the materials yielded higher discoloration as the ethanol concentration increased, they did not show signs of pitting, loose corrosion by-products in the test fluid, or have a mass loss that exceeded a rate that would cause a failure within a 20-year life cycle.

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TERMINOLOGY

E10 - Fuel consisting of 90% gasoline and 10% ethanol

E20 - Fuel consisting of 80% gasoline and 20% ethanol

ASTM Test Fuel C - Test Fuel C is composed of 50% toluene and 50% iso-octane.

Aggressive ethanol - Synthetic ethanol 816.00 g, de-ionized water 8.103 g, sodium chloride 0.004 g, sulfuric acid 0.021 g, glacial acetic acid 0.061 g (SAE J1681 appendix E.1.2)

C(E10)_A - Fuel consisting of 90% ASTM test Fuel C and 10% aggressive ethanol

C(E20)_A - Fuel consisting of 80% ASTM test Fuel C and 20% aggressive ethanol

CONTACT

Department of Automotive Engineering Technology
Minnesota State University, Mankato
205 Trafton Science Center East
Mankato, MN 56001
Phone: 507-389-6383

APPENDIX A



Minnesota Center for Automotive Research

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FROM Bruce Jones, Gary Mead, & Paul Steevens
Minnesota Center for Automotive Research
Minnesota State University, Mankato
Trafton Science Center 205E
Mankato, MN 56001
(507) 389-6383
(507) 389-5002 (fax)

RE E20 Material Compatibility Testing Procedure - Metals

Introduction

This document is intended to outline the material compatibility testing procedures used by the Minnesota Center for Automotive Research (MnCAR) for the purpose of measuring the material compatibility characteristics of metals commonly found in automotive fuel systems.

Standards Used

Proposed testing will follow the procedures outlined in SAE J1747, J1681, and ASTM G1, G31 for immersion testing of metals.

SAE J1747 (Dec94): *Recommended Methods for Conducting Corrosion Tests in Gasoline/Methanol Fuel Mixtures*

SAE J1681 (Jan00): *Gasoline, Alcohol, and Diesel Fuel Surrogates for Materials Testing*

ASTM G1-03: *Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens*

ASTM G31-72 (Re-approved 2004): *Standard Practice for Laboratory Immersion Corrosion Testing of Metals*

Properties Examined

mass loss/gain
pitting
appearance change

Metals to Test

Nineteen metals, nine specimens of each (3 specimens x 3 fuels): 1.5 x 1.5 x 0.125 in. squares.

brass 260	steel 1018	aluminum 6061
brass 360	steel 1018 nickel plated	aluminum 3003
cast iron	steel 1018 tin plated	cast aluminum mic 6
copper 110	steel 1018 zinc plated	magnesium AZ91D
lead	steel 1018 zinc di-chromate hexavalent free plated	
solder 60/40	steel 1018 zinc tri-chromate hexavalent chrome plated	
terne plate	steel 1018 zinc/nickel plated	
Zamak #5		

Test Fuels

Three test fuels used consisting of

C	Surrogate gasoline- "base" ASTM Fuel C 50/50 toluene iso-octane mixture (500 ml toluene and 500 ml iso-octane)
C(E10) _A	E10 fuel- 90% Fuel C + 10% aggressive ethanol (450 ml toluene, 450 ml iso-octane, 100 ml aggressive ethanol)
C(E20) _A	E20 fuel- 80% Fuel C + 20% aggressive ethanol (400 ml toluene, 400 ml iso-octane, 200 ml aggressive ethanol)

Aggressive ethanol consists of synthetic ethanol 816.00 g, de-ionized water 8.103 g, sodium chloride 0.004 g, sulfuric acid 0.021 g, and glacial acetic acid 0.061 g (SAE J1681 appendix E.1.2).

Required Material

- Containers for testing- HDPE (high density polyethylene) bottles with a 1L capacity.
- Oven capable of uniformly heating the HPDE bottles to 45 ± 2 °C for 2000 hours.
- HDPE stands to separate the specimens while in the bottles.
- Analytical scale with a resolution of 0.5 mg to weigh the test specimens.
- Micrometer with and accuracy ± 0.001 in to measure specimens.

Specimen Preparation

- Prepare the 1.5 x 1.5 x 0.125 in. specimens by stamping an ID number on each and drilling mounting holes where necessary. All preparation work on plated specimens must be done before plating so that no base material is exposed during testing (ASTM G31, 7.5, 7.6; SAE J1747, 4.1.4.1, 4.1.4.2).
- Clean each specimen by scrubbing with a bleach-free scouring powder (Bon Ami), followed by a water rinse, and finally a rinse in acetone. All handling of the specimen from this point out must be performed using gloves or forceps to avoid contamination (ASTM G31, 7.8).
- Record each specimen's weight using an analytical balance accurate to at least ± 0.5 mg (ASTM G31, 7.9).
- Measure the dimensions of each specimen using a micrometer accurate to at least 0.001 in. Calculate the surface area subtracting any holes (ASTM G31, 7.9).
- Photograph the specimens to show original color and surface texture.

Test Procedure

- Fill the 1 L HDPE bottles with 510 mL of the appropriate test fluid (SAE J1747, 4.1.3.1, 4.1.4.2).

NOTE: In order to ensure even heating and exposure, all specimens should be placed in, or removed from the test fluid at the same time. Also, all bottles should be placed in or removed from the oven at the same time.

- Place three specimens of the same material on the HDPE test stand. After all of the test stands are loaded, place all of them into their appropriate test bottles so that the bottom specimen is completely immersed, the second specimen halfway immersed, and the third just exposed to vapors. Place the lid on the bottle, but do not tighten at this time to avoid pressure build up (ASTM G31, 8.10.3; SAE J1747, 4.1.4.2).

3. Preheat the oven to 45 °C and maintain this temperature within ± 2 °C. Place all of the bottles in the oven at the same time and allow the temperature of the specimens to stabilize at 45 ± 2 °C. Once stabilized, fully tighten the lids on the bottles. This procedure will ensure pressure buildup in the bottles is minimized (SAE J1747, 4.1.3.2, 4.1.3.3, 4.1.5.4).

NOTE: During fuel change or measurement, always remove all of the bottles at the same time from the oven to avoid uneven exposure. Immediately remove all of the test stands/specimens from all of the bottles to allow them to cool and dry.

4. Change the test fluid weekly to minimize bulk solution composition changes, oxygen depletion, and to replenish ionic contaminants. Note any changes in the test fluid color or loose corrosion by-products weekly (ASTM 31, 4.1.4; SAE J1747, 4.1.2.2).

5. Continue heating the submerged metal specimens for 2000 hours, and take corrosion measurements after the 1st, 3rd, 6th, and 12th week (SAE J1747, 4.1.5.5).

Specimen Inspection and Cleaning Procedure

1. Perform inspection and measurement after the 1st, 3rd, 6th, and 12th week while replacing the test fluid (SAE J1747, 4.1.5.5).

2. Gloves or forceps must be used at all times when handling specimens to prevent contamination.

NOTE: Always remove all of the bottles at the same time from the oven to avoid uneven exposure. Immediately remove all of the test stands/specimens from all of the bottles to allow them to cool and dry.

3. Photograph the specimens after they are dried, and weigh them before cleaning to determine any mass gains (ASTM 31, 9.1).

4. Clean the specimens with a bristle brush and a bleach-free scouring powder. If heavily corroded areas can not be cleaned by brushing, air driven walnut shells are to be used (ASTM 31, 9.3.1).

5. After cleaning, weigh the specimens to determine mass loss. The mass loss will be the principle method for determining the level of corrosion for each material (ASTM 31, 10.1).

6. If any pitting is discovered, measure it using a micrometer with pointed anvils. Take a photograph of the sample to show the pitting. Do not establish a loss per year value, as pitting is rarely consistent (ASTM 31, 10.2.1).

7. If it appears that internal attack has not been a factor in the corrosion, then calculate the mass loss, in millimeters per year, with the following formula: $(K * W) / (A * T * D)$, where K = an ASTM constant (used to convert to millimeters per year), W = mass loss in grams to the nearest 0.001 g to correct for any losses during cleaning, A = area in cm^2 to the nearest 0.010 cm^2 , T = time in exposure in hours to the nearest 0.001 h, and D = density in g/cm^3 (ASTM 31, 11.2).

8. If testing is to continue, refer back to steps 1-5 of the Test Procedure section.

APPENDIX B MASS CHANGE AND CORROSION RATE

	Fuel C			Fuel E10			Fuel E20					
	Vapor	Mixed	Liquid	Vapor	Mixed	Liquid	Vapor	Mixed	Liquid			
Magnesium AZ91D	before	4.5660	4.2890	4.3270	before	4.5637	4.0476	4.2334	before	4.0248	4.3013	4.8323
Change in Mass (g)	after	4.5497	4.2589	4.3053	after	4.5402	4.0194	4.2108	after	3.9995	4.2790	4.8138
Corrosion Rate (mm/year)		-0.0163	-0.0301	-0.0217		-0.0235	-0.0282	-0.0226		-0.0253	-0.0223	-0.0185
		-0.0138	-0.0248	-0.0179		-0.0202	-0.0244	-0.0191		-0.0224	-0.0185	-0.0150
1018 Steel	before	35.1726	35.1298	35.1278	before	35.1176	35.2521	35.1395	before	35.0740	35.3728	35.1840
Change in Mass (g)	after	35.1726	35.1297	35.1275	after	35.1179	35.2518	35.1403	after	35.0747	35.3721	35.1817
Corrosion Rate (mm/year)		NM	NM	NM		NM	NM	0.0008		NM	NM	-0.0023
		NC/M	NC/M	NC/M		NC/M	NC/M	NS		NC/M	NC/M	NS
Copper	before	39.8973	40.2768	39.9907	before	40.0638	40.1193	40.1089	before	40.1089	40.0810	39.9104
Change in Mass (g)	after	39.8969	40.2768	39.9906	after	40.0645	40.1172	40.1029	after	40.1086	40.0747	39.9016
Corrosion Rate (mm/year)		NM	NM	NM		NM	-0.0021	-0.0060		NM	-0.0063	-0.0088
		NC/M	NC/M	NC/M		NC/M	NS	NS		NC/M	NS	NS
Brass 360	before	38.0175	37.8497	38.0058	before	38.0088	37.9634	37.9290	before	38.0100	38.0215	37.9682
Change in Mass (g)	after	38.0170	37.8487	38.0061	after	38.0076	37.9608	37.9244	after	38.0093	38.0154	37.9605
Corrosion Rate (mm/year)		NM	-0.0010	NM		-0.0012	-0.0026	-0.0046		NM	-0.0061	-0.0077
		NC/M	NS	NC/M		NS	NS	NS		NC/M	NS	NS
Aluminum 3003	before	11.9894	12.0125	12.1421	before	12.0231	11.9805	12.0513	before	12.1142	12.1351	12.0491
Change in Mass (g)	after	11.9897	12.0130	12.1433	after	12.0231	11.9801	12.0515	after	12.1145	12.1343	12.0466
Corrosion Rate (mm/year)		NM	NM	0.0012		NM	NM	NM		NM	-0.0008	-0.0025
		NC/M	NC/M	NS		NC/M	NC/M	NC/M		NC/M	NS	NS

Key**Highlighted Cells**

Significant Change in Mass (> 0.0008 g)

NM Not Measurable because mass change was outside accuracy/precision capability of scale (< 0.0008 g)

NC/M Not Calculated because Mass change was outside accuracy/precision capability of scale

NC/P Not Calculated because Pitting was observed

NS Not Significant enough of a corrosion rate (< 0.0025 mm/yr)

	Fuel C			Fuel E10			Fuel E20					
	Vapor	Mixed	Liquid	Vapor	Mixed	Liquid	Vapor	Mixed	Liquid			
Aluminum 6061	before	12.5156	12.5067	12.5564	before	12.4225	12.4891	12.5206	before	12.5053	12.5671	12.5244
Change in Mass (g)	after	12.5147	12.5068	12.5563	after	12.4217	12.4885	12.5202	after	12.5047	12.5663	12.5228
Corrosion Rate (mm/year)		-0.0009	NM	NM		-0.0008	NM	NM		NM	-0.0008	-0.0016
		NS	NC/M	NC/M		NS	NC/M	NC/M		NC/M	NS	NS

Lead	before	51.7746	51.0825	51.6412	before	52.6858	52.6618	51.7176	before	51.0475	52.1925	52.2263
Change in Mass (g)	after	51.7684	51.0756	51.6332	after	52.6845	52.6544	51.7223	after	51.0411	52.1842	52.2085
Corrosion Rate (mm/year)		-0.0062	-0.0069	-0.0080		-0.0013	-0.0074	0.0047		-0.0064	-0.0083	-0.0178
		NS	NS	NS		NS	NS	NS		NS	NS	NS

1018 Nickel Coated Steel	before	35.8358	36.1278	35.7120	before	35.8500	36.0347	35.7876	before	35.6869	35.6886	36.0737
Change in Mass (g)	after	35.8354	36.1274	35.7130	after	35.8499	36.0342	35.7856	after	35.6866	35.6861	36.0707
Corrosion Rate (mm/year)		NM	NM	0.0010		NM	NM	-0.0020		NM	-0.0025	-0.0030
		NC/M	NC/M	0.0001		NC/M	NC/M	NS		NC/M	NS	NS

1018 Zinc Di-Chromate (Hexavalent Free) Coated Steel	before	35.5065	35.5188	35.3381	before	35.6685	35.8266	35.5859	before	35.9923	35.6300	36.1662
Change in Mass (g)	after	35.5049	35.5160	35.3361	after	35.6679	35.8250	35.5825	after	35.9916	35.6265	36.1593
Corrosion Rate (mm/year)		-0.0016	-0.0028	-0.0020		NM	-0.0016	-0.0034		NM	-0.0035	-0.0069
		NS	NS	NS		NC/M	NS	NS		NC/M	NS	NS

1018 Zinc Tri-Chromate (Hexavalent Chrome) Coated Steel	before	35.2673	35.5167	35.0492	before	35.5073	35.2863	35.4092	before	35.3760	35.4253	35.0952
Change in Mass (g)	after	35.2670	35.5162	35.0496	after	35.5077	35.2851	35.4063	after	35.3761	35.4233	35.0891
Corrosion Rate (mm/year)		NM	NM	NM		NM	-0.0012	-0.0029		NM	-0.0020	-0.0061
		NC/M	NC/M	NC/M		NC/M	NS	NS		NC/M	NS	NS

Key	
Highlighted Cells	Significant Change in Mass (> 0.0008 g)
NM	Not Measurable because mass change was outside accuracy/precision capability of scale (< 0.0008 g)
NC/M	Not Calculated because Mass change was outside accuracy/precision capability of scale
NC/P	Not Calculated because Pitting was observed
NS	Not Significant enough of a corrosion rate (< 0.0025 mm/yr)

	Fuel C			Fuel E10			Fuel E20					
	Vapor	Mixed	Liquid	Vapor	Mixed	Liquid	Vapor	Mixed	Liquid			
1018 Zinc Coated Steel	before	35.8451	36.0073	36.0557	before	35.7778	35.8873	35.6474	before	35.4252	35.8586	35.5483
Change in Mass (g)	after	35.8451	36.0061	36.0542	after	35.7776	35.8864	35.6462	after	35.4251	35.8573	35.5467
Corrosion Rate (mm/year)		NM	-0.0012	-0.0015		NM	-0.0009	-0.0012		NM	-0.0013	-0.0016
		NC/M	NS	NS		NC/M	NS	NS		NC/M	NS	NS
1018 Tin Coated Steel	before	35.9483	35.4653	35.9028	before	35.5163	36.0325	35.9464	before	35.7825	35.7930	35.8908
Change in Mass (g)	after	35.9473	35.4636	35.9012	after	35.5165	36.0306	35.9444	after	35.7826	35.7917	35.8895
Corrosion Rate (mm/year)		NS	NS	NS		NC/M	NS	NS		NC/M	NS	NS
		NS	NS	NS		NC/M	NS	NS		NC/M	NS	NS
1018 Zinc/Nickel Coated Steel	before	35.8610	35.8227	36.1638	before	35.9817	35.9930	35.8024	before	36.2028	36.0410	35.9459
Change in Mass (g)	after	35.8598	35.8216	36.1621	after	35.9812	35.9922	35.8000	after	36.2024	36.0385	35.9401
Corrosion Rate (mm/year)		NS	NS	NS		NC/M	NS	NS		NC/M	NS	NS
		NS	NS	NS		NC/M	NS	NS		NC/M	NS	NS
Brass 260	before	38.7669	38.8832	38.8826	before	38.8417	38.8662	38.8704	before	38.8192*	38.7110	38.8467
Change in Mass (g)	after	38.7658	38.8870	38.8820	after	38.8414	38.8635	38.8658	after	38.8179	38.7061	38.8380
Corrosion Rate (mm/year)		NS	NS	NC/M		NC/M	NS	NS		NS	NS	NS
		NS	NS	NC/M		NC/M	NS	NS		NS	NS	NS
Terne Plate	before	17.0354	16.7382	17.1234	before	16.8331	16.7840	16.3970	before	16.7526	16.4992	16.5597
Change in Mass (g)	after	17.0320	16.7336	17.1201	after	16.8302	16.7778	16.3855	after	16.7514	16.4877	16.5404
Corrosion Rate (mm/year)		NS	NS	NS		NS	NS	NS		NS	NS	-0.0036
		NS	NS	NS		NS	NS	NS		NS	NS	-0.0036

Key	
Highlighted Cells	Significant Change in Mass (> 0.0008 g)
NM	Not Measurable because mass change was outside accuracy/precision capability of scale (< 0.0008 g)
NC/M	Not Calculated because Mass change was outside accuracy/precision capability of scale
NC/P	Not Calculated because Pitting was observed
NS	Not Significant enough of a corrosion rate (< 0.0025 mm/yr)

* For Brass 260, the "before" weight measurement was lost. The week 1 data has been substituted for the before measurement.

	Fuel C			Fuel E10			Fuel E20					
	Vapor	Mixed	Liquid	Vapor	Mixed	Liquid	Vapor	Mixed	Liquid			
Solder 60Tin/40Lead	before	37.9335	38.5033	37.2485	before	38.9853	37.7488	39.8251	before	33.7774	40.0972	35.1012
Change in Mass (g)	after	37.9305	38.4997	37.2430	after	38.9837	37.7422	39.8182	after	33.7764	40.0919	35.0944
Corrosion Rate (mm/year)		-0.0030	-0.0036	-0.0055		-0.0016	-0.0066	-0.0069		-0.0010	-0.0053	-0.0068
		NS	NS	NS		NS	NS	NS		NS	NS	NS
Zamak 5	before	32.0851	33.3180	29.1921	before	31.4755	27.6252	30.9663	before	32.2466	30.8127	28.0344
Change in Mass (g)	after	32.0842	33.3148	29.1913	after	31.4748	27.5859	30.9095	after	32.2462	30.6809	27.6960
Corrosion Rate (mm/year)		-0.0009	-0.0032	-0.0008		NM	-0.0393	-0.0568		NM	-0.1318	-0.3384
		NC/M	NC/M	NC/M		NC/M	NC/P	NC/P		NC/M	NC/P	NC/P
Cast Aluminum	before	12.2150	12.5064	11.2828	before	11.6278	11.8106	12.4761	before	12.2339	12.3675	11.5788
Change in Mass (g)	after	12.2133	12.5059	11.2823	after	11.6279	11.8113	12.4758	after	12.2341	12.3690	11.5811
Corrosion Rate (mm/year)		-0.0017	NM	NM		NM	NM	NM		NM	0.0015	0.0023
		NS	NC/M	NC/M		NC/M	NC/M	NC/M		NC/M	NS	NS
Cast Iron	before	33.6240	30.4415	32.7912	before	32.5202	30.5405	33.4194	before	34.3885	32.8555	32.7844
Change in Mass (g)	after	33.6238	30.4439	32.7944	after	32.5227	30.5450	33.4234	after	34.3925	32.8593	32.7890
Corrosion Rate (mm/year)		NM	0.0024	0.0032		0.0025	0.0045	0.0040		0.0040	0.0038	0.0046
		NC/M	NS	NS		NS	NS	NS		NS	NS	NS

Key	
Highlighted Cells	Significant Change in Mass (> 0.0008 g)
NM	Not Measurable because mass change was outside accuracy/precision capability of scale (< 0.0008 g)
NC/M	Not Calculated because Mass change was outside accuracy/precision capability of scale
NC/P	Not Calculated because Pitting was observed
NS	Not Significant enough of a corrosion rate (< 0.0025 mm/yr)