## The Effects of E20 on Elastomers Used in Automotive Fuel System Components

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### ABSTRACT

The focus of this study was to compare the effects of E20 versus E10 and gasoline on elastomers found in automotive, marine, and small engine fuel system components and fuel dispensing equipment. The elastomer 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 55 °C for 500 hours. Changes in the following properties were investigated before immersion, after immersion, and after dry-out: appearance, volume, weight, tensile strength, elongation, and hardness.

#### 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,  $C_2H_5OH$ , 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 from the early 1980s to current 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 compatibility, tailpipe and evaporative material 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 did 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

Six 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 procedures developed were a combination of SAE J1748 Methods for determining physical properties of polymeric materials exposed to gasoline/oxygenate fuel mixtures, ASTM D471-06 Standard test method for rubber property - effect of liquids, ASTM D412-06a Standard test methods for vulcanized rubber and thermoplastic elastomers tension, ASTM D3183-02 Standard practice for rubber preparation of pieces for test purposes from products, and ASTM D2240-04 Standard test method for rubber property - durometer hardness (SAE, 1998; ASTM, 2006, 2006, 2002, 2004). The test fuels were blended as per SAE J1681, Gasoline alcohol and diesel fuel surrogates for materials testing (SAE, 2000). SAE J1748 modifies ASTM D471 to make it fuel-testing specific. After formulating the test procedures to reflect specific equipment and resources, they were forwarded for peer review and altered according to suggestions from several OEM fuel system engineers and Tier I and II suppliers. As per SAE J1748 and ASTM D471, specimens were completely immersed for 500 hours at 55  $\pm$  2 °C before testing. The following properties were

measured before immersion, after immersion, and after dry-out: volume, weight, appearance, tensile strength, ultimate elongation, and hardness (durometer M).

The elastomers testing required two different shaped test specimens. The first was a 1 x 2 in. rectangle that was 0.080 in. thick. This specimen shape was used for the volume, weight, and appearance change testing. It will be referred to as the appearance specimen (see Figure 1). Five appearance specimens per fuel, per elastomer were used. The next specimen shape used was a Type C tensile dumbbell as specified by ASTM D412 (2006). These were used for tensile strength, ultimate elongation, and hardness testing. These specimens will be referred to as the tensile specimens. Ten tensile specimens per fuel, per elastomer were immersed. Five were used for wet testing and the other five were used for dry-out testing. Also, five specimens per elastomer that were not immersed were used as a reference.



Figure 1. Appearance specimens with ID tags ready for immersion

All testing and data collection took place in a room equipped with a ventilation system designed to handle The specimens were cut from hazardous fumes. molded test plagues in accordance with ASTM D3183 (2002). The volume of each appearance specimen was measured using the fluid displacement method in accordance with ASTM D471 (2006). The weight of each was measured with an analytical balance to  $\pm$  0.0001 g. They were also photographed to show pre-immersion color and surface texture. The tensile specimens' thickness and width were measured with a micrometer to ± 0.001 in. Hardness, durometer type M, was also measured on the clamping ends of the tensile specimens in accordance with ASTM D2240 (2004). After the initial measurements were made, the specimens were suspended via stainless steel wire and completely immersed in a glass container with one of the appropriate test fluids: C, C(E10)<sub>A</sub>, and C(E20)<sub>A</sub>. The containers were sealed and placed in an oven at 55 ± 2 °C for 500 hours. The test fluid was replaced daily for the first three days and then weekly thereafter. Any changes in the test fluid color or loose materials in the bottom of the containers were noted.

After 500 hours, the containers were removed from the oven and the specimens were placed in cool, clean fluid for 60 minutes to cool. The volume and weight of the appearance samples were measured in the same manner as the pre-immersion measurements. They were also photographed at this time to show the appearance changes due to immersion. The hardness was measured on the clamping end of the tensile specimens before they were pulled. One set of five immersion specimens per elastomer, per fuel was pulled to measure tensile strength and ultimate elongation. Also, one set of five specimens that had not been immersed was pulled as a reference. The pulls were made in accordance to ASTM D412 at a speed of 500 ± 50 mm/min (2006). All measurements were made immediately after the samples were removed from the fuel to minimize fuel dry-out.

The appearance specimens and remaining untested tensile specimens were placed in the oven to dry at  $55 \pm 2$  °C for 40 hours. After the 40-hour period, they were removed and measured again to obtain dry-out data. The specimens were measured in the same manner as the post-immersion measurements were taken. Refer to Appendix A for a detailed step-by-step procedure.

#### MATERIALS

Based on the review of numerous other material compatibility studies, only the raw materials that are 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 a particular elastomer could cover hoses, seals, and diaphragms 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 elastomers was assembled, it was forwarded to a group of fuel system engineers from several OEMs and Tier I and II suppliers for peer review. The eight elastomers tested in this study are listed below.

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- acrylic rubber (ACM) [Hytemp®]
- epichlorohydrin homopolymer (CO)
- epichlorohydnin ethylene oxide copolymer (ECO)
- polychloroprene (CR) [Neoprene®]
- nitrile rubber (NBR) [Buna N] with medium ACN content
- nitrile rubber (NBR) [Buna N] with high ACN content
- nitrile/PVC blend (OZO) [Paracril®]
- fluoroelastomer (FKM) with dipolymers of VF2/HFP and 65% fluorine [Viton® A]

These eight elastomers will be referred to by their two or three letter abbreviations for the remainder of this paper. Finally, several elastomers such as acrylic ethylene (AEM) [Vamac®], chlorinated polyethylene (CPE), chlorosulfonated polyethylene (CSM) [Hypalon®], hydrogenated nitrile rubber (HNBR), fluoroelastomer (FKM) with terpolymers of VF2/HFP/TFE and 68% fluorine [Viton® B], fluoroelastomer (FKM) with terpolymers of VF2/HFP/TFE and 70% fluorine [Viton® GFLT], and Santoprene® (PVDT) were not tested because they are commonly used in the fuel systems of flex-fuel vehicles and therefore have already been qualified for ethanol use.

#### 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 specimens were placed in 16-oz glass jars with Teflon® lined lids. Two jars per elastomer, per fuel were used for a total of 48 jars. Stainless safety wire was used to suspend the specimens, via 1/8 in. hole in the end of each one, so that they were completely immersed but did not rest on the bottom of the jar (see Figure 2).

An explosion-proof friction air oven was used to maintain the samples at  $55 \pm 2$  °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.



Figure 2. Specimens suspended from lid and glass jar

#### DATA COLLECTION

The weight, volume, and hardness of each specimen was recorded before being exposed to the test fluids, after exposure, and after dry-out. The tensile strength and elongation of the specimens was measured after the immersion period on non-immersed, immersed, and dryout specimens. The specimens were also photographed to show pre-immersion, post-immersion, and dry-out color and surface texture changes. The test fuel was also checked for color change and loose by-products.

The volume of each appearance specimen was measured using the fluid displacement method in accordance with ASTM D471 (2006). To measure the volume, a quad beam balance was used with a resolution of 0.01 g (see Figure 3). The specimens were first weighed in air and then weighed in their appropriate test fuels. Next, the specific gravity of each test fuel was measured using a hydrometer with a resolution of 0.001. Then, using the formula below with the weights and the density, the volume of each specimen was calculated.

$$V = (M_A - M_F) / d$$

V = volume (cc) $M_A = mass in air (g)$  $M_F = mass in fuel (g)$ d = density (g/cc)



Figure 3. Quad beam balance used for volume measurements

For weight change, a Radwag WAX 220 analytical balance was used to measure the mass of the test samples. The balance has a linearity of  $\forall$  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 that is under 0.0008 g could be due to scale error and should not be considered a measurable change. The specimens were measured before being immersed. Next, they were weighed immediately after the immersion by blotting them dry with a lint-free cloth and placing them in a sealed weighing bottle. Finally, they were weight after the dry-out.

A type M durometer was used to measure the hardness of the specimens for this study because it can be accurately used on thin specimens and its readings correlate to a type A durometer (see Figure 4). The durometer has a resolution of 1 hit point, and an accuracy of  $\pm 4$  hit points. The measurements were taken on the clamping ends of the tensile specimens before they were pulled. Six hits were made on each specimen in accordance with ASTM D2240 (2004). Measurements were taken on non-immersed, immersed, and dry-out samples.

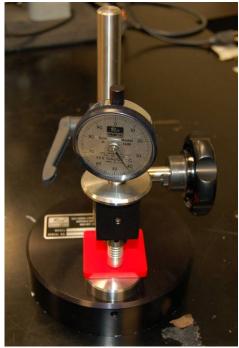


Figure 4. Durometer

The tensile specimens' thicknesses and widths were measured with a micrometer to ± 0.001 in. Three measurements were made for thickness and width, one at the center and one at each end of the reduced section. The medians of the three thickness and width measurements were used to calculate the cross sectional area of the specimen (ASTM D412). Next, tensile and elongation measurements were made with an Instron Model 3369 tensile machine with an 11,250 lbf load cell (see Figure 5). The specimens were pulled at a uniform rate of 500 ± 50 mm/min until they broke. Measurements were made on non-immersed, immersed, and dry-out specimens. All calculations on immersed and dry-out specimens, as per SAE J1748, were made using the original cross-sectional area measurements taken before immersion.



Figure 5. Tensile and Elongation measurement equipment

#### DATA ANALYSIS

Six properties were used in this study to investigate the effects of E20 as compared to E10 and gasoline. The six properties include visual appearance, volume, weight, tensile strength, elongation, and hardness. The data from the E20 specimens were compared to that of the E10 and Fuel C specimens because the latter two represent approved fuels. With the variety of applications that the elastomers used in this study represent, it was very difficult to distinguish at what point a change would represent a potential problem. For that reason, much of the data analysis was of a comparative nature between the results of E20 and the results of E10 and Fuel C. If E20 did not cause any more significant changes than E10 or Fuel C, then a material was deemed compatible.

#### VISUAL EXAMINATION

The first method of determining the effects of the different fuel blends was through a visual examination of each specimen. Pictures of the specimens were taken before immersion, after immersion, and after dry-out. The pictures were compared to note any changes in color, surface texture, glossiness, clouding, bubbling, tackiness, and cracking. Minor color and glossiness changes along with clouding were deemed acceptable. Changes in surface texture, cracking, bubbling, and tackiness were considered unacceptable because these could potentially lead to component failure. The test fuel was also checked after immersion for color change and loose by-products that could have been leached out of the specimens. Minor color changes in the test fluid were considered acceptable. It should be noted that

material leached out of elastomers can potentially cause problems in the fuel system and engine, but due to the budget of this study, the spent test fuel was not analyzed past color change for the presence of these. Loose byproducts were considered unacceptable because they represent a rapid degradation of the material along with the fact that they could potentially clog up other components.

#### VOLUME CHANGE

The volumes of the specimens were measured before immersion, after immersion, and after dry-out. The percent change in volume was calculated between preimmersion and post-immersion and between preimmersion and dry-out. The percent change in volume was used to determine if E20 caused a material to swell significantly differently than Fuel C or E10. Excessive swelling of a material can cause potential problems such as extruding, accelerated wear, and binding. Excessive shrinkage can also represent potential problems. Both excessive swelling and shrinking were considered unacceptable.

#### WEIGHT CHANGE

The weights of the specimens were measured before immersion, after immersion, and after dry-out. The preimmersion weight was compared to the post-immersion weight and the dry-out weight. From this data, the percent change in weight was calculated. The change in weight can indicate how much fuel was absorbed into the material for the pre-immersion and post-immersion comparison. While the change in weight when comparing pre-immersion to dry-out can indicate how much material was leached out of the specimens. The percent change in both states for E20, wet and dry, was compared to the percent change in both states for E10 and Fuel C while looking for significantly different changes that would be unacceptable.

#### HARDNESS CHANGE

The hardness of each specimen was measured before immersion, after immersion, and after dry-out. Using these measurements, the percent change in hardness comparing was calculated by pre-immersion measurements to the post-immersion measurements and pre-immersion measurements to drv-out measurements. The percent change in hardness of the E20 specimens was compared to that of the E10 and Fuel C specimens to see if any significant differences Excessive hardening or softening of an existed. elastomer can lead to potential problems and is not acceptable. If an elastomer becomes too hard it might lose flexibility or crack. On the other hand, if an elastomer becomes too soft it might collapse under load.

#### TENSILE STRENGTH AND ELONGATION

All of the tensile specimens were evaluated after the 500-hour immersion period ended. Three sets of specimens per elastomer, per fuel were pulled: nonimmersed, immersed, and dry-out. As with the other properties, the analyses was comparative between the E20 specimens and the E10 and Fuel C specimens while looking for significant differences. If E20 increased or reduced the tensile strength or elongation significantly more than E10 or Fuel C, it was considered unacceptable. Excessive reductions in tensile strength can lead to a component failure because the material will not be able to handle the intended loads. Also, significant increases in tensile strength, which typically result in a loss of elongation, can cause potential problems, too.

### RESULTS

Visual appearance, mass, volume, tensile, elongation, and hardness change data was used to verify if a material was compatible with the fuels. For an elastomer to be considered compatible in E20, it must not undergo significantly different changes than it would in gasoline or E10. The tensile strength, ultimate elongation, and hardness can not be reduced to the point that a component made of these would fail. Also, volume and weight changes could not be such that a component failure would be caused. Finally, discoloration was deemed acceptable as long as it did not affect the component's function.

#### VISUAL CHANGE

All of the specimens were visually inspected throughout the course of the study. None of the specimens exhibited any changes in color, texture, or glossiness. Also, there were no sings of cracking, bubbling, or tackiness. The only visual change apparent in some of the specimens was a size change, which is discussed below in the volume change section.

In examining the spent fuel after each fuel change, no loose by-products were noticed in any of the containers. After the first day of immersion, five elastomers: CO, ECO, CR, NBR-M, and OZO discolored the fuel slightly, while NBR-H discolored the fuel to a greater extent (see Figure 6). Also, NBR-H was the only elastomer that exhibited a greater degree of fuel discoloration with both ethanol fuels than in Fuel C (see Figure 7). This discoloration difference between Fuel C and both ethanol fuels was not noticeable by the end of week 1 (see Figure 8). By the end of the first week of immersion, only three elastomers, CO, ECO, and NBR-H, discolored the fuel very slightly. One elastomer, ACM, caused test Fuel C to become cloudy for the first week (see Figure 9). No other cloudiness was noticed in any other test fuel. Overall, none of the fuel discolorations were deemed significant enough to raise any concerns.



Figure 6. Discoloration comparison of the fuel after day 1 (front row E20; middle row E10; back row Fuel C)



Figure 7. Fluid discoloration from NBR-H after day 1 (Fuel C, left; E10, middle; E20, right)



Figure 8. Fluid discoloration from NBR-H after week 1 (Fuel C, left; E10, middle; E20, right)



Figure 9. Cloudiness caused by ACM in Fuel C

## VOLUME CHANGE

All of the elastomers swelled to some extent after being immersed in each of the test fuels. Refer to Appendix B for a graph comparing the pre-immersion and postimmersion volumes and Appendix D for the data. Elastomer ACM swelled the most in all three fuels (see Figure 10 for a visual comparison). With the exception of CR, all of the other elastomers swelled to a greater extent in the ethanol fuels than Fuel C. Only CR swelled more in Fuel C than in the ethanol blends. Several of the elastomers swelled a slight bit more with E20 than E10, but the difference was not large enough to be a concern. Only ECO swelled a noticeable extent larger in E20 than E10 or Fuel C but not a significantly large enough amount to raise concerns.

Seven of the eight elastomers shrank down below their pre-immersion size after the dry-out period. Only FKM remained larger than its original size in all three fuels, possibly due to an insufficient dry-out period as indicated by the weight change data. Refer to Appendix C for a graph comparing the pre-immersion and dry-out volumes and Appendix D for the data. No significant differences were found among any of the elastomers in any of the fuels after the dry-out period.



Figure 10. Visual volume change of ACM in E10 (top left specimen shows original size)

#### WEIGHT CHANGE

After the immersion period, the weights of all of the specimens in all three fuels increased. Refer to Appendix E for a graph comparing the pre-immersion and post-immersion weights and Appendix G for the data. Seven of the eight elastomers' weight increased more in the ethanol blends than in Fuel C. Only CR's weight increased more in Fuel C than in the ethanol blends. Only one elastomer, ACM, exhibited a noticeable difference in E20 than in E10, but it was not to a level that would warrant concern.

After the dry-out period, all but one elastomer, FKM, exhibited a weight loss in comparison to the preimmersion weights. In all three fuels, FKM weighed more after dry-out than it did before immersion. This was believed to be caused by an insufficient dry-out time for this particular elastomer. Refer to Appendix F for a graph comparing the pre-immersion and dry-out weights and Appendix G for the data. Overall, no significant differences existed between E20 and E10 or Fuel C after the dry-out.

#### HARDNESS CHANGE

In terms of hardness, all of the elastomers in all three fuels became softer after being immersed. Refer to Appendix H for a graph comparing the pre-immersion and post-immersion hardness and Appendix J for the data. One elastomer, ACM, became softer in Fuel C than in either of the ethanol fuels. Six of the elastomers: CO, ECO, NBR-M, NBR-H, OZO, and FKM became softer in the ethanol fuels than in Fuel C. None of the elastomers exhibited a significantly different change in hardness when immersed in E20 when compared to either E10 or Fuel C.

After the dry-out period, seven of the elastomers became harder than they were before immersion in all three fuels. Only FKM remained softer than its preimmersion state in all three fuels. As mentioned earlier, this was believed to be caused by an insufficient dry-out period for this elastomer. Refer to Appendix I for a graph comparing the pre-immersion and dry-out hardness and Appendix J for the data. The data was very similar for each type of elastomer and no significant differences were found between E20 and E10 or Fuel C.

#### TENSILE STRENGTH CHANGE

The tensile strength was reduced, when compared to non-immersed data, on all eight elastomers after being immersed in any of the three fuels. Refer to Appendix K for a graph comparing the non-immersion and postimmersion tensile strengths and Appendix M for the data. In seven of the elastomers, the ethanol blends reduced the tensile strength more than Fuel C did. Only CR was affected more by fuel C than the ethanol blends. None of the elastomers exhibited a significantly different change in tensile strength after being immersed in E20 than they did in either E10 or Fuel C.

The tensile strengths of all of the elastomers in all of the fuels increased after dry-out in comparison to the immersion tensile strengths. When the tensile strengths of the dry-out specimens were compared to the nonimmersed specimens, some were slightly higher and some were slightly lower in all three fuels with the exception of ACM soaked in E20. Refer to Appendix L for a graph comparing the non-immersion and dry-out tensile strengths and Appendix M for the data. The ACM specimens soaked in E20 after dry-out, exhibited an 18.5% loss in tensile strength while the specimens soaked in E10 and Fuel C exhibited an increase of 7.1% and 1.6%, respectively. Although a difference exists, it does not represent a concern because ACM lost approximately 80% of its tensile strength while immersed in any of the three fuels. If a component made of ACM was going to fail, it would likely happen while immersed, not after dry-out. Other than the difference noted with ACM in E20, none of the other elastomers exhibited a significant difference in E20 as compared to E10 or Fuel C after dry-out.

#### ELONGATION CHANGE

When the data was compared between non-immersed and immersed elastomer elongation, all of the elastomers exhibited a reduction in elongation in all three fuels. Refer to Appendix N for a graph comparing the non-immersion and immersion elongations and Appendix P for the data. Seven of the elastomers, with the exception of CR, were affected slightly more by the ethanol blends than Fuel C. Only one elastomer, ECO, was affected more by E20 than E10 or Fuel C, although the difference was only 6.6%, which did not raise any concerns. Other than ECO, none of the other elastomers exhibited a significant difference in elongation after being immersed in E20 than they did in E10 or Fuel C.

All of the dry-out specimens in all three fuels elongated more than the immersion specimens, but less than the non-immersion specimens, with the exception of ACM. Refer to Appendix O for a graph comparing the nonimmersion and dry-out elongations and Appendix P for the data. The ACM dry-out Fuel C specimens elongated slightly more than the non-immersed ACM Fuel C These were the only specimens that specimens. elongated more than the non-immersed specimens. The ACM dry-out E20 specimens exhibited a 31.4% reduction in elongation, while the E10 specimens exhibited a 4.8% reduction and the Fuel C specimens exhibited a 5.0% increase in elongation. Although a difference exists, it does not represent a concern because ACM's elongation was reduced by over 70% while immersed in any of the three fuels. If a component made of ACM was going to fail, it would likely happen while immersed, not after dry-out. Other than the difference noted with ACM in E20, none of the other elastomers exhibited a significant difference in E20 as compared to E10 or Fuel C after dry-out.

#### CONCLUSIONS

This study tested and compared the effects of E20 to that of E10 and Fuel C on eight different elastomers used in automotive, marine, and small engine fuel systems and fuel dispensing equipment. The following properties were measured before immersion, after and after dry-out: immersion. volume, weight, appearance, tensile strength, ultimate elongation, and hardness (durometer M). On many of the elastomers, both ethanol blends caused slightly different changes than did Fuel C. Only in a few cases did E20 cause a change greater than E10 or Fuel C. In each of these cases, the changes were not of a magnitude great enough to represent a concern. One elastomer, ACM, consistently had the greatest change in all three fuels for each of the properties examined with the exception of hardness through out the study.

#### ACKNOWLEDGEMENTS

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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, deionized 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)**<sub>A</sub> - Fuel consisting of 90% ASTM test Fuel C and 10% aggressive ethanol

**C(E20)**<sub>A</sub> - Fuel consisting of 80% ASTM test Fuel C and 20% aggressive ethanol

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### **APPENDIX A**



## **Minnesota Center for Automotive Research**

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**RE** E20 Material Compatibility Testing Procedures - Elastomers

#### Introduction

This document outlines the testing procedures used by the Minnesota Center for Automotive Research (MnCAR) for measuring material compatibility of elastomers commonly found in automotive fuel systems. The purpose of this testing was to determine if E20 had a larger negative impact on fuel system elastomers than gasoline or E10.

#### **Standards Used**

Proposed testing will follow the procedures outlined in

SAE J1748	Methods for determining physical properties of polymeric materials exposed to gasoline/oxygenate Fuel Mixtures (Jan 98)
SAE J1681	Gasoline, alcohol, and diesel fuel surrogates for material testing (Jan 00)
ASTM D471-06	Standard Test Method for Rubber Property – Effect of Liquids
ASTM D412-06a	Standard Test Methods for Vulcanized Rubber and Thermoplastic Elastomers - Tension
ASTM D3183-02	Standard Practice for Rubber – Preparation of Pieces for Test Purposes from Products
ASTM D2240-04	Standard Test Method for Rubber Property - Durometer Hardness
ASTM D618	Standard Practice for Conditioning Plastics for Testing
ASTM D543	Standard Practices for Evaluating the Resistance of Plastics to Chemical Reagents

#### **Properties examined**

weight change volume change appearance tensile percent change in elongation hardness (Durometer M)

These properties will be measured before soaking, after soaking, and after dry-out.

#### Rubbers to Test

ACM	acrylic rubber (Hytemp®)
CO	epichlorohydnin homopolymer
ECO	epichlorohydnin ethylene oxide copolymer
CR	polychloroprene (Neoprene®)
NBR-M	nitrile/buna N medium ACN
NBR-H	nitrile/buna N high ACN
OZO	paracril (nitrile/PVC blend)
FKM	fluoroelastomer with dipolymers of VF2/HFP 65% fluorine (Viton A®)

#### Test Fuels (SAE J1681)

Three test fuels will be used consisting of

- CSurrogate gasoline- "base" ASTM Fuel C 50/50 toluene iso-octane mixture (500 ml toluene and<br/>500 ml iso-octane)C(E10)<sub>A</sub>E10 fuel- 90% fuel C + 10% aggressive ethanol (450 ml toluene, 450 ml iso-octane, 100 ml
- $C(E10)_A$  E10 fuel- 90% fuel C + 10% aggressive ethanol (450 ml toluene, 450 ml iso-octane, 100 ml aggressive ethanol)
- C(E20)<sub>A</sub> 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).

#### **Specimen Preparation and Condition**

1. Prepare five specimens of each material being tested for each fuel and for each property.

**Note:** two extra sets of five tensile specimens of each material will be needed to determine non-immersion properties and dry-out changes since these specimens get destroyed during each phase of testing (SAE J1748 5.1.2).

2. Specimens for tensile tests will be molded following ASTM D3183 and cut to shape using Die C (ASTM D412 11.1).

#### **Specimen Sizes**

Specimens for Weight, Volume, and Appearance Change: Rectangle  $25 \times 50 \times 2.0 \text{ mm} \pm 0.1 \text{ mm} (1 \times 2 \times 0.08 \text{ in.} \pm 0.004 \text{ in.})$  (ASTM D471 9.1).

Specimens for Tensile Strength: Type C dumbbell tensile specimens  $115 \times 25 \times 2 \text{ mm} \pm 0.25 \text{ mm} (4.5 \times 1 \times 0.08 \text{ in.} \pm 0.008 \text{ in.})$  that neck down to 6 mm + 0.00 mm - 0.05 mm (0.250 in. + 0.000 in. -0.002 in.) (ASTM D412 11.1).

3. ASTM D 618 Procedure A will be used for conditioning

Condition specimens under 7mm (0.25 in.) thick for a minimum of 40 hours at 23 °C  $\pm$  2 °C at 50%  $\pm$  5% humidity. Allow adequate air circulation to all specimens by either hanging from metal clips or placing them on a wire screen with at least 25 mm (1 in.) of room between them and the bench (ASTM D618 8.1).

#### **Pre Immersion Measurements**

1. Prepare and condition specimens in the manner described above.

2. Photograph all specimens to show original appearance.

3. After conditioning, weigh each appearance specimen on an analytical balance in grams to four decimal places (SAE J1748 4.1.3).

4. Measure the dimensions of each specimen on the tensile specimens. Three measurements shall be made for thickness, one at the center, and one at the end of the reduced section. The median of the three shall be used. Specimen with a difference between the maximum and minimum thickness exceeding 0.08 mm (0.003 in.) shall be discarded. The width of the specimen shall be measured in the same three spots. Measure to the nearest  $\pm$  0.025 mm ( $\pm$  0.001 in.) (ASTM D412 11.1.2) (ASTM D638 10.1.1).

5. Measure the hardness, using the procedure described below, on of each tensile sample on the clamping ends in a manner that will not influence the tensile testing.

**NOTE:** The tensile sample is also being used for hardness testing due to limited oven space. Testing the hardness at the clamping ends before tensile testing should not affect the tensile tests.

6. Volume measurement (fluid displacement method) samples shall be measured in air to the nearest 0.01 g and then suspended in specific test fuel and re-weighed.

#### Immersion

1. Suspend the specimens in glass jars to avoid contact with the walls or bottom of the container using stainless steel wire and glass beads to separate each specimen. It may be necessary to attach small weights made from stainless steel to prevent floating (ASTM D471 10.1).

2. For the weight/ volume/appearance change specimens, fill the glass containers with a minimum of 166 mL of the appropriate test fluid per five specimens or until the fluid level is 3 cm above the top of the specimens to compensate for swelling (SAE J1748 4.1.2), (ASTM D471 10.1, 11.1, 12.1).

3. For tensile/hardness specimens, fill the glass containers with a minimum of 250 mL of the appropriate test fluid per five specimens, or until the fluid level is 3 cm above the top of the specimens to compensate for swelling (SAE J1748 4.1.2), (ASTM D471 15.2).

4. Seal the container to avoid evaporative losses.

- 5. Place the container in the oven and allow the test fuel to reach 55 °C ± 2 °C (131 °F) (SAE J1748 5.2).
- 6. Replace the fuel daily for the first three days and weekly thereafter (SAE J1748 4.1.2).
- 7. Continue exposure for 500 hours (SAE J1748 5.1.3).

#### Change in Weight, Appearance, and Volume (ASTM D471)

1. After soaking for the required length of time, remove the test specimens and allow them to cool to room temperature by placing them into a cool, clean portion of the test fluid for 30 to 60 minutes (ASTM D471 10.2).

#### Measure Weight

2. Blot dry with filter paper free of lint and foreign material and place in a tarred weighing bottle within 10 seconds to minimize evaporative weight loss. Weigh each specimen to the same level of precision as the pre-immersion weighing (SAE J1748 6.2, ASTM D471 10.2).

#### Note Appearance

3. Examine the appearance of each specimen noting any loss of gloss, developed texture, decomposition, discoloration, swelling, clouding, tackiness, rubberiness, crazing, bubbling, cracking, solubility, etc... Photograph any changes (ASTM D543 11.5).

#### Measure Volume (fluid displacement method)

4. Obtain the mass of each specimen by hanging it from a quad beam balance in a jar of specific test fuel. Weigh to the nearest 0.01 gram, and record the mass. Then blot dry the specimen and weigh in air at room temperature (ASTM D471 11.1).

5. Leave samples in open air environment for 4 hours before placing the specimens in an oven at 55 °C  $\pm$  2 °C (131°F) and let dry for 40 hours. Allow the specimens to cool to room temperature before final measurements are made (ASTM D471 14.2).

6. Measure the specimens in the same manner as pre-immersion. Calculate the mass of material extracted from the specimens and express it as a percent of the original mass (ASTM D471 14.2).

#### Durometer Hardness (ASTM D2240-04)

1. After soaking for the required length of time, remove the containers from the oven and place the specimens in clean room temperature fluid and allow them to be cooled for 30 to 60 minutes (ASTM D471 15.2).

**NOTE:** Hardness measurements will be made on the clamping ends of the tensile specimens before the tensile tests in a manner that will not influence the tensile testing.

2. Testing is to take place within 3 minutes, with the exception of the dry-out specimens, of removing the specimen from the room temperature fuel to minimize the effects of dry-out (SAE J1748 6.2.1).

3. The dry-out tensile/hardness specimens will be suspended at room temperature for 4 hours to dry. Then placed in an oven and baked at 55 °C  $\pm$  2 °C (131°F) for at least 40 hours. Allow the samples to cool to room temperature before testing (ASTM D471 15.4.2).

4. Durometer hardness shall be performed on a machine meeting all requirements of ASTM 2240-04. A type 3 operating stand with adjustable pneumatic dampening will be used with a type M durometers. The type M durometer has been chosen due to its ability to be used on thin test samples and correlation to type A durometers (ASTM D2240 9.1).

5. Vertical distance from the presser foot to the contact surface of the test specimen shall be  $25.4 \pm 2.5$  mm (1.00  $\pm$  0.100 in.).

6. Make six determinations of hardness at different positions on the specimen at least 0.80 mm (0.03 in.) apart (ASTM D2240 9.18).

7. Immediately proceed to step 4 of the tensile testing section.

#### Tensile Strength (ASTM D412-06a)

1. After soaking for the required length of time, remove the containers from the oven and place the specimens in clean room temperature fluid and allow them to be cooled for 30 to 60 minutes (ASTM D471 15.2).

2. Testing is to take place within 3 minutes, with the exception of the dry-out specimens, of removing the specimen from the room temperature fuel to minimize the effects of dry-out (SAE J1748 6.2.1).

3. The dry-out property tensile/hardness specimens will be suspended at room temperature for 4 hours to dry. Then placed in an oven and baked at 55 °C  $\pm$  2 °C (131°F) for 40 hours. Allow the samples to cool to room temperature before testing (ASTM D471 15.4.2).

4. Tensile testing shall be performed on a machine meeting all requirements of section 6 of ASTM D412.

5. Specimens shall be pulled at a uniform rate of 500 ± 50 mm/min (20 ± 2 in./min) until failure (ASTM D412 6.1).

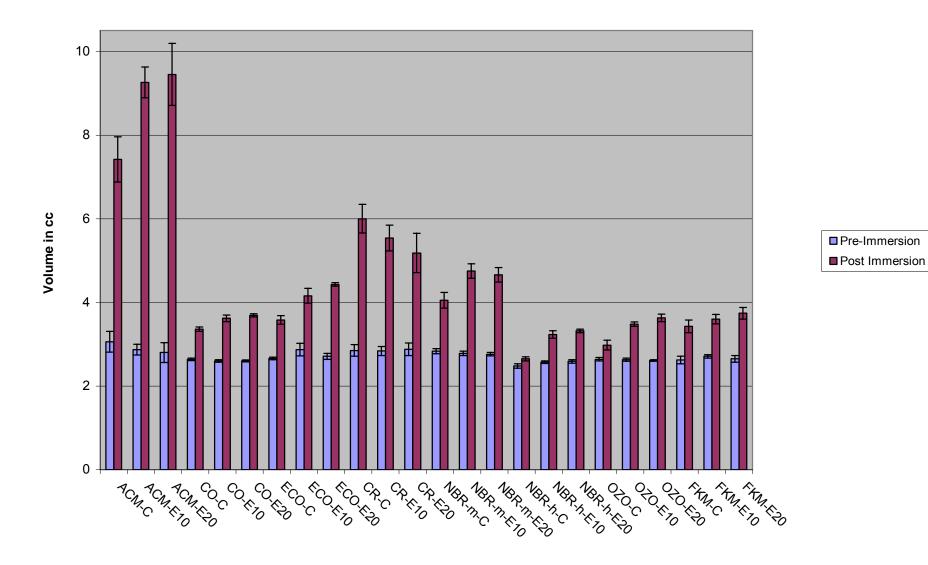
6. Record the force at the elongations specified for the test and at the time of rupture (ASTM D 412 12.1).

7. Base all calculations on the original measurements taken before immersion (SAE J1748 6.1.2).

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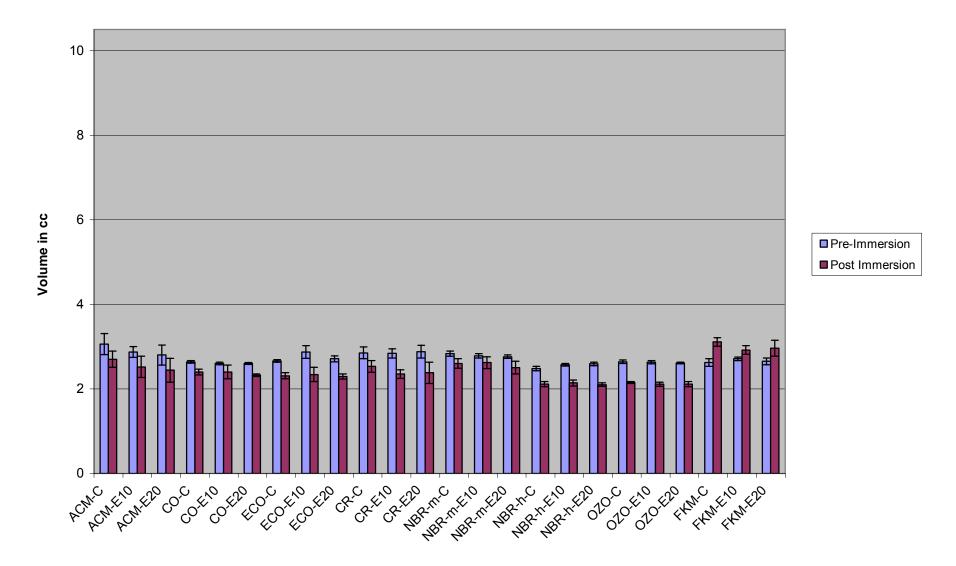
#### **APPENDIX B**

## **Elastomer Volume Change of Wet Samples**



#### **APPENDIX C**

## **Elastomers Volume of Dry Samples**



## APPENDIX D

# Minnesota State University, Mankato astomer Study Volume Results

E20 Elastomer Study Volume R Averages of 5 samples per fuel

Volume in CC - Wet					
		Fuel C	E10	E20	
ACM	Pre-Immersion	3.06	2.87	2.80	
	Post Immersion	7.42	9.26	9.45	
	% Change	142.5%	222.6%	237.5%	
Pre-Immersion	Std Dev	0.25	0.13	0.24	
Post Immersion	Std Dev	0.54	0.37	0.74	

Volume in CC - Wet					
		Fuel C	E10	E20	
СО	Pre-Immersion	2.64	2.60	2.60	
	Post Immersion	3.36	3.62	3.69	
	% Change	27.5%	39.2%	41.9%	
Pre-Immersion	Std Dev	0.03	0.03	0.02	
Post Immersion	Std Dev	0.05	0.08	0.04	

Volume in CC - Wet					
		Fuel C	E10	E20	
ECO	Pre-Immersion	2.66	2.87	2.71	
	Post Immersion	3.58	4.16	4.43	
	% Change	34.6%	44.9%	63.5%	
Pre-Immersion	Std Dev	0.03	0.15	0.07	
Post Immersion	Std Dev	0.10	0.18	0.04	

Volume in CC - Wet					
		Fuel C	E10	E20	
CR	Pre-Immersion	2.85	2.84	2.88	
	Post Immersion	6.00	5.54	5.18	
	% Change	110.5%	95.1%	79.9%	
Pre-Immersion	Std Dev	0.14	0.11	0.15	
Post Immersion	Std Dev	0.34	0.31	0.47	

Volume in CC - Dry				
		Fuel C	E10	E20
ACM	Pre-Immersion	3.06	2.87	2.80
	Post Immersion	2.70	2.52	2.44
	% Change	-11.8%	-12.2%	-12.9%
Pre-Immersion	Std Dev	0.25	0.13	0.24
Post Immersion	Std Dev	0.19	0.25	0.28

Volume in CC - Dry					
		Fuel C	E10	E20	
СО	Pre-Immersion	2.64	2.60	2.60	
	Post Immersion	2.40	2.40	2.32	
	% Change	-9.0%	-7.7%	-10.8%	
Pre-Immersion	Std Dev	0.03	0.03	0.02	
Post Immersion	Std Dev	0.07	0.16	0.03	

Volume in CC - Dry					
		Fuel C	E10	E20	
ECO	Pre-Immersion	2.66	2.87	2.71	
	Post Immersion	2.31	2.34	2.29	
	% Change	-13.2%	-18.5%	-15.5%	
Pre-Immersion	Std Dev	0.03	0.15	0.07	
Post Immersion	Std Dev	0.07	0.17	0.06	

Volume in CC - Dry					
		Fuel C	E10	E20	
CR	Pre-Immersion	2.85	2.84	2.88	
	Post Immersion	2.53	2.35	2.38	
	% Change	-11.2%	-17.3%	-17.4%	
Pre-Immersion	Std Dev	0.14	0.11	0.15	
Post Immersion	Std Dev	0.14	0.10	0.25	

## APPENDIX D (CONTINUED)

#### Minnesota State University, Mankato

E20 Elastomer Study Volume Results

Averages of 5 samples per fuel

Volume in CC - Wet					
		Fuel C	E10	E20	
NBR - medium	Pre-Immersion	2.83	2.78	2.76	
	Post Immersion	4.05	4.75	4.66	
	% Change	43.1%	70.9%	68.8%	
Pre-Immersion	Std Dev	0.06	0.05	0.04	
Post Immersion	Std Dev	0.19	0.17	0.17	

Volume in CC - Wet					
		Fuel C	E10	E20	
NBR - high	Pre-Immersion	2.48	2.57	2.59	
_	Post Immersion	2.65	3.23	3.32	
	% Change	6.9%	25.7%	28.2%	
Pre-Immersion	Std Dev	0.05	0.03	0.04	
Post Immersion	Std Dev	0.05	0.09	0.04	

Volume in CC - Wet				
		Fuel C	E10	E20
OZO	Pre-Immersion	2.64	2.63	2.61
	Post Immersion	2.98	3.48	3.63
	% Change	12.9%	32.3%	39.1%
Pre-Immersion	Std Dev	0.04	0.04	0.02
Post Immersion	Std Dev	0.12	0.05	0.09

Volume in CC - Wet				
		Fuel C	E10	E20
FKM	Pre-Immersion	2.62	2.71	2.65
	Post Immersion	3.43	3.60	3.74
	% Change	30.9%	32.8%	41.1%
Pre-Immersion	Std Dev	0.09	0.04	0.08
Post Immersion	Std Dev	0.15	0.11	0.14

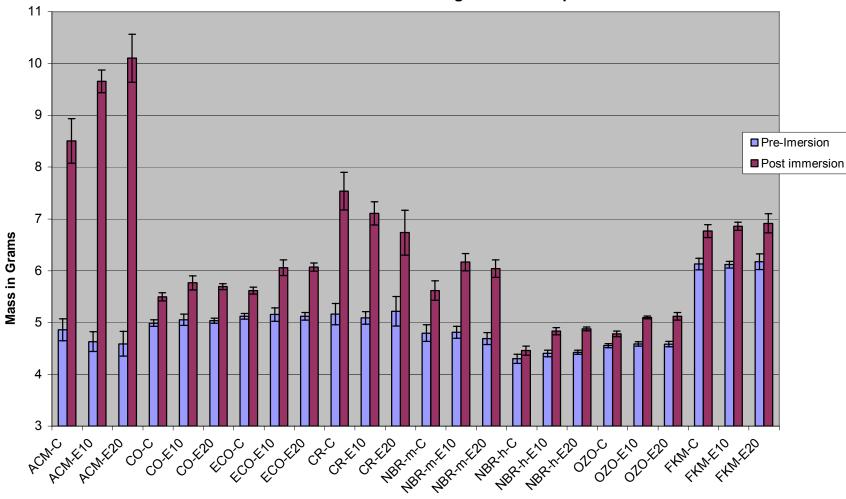
Volume in CC - Dry				
		Fuel C	E10	E20
NBR - medium	Pre-Immersion	2.83	2.78	2.76
	Post Immersion	2.60	2.62	2.50
	% Change	-8.1%	-5.8%	-9.4%
Pre-Immersion	Std Dev	0.06	0.05	0.04
Post Immersion	Std Dev	0.11	0.14	0.15

Volume in CC - Dry				
		Fuel C	E10	E20
NBR - high	Pre-Immersion	2.48	2.57	2.59
_	Post Immersion	2.11	2.14	2.10
	% Change	-14.9%	-16.7%	-18.9%
Pre-Immersion	Std Dev	0.05	0.03	0.04
Post Immersion	Std Dev	0.06	0.07	0.04

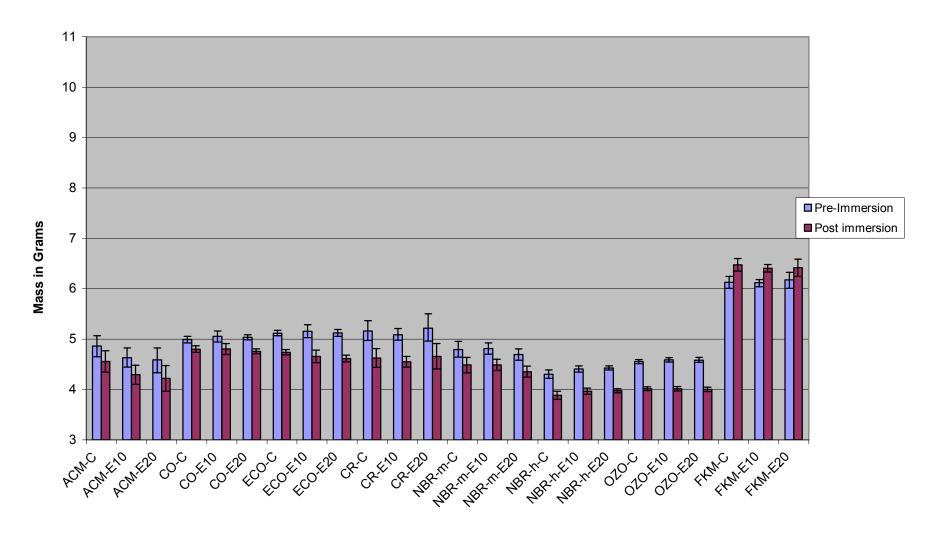
Volume in CC - Dry				
		Fuel C	E10	E20
OZO	Pre-Immersion	2.64	2.63	2.61
	Post Immersion	2.15	2.11	2.11
	% Change	-18.6%	-19.8%	-19.2%
Pre-Immersion	Std Dev	0.04	0.04	0.02
Post Immersion	Std Dev	0.02	0.05	0.06

Volume in CC - Dry				
		Fuel C	E10	E20
FKM	Pre-Immersion	2.62	2.71	2.65
	Post Immersion	3.11	2.92	2.96
	% Change	18.7%	7.7%	11.7%
Pre-Immersion	Std Dev	0.09	0.04	0.08
Post Immersion	Std Dev	0.10	0.10	0.19

#### APPENDIX E



## **Elastomer Mass Change of Wet Samples**



## **Elastomer Mass Change of Dry Samples**

2-22-2008

## **APPENDIX G**

# Minnesota State University, Mankato astomer Study Mass Results

E20 Elastomer Study

Averages of 5 samples per fuel

Mass in Grams - Wet				
		Fuel C	E10	E20
ACM	Pre-Immersion	4.859	4.632	4.588
	Post Immersion	8.506	9.657	10.101
	% Change	75.1%	108.5%	120.2%
Pre-Immersion	Std Dev	0.21	0.19	0.24
Post Immersion	Std Dev	0.43	0.22	0.46

Mass in Grams - Wet				
		Fuel C	E10	E20
СО	Pre-Immersion	4.989	5.053	5.036
	Post Immersion	5.495	5.765	5.693
	% Change	10.1%	14.1%	13.1%
Pre-Immersion	Std Dev	0.06	0.11	0.05
Post Immersion	Std Dev	0.08	0.14	0.06

Mass in Grams - Wet				
		Fuel C	E10	E20
ECO	Pre-Immersion	5.118	5.153	5.122
	Post Immersion	5.616	6.058	6.072
	% Change	9.7%	17.6%	18.6%
Pre-Immersion	Std Dev	0.06	0.13	0.07
Post Immersion	Std Dev	0.06	0.15	0.08

Mass in Grams - Wet				
		Fuel C	E10	E20
CR	Pre-Immersion	5.160	5.088	5.215
	Post Immersion	7.535	7.106	6.735
	% Change	46.0%	39.7%	29.1%
Pre-Immersion	Std Dev	0.21	0.12	0.29
Post Immersion	Std Dev	0.36	0.23	0.43

Mass in Grams - Dry				
		Fuel C	E10	E20
ACM	Pre-Immersion	4.859	4.632	4.588
	Post Immersion	4.554	4.294	4.221
	% Change	-6.3%	-7.3%	-8.0%
Pre-Immersion	Std Dev	0.21	0.19	0.24
Post Immersion	Std Dev	0.21	0.19	0.26

Mass in Grams - Dry				
		Fuel C	E10	E20
СО	Pre-Immersion	4.989	5.053	5.036
	Post Immersion	4.801	4.804	4.758
	% Change	-3.8%	-4.9%	-5.5%
Pre-Immersion	Std Dev	0.06	0.11	0.05
Post Immersion	Std Dev	0.06	0.11	0.05

Mass in Grams - Dry				
		Fuel C	E10	E20
ECO	Pre-Immersion	5.118	5.153	5.122
	Post Immersion	4.738	4.657	4.615
	% Change	-7.4%	-9.6%	-9.9%
Pre-Immersion	Std Dev	0.06	0.13	0.07
Post Immersion	Std Dev	0.05	0.13	0.07

Mass in Grams - Dry				
		Fuel C	E10	E20
CR	Pre-Immersion	5.160	5.088	5.215
	Post Immersion	4.625	4.549	4.658
	% Change	-10.4%	-10.6%	-10.7%
Pre-Immersion	Std Dev	0.21	0.12	0.29
Post Immersion	Std Dev	0.18	0.11	0.25

## APPENDIX G (CONTINUED)

#### Minnesota State University, Mankato

E20 Elastomer Study Mass Results

Averages of 5 samples per fuel

Mass in Grams - Wet				
		Fuel C	E10	E20
NBR - medium	Pre-Immersion	4.795	4.810	4.691
	Post Immersion	5.616	6.165	6.041
	% Change	17.1%	28.2%	28.8%
Pre-Immersion	Std Dev	0.16	0.11	0.11
Post Immersion	Std Dev	0.19	0.17	0.17

Mass in Grams - Wet				
		Fuel C	E10	E20
NBR - high	Pre-Immersion	4.300	4.404	4.426
	Post Immersion	4.457	4.833	4.880
	% Change	3.7%	9.7%	10.3%
Pre-Immersion	Std Dev	0.09	0.06	0.04
Post Immersion	Std Dev	0.09	0.07	0.04

Mass in Grams - Wet				
		Fuel C	E10	E20
OZO	Pre-Immersion	4.554	4.587	4.583
	Post Immersion	4.779	5.098	5.119
	% Change	4.9%	11.1%	11.7%
Pre-Immersion	Std Dev	0.04	0.04	0.05
Post Immersion	Std Dev	0.05	0.03	0.07

Mass in Grams - Wet				
		Fuel C	E10	E20
FKM	Pre-Immersion	6.128	6.116	6.174
	Post Immersion	6.765	6.859	6.915
	% Change	10.4%	12.1%	12.0%
Pre-Immersion	Std Dev	0.11	0.07	0.15
Post Immersion	Std Dev	0.13	0.08	0.18

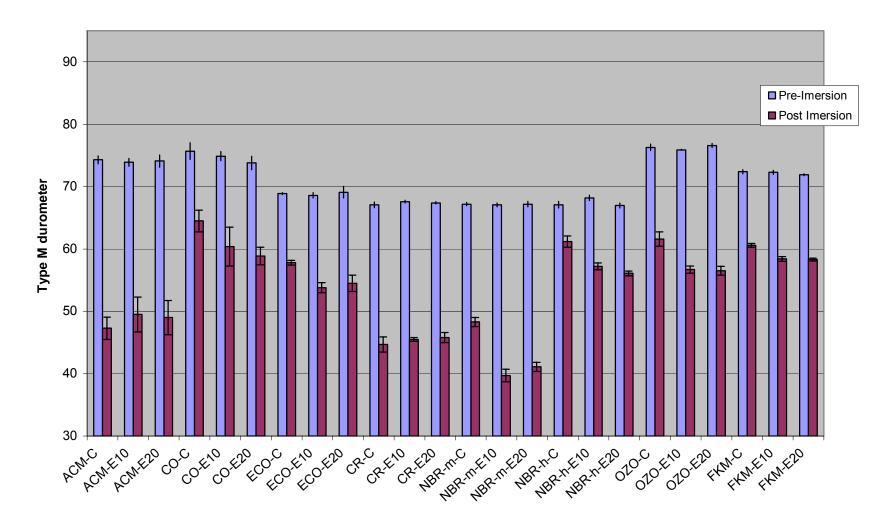
Mass in Grams - Dry				
		Fuel C	E10	E20
NBR - medium	Pre-Immersion	4.795	4.810	4.691
	Post Immersion	4.486	4.488	4.354
	% Change	-6.4%	-6.7%	-7.2%
Pre-Immersion	Std Dev	0.16	0.11	0.11
Post Immersion	Std Dev	0.15	0.11	0.11

Mass in Grams - Dry				
		Fuel C	E10	E20
NBR - high	Pre-Immersion	4.300	4.404	4.426
	Post Immersion	3.885	3.964	3.975
	% Change	-9.7%	-10.0%	-10.2%
Pre-Immersion	Std Dev	0.09	0.06	0.04
Post Immersion	Std Dev	0.08	0.06	0.04

Mass in Grams - Dry				
		Fuel C	E10	E20
OZO	Pre-Immersion	4.554	4.587	4.583
	Post Immersion	4.014	4.017	4.004
	% Change	-11.9%	-12.4%	-12.6%
Pre-Immersion	Std Dev	0.04	0.04	0.05
Post Immersion	Std Dev	0.04	0.04	0.04

Mass in Grams - Dry				
		Fuel C	E10	E20
FKM	Pre-Immersion	6.128	6.116	6.174
	Post Immersion	6.472	6.405	6.416
	% Change	5.6%	4.7%	3.9%
Pre-Immersion	Std Dev	0.11	0.07	0.15
Post Immersion	Std Dev	0.12	0.08	0.17

## **Elastomer Hardness Wet Samples**



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## 90 Pre-Imersion 80 Post Immersion 围 Ш **Type M Durometer** 70 Н Н 田 60 50 40 30 Charles and the second second

## **Elastomer Hardness Dry Samples**

### Minnesota State University, Mankato

E20 Elastomer StudyType M Durometer Hardness ResultsAverages of 6 points per sample, 5 samples per fuel E20 Elastomer Study

Type M Durometer Hardness - Wet				
		Fuel C	E10	E20
ACM	Pre-Immersion	74	74	74
	Post Immersion	47	50	49
	% Change	-36.5%	-32.4%	-33.8%
Pre-Immersion	Std Dev	0.61	0.63	0.98
Post Immersion	Std Dev	1.78	2.79	2.73

Type M Durometer Hardness - Wet				
		Fuel C	E10	E20
СО	Pre-Immersion	76	75	74
	Post Immersion	65	60	59
	% Change	-14.5%	-20.0%	-20.3%
Pre-Immersion	Std Dev	1.35	0.73	1.08
Post Immersion	Std Dev	1.73	3.13	1.41

Type M Durometer Hardness - Wet				
		Fuel C	E10	E20
ECO	Pre-Immersion	69	69	69
	Post Immersion	58	54	55
	% Change	-15.9%	-21.7%	-20.3%
Pre-Immersion	Std Dev	0.14	0.43	0.94
Post Immersion	Std Dev	0.36	0.82	1.29

Type M Durometer Hardness - Wet				
		Fuel C	E10	E20
CR	Pre-Immersion	67	68	67
	Post Immersion	45	46	46
	% Change	-32.8%	-32.4%	-31.3%
Pre-Immersion	Std Dev	0.45	0.22	0.19
Post Immersion	Std Dev	1.21	0.31	0.81

Type M Durometer Hardness - Dry				
		Fuel C	E10	E20
ACM	Pre-Immersion	73	74	74
	Post Immersion	77	79	78
	% Change	5.5%	6.8%	5.4%
Pre-Immersion	Std Dev	1.51	0.86	0.83
Post Immersion	Std Dev	0.63	0.87	0.29

Type M Durometer Hardness - Dry				
		Fuel C	E10	E20
СО	Pre-Immersion	75	75	74
	Post Immersion	79	81	80
	% Change	5.3%	8.0%	8.1%
Pre-Immersion	Std Dev	0.94	1.03	0.55
Post Immersion	Std Dev	0.81	1.12	0.58

Type M Durometer Hardness - Dry				
		Fuel C	E10	E20
ECO	Pre-Immersion	69	69	69
	Post Immersion	78	82	81
	% Change	13.0%	18.8%	17.4%
Pre-Immersion	Std Dev	0.07	0.74	0.46
Post Immersion	Std Dev	0.30	0.41	0.57

Type M Durometer Hardness - Dry				
		Fuel C	E10	E20
CR	Pre-Immersion	67	68	68
	Post Immersion	78	79	80
	% Change	16.4%	16.2%	17.6%
Pre-Immersion	Std Dev	0.41	0.39	0.43
Post Immersion	Std Dev	0.42	0.22	0.25

## **APPENDIX J (CONTINUED)**

### Minnesota State University, Mankato

E20 Elastomer Study Type M Durometer Hardness Results

Averages of 6 points per sample, 5 samples per fuel

Type M Durometer Hardness - Wet				
		Fuel C	E10	E20
NBR-medium	Pre-Immersion	67	67	67
	Post Immersion	48	40	41
	% Change	-28.4%	-40.3%	-38.8%
Pre-Immersion	Std Dev	0.29	0.30	0.44
Post Immersion	Std Dev	0.74	1.02	0.72

Type M Durometer Hardness - Wet				
		Fuel C	E10	E20
NBR-high	Pre-Immersion	67	68	67
	Post Immersion	61	57	56
	% Change	-9.0%	-16.2%	-16.4%
Pre-Immersion	Std Dev	0.52	0.42	0.40
Post Immersion	Std Dev	0.89	0.55	0.38

Type M Durometer Hardness - Wet				
		Fuel C	E10	E20
OZO	Pre-Immersion	76	76	77
	Post Immersion	62	57	57
	% Change	-18.4%	-25.0%	-26.0%
Pre-Immersion	Std Dev	0.52	0.07	0.32
Post Immersion	Std Dev	1.17	0.57	0.70

Type M Durometer Hardness - Wet				
		Fuel C	E10	E20
FKM	Pre-Immersion	72	72	72
	Post Immersion	61	58	58
	% Change	-15.3%	-19.4%	-19.4%
Pre-Immersion	Std Dev	0.32	0.32	0.14
Post Immersion	Std Dev	0.30	0.40	0.22

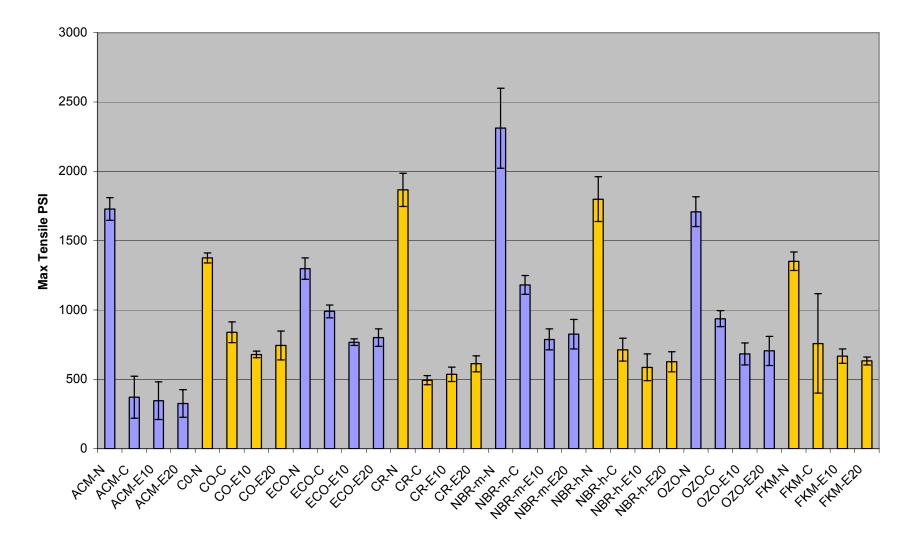
Type M Durometer Hardness - Dry				
		Fuel C	E10	E20
NBR-medium	Pre-Immersion	67	67	67
	Post Immersion	75	75	75
	% Change	11.9%	11.9%	11.9%
Pre-Immersion	Std Dev	0.00	0.30	0.07
Post Immersion	Std Dev	0.43	0.19	0.40

Type M Durometer Hardness - Dry				
		Fuel C	E10	E20
NBR-high	Pre-Immersion	67	67	67
	Post Immersion	79	79	80
	% Change	17.9%	17.9%	19.4%
Pre-Immersion	Std Dev	0.36	0.69	0.40
Post Immersion	Std Dev	0.27	0.30	0.32

Type M Durometer Hardness - Dry					
		Fuel C	E10	E20	
OZO	Pre-Immersion	76	76	76	
	Post Immersion	90	90	91	
	% Change	18.4%	18.4%	19.7%	
Pre-Immersion	Std Dev	0.45	0.84	0.32	
Post Immersion	Std Dev	0.41	0.52	0.26	

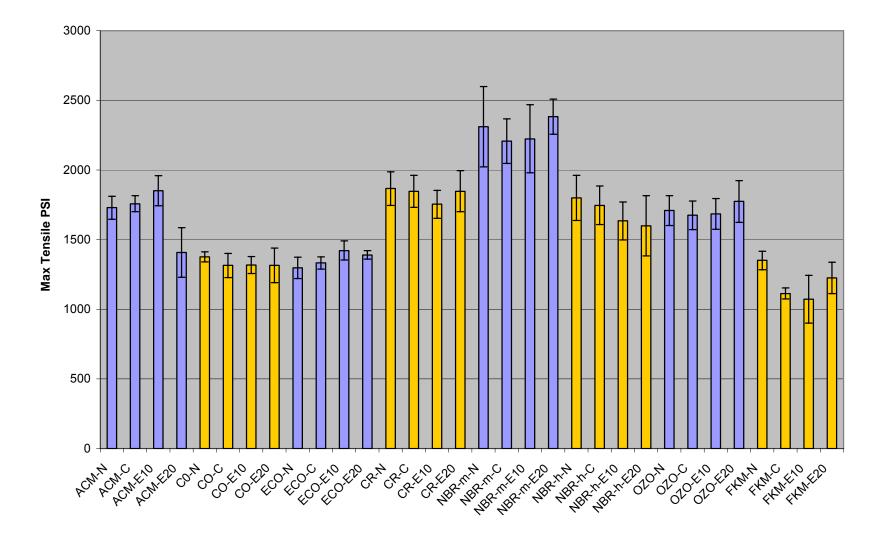
Type M Durometer Hardness - Dry					
		Fuel C	E10	E20	
FKM	Pre-Immersion	72	72	72	
	Post Immersion	65	66	66	
	% Change	-9.7%	-8.3%	-8.3%	
Pre-Immersion	Std Dev	0.17	0.30	0.93	
Post Immersion	Std Dev	0.75	0.18	0.30	

## **Elastomer Tensile Wet Results**



APPENDIX L

## **Elastomer Tensile Dry Samples**



## APPENDIX M

## Minnesota State University, Mankato E20 Elastomer Study Tensile Results Averages of 5 samples per fuel

Tensile Results - Wet					
ACM	No Soak	Fuel C	E10	E20	
Tensile PSI	1729	371	346	326	
Standard Deviation	82	152	136	100	
% Change		-79	-80	-81	

Tensile Results - Dry					
ACM	No Soak	Fuel C	E10	E20	
Tensile PSI	1729	1757	1851	1408	
Standard Deviation	82	57	107	178	
% Change		2	7	-19	

Tensile Results - Wet					
CO	No Soak	Fuel C	E10	E20	
Tensile PSI	1376	839	680	744	
Standard Deviation	36	75	25	104	
% Change		-73	-75	-76	

Tensile Results - Dry					
CO	No Soak	Fuel C	E10	E20	
Tensile PSI	1376	1314	1317	1315	
Standard Deviation	36	86	60	124	
% Change		-5	-4	-4	

Tensile Results - Wet					
ECO	No Soak	Fuel C	E10	E20	
Tensile PSI	1298	990	768	800	
Standard Deviation	77	47	23	63	
% Change		-24	-41	-38	

Tensile Results - Dry					
ECO	No Soak	Fuel C	E10	E20	
Tensile PSI	1298	1333	1422	1390	
Standard Deviation	77	44	69	30	
% Change		3	10	7	

Tensile Results - Wet					
CR No Soak Fuel C E10 E20					
Tensile PSI	1867	494	536	613	
Standard Deviation	121	32	53	58	
% Change		-74	-71	-67	

Tensile Results - Dry					
CR	No Soak	Fuel C	E10	E20	
Tensile PSI	1867	1846	1754	1847	
Standard Deviation	121	115	100	147	
% Change		-1	-6	-1	

## APPENDIX M (CONTINUED)

## Minnesota State University, Mankato E20 Elastomer Study Tensile Results Averages of 5 samples per fuel

Tensile Results - Wet					
NBR-medium	No Soak	Fuel C	E10	E20	
Tensile PSI	2311	1181	788	826	
Standard Deviation	288	68	75	106	
% Change		-49	-66	-64	

Tensile Results - Dry					
NBR-medium	No Soak	Fuel C	E10	E20	
Tensile PSI	2311	2207	2224	2382	
Standard Deviation	288	160	244	126	
% Change		-5	-4	3	

Tensile Results - Wet					
NBR-high	No Soak	Fuel C	E10	E20	
Tensile PSI	1799	713	587	627	
Standard Deviation	162	83	96	74	
% Change		-60	-67	65	

Tensile Results - Dry					
NBR-high	No Soak	Fuel C	E10	E20	
Tensile PSI	1799	1746	1634	1598	
Standard Deviation	162	138	135	216	
% Change		-3	-9	-11	

Tensile Results - Wet					
OZO	No Soak	Fuel C	E10	E20	
Tensile PSI	1709	937	683	705	
Standard Deviation	107	58	78	105	
% Change		-45	-60	-59	

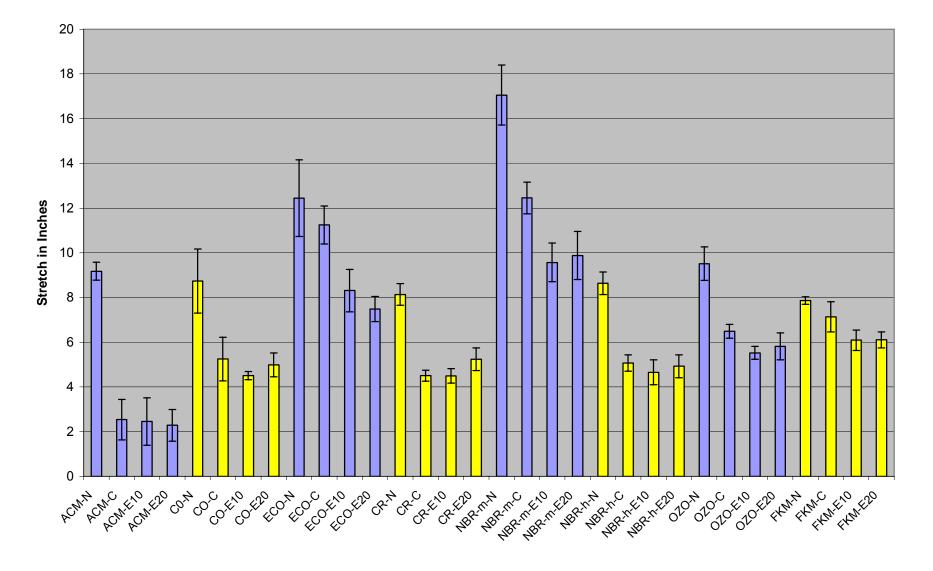
Tensile Results - Dry					
OZO	No Soak	Fuel C	E10	E20	
Tensile PSI	1709	1675	1685	1775	
Standard Deviation	107	102	111	150	
% Change		-2	-1	-4	

Tensile Results - Wet					
FKM	No Soak	Fuel C	E10	E20	
Tensile PSI	1351	758	667	632	
Standard Deviation	66	359	53	28	
% Change		-44	-51	-53	

Tensile Results - Dry					
FKM	No Soak	Fuel C	E10	E20	
Tensile PSI	1351	1113	1072	1225	
Standard Deviation	66	39	171	113	
% Change		-18	-21	-9	

#### **APPENDIX N**

## Elastomer Elongation of Wet Samples



#### **APPENDIX O**

20

Stretch in Inches

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## Elastomer Elongation of Dry Samples

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## **APPENDIX P**

## Minnesota State University, Mankato E20 Elastomer Study Elongation Results Averages of 5 samples per fuel

Elongation Results - Wet					
ACM	No Soak	Fuel C	E10	E20	
Extension at max load	9.176	2.536	2.451	2.283	
Standard Deviation	0.04	0.91	1.06	0.07	
% Change in Elongation		0.72	0.73	0.75	

Elongation Results - Dry					
ACM	No Soak	Fuel C	E10	E20	
Extension at max load	9.176	9.637	8.736	6.293	
Standard Deviation	0.04	0.74	0.52	0.61	
% Change in Elongation		0.05	-0.05	-0.31	

Elongation Results - Wet					
CO	No Soak	Fuel C	E10	E20	
Extension at max load	8.734	5.246	4.507	4.982	
Standard Deviation	1.43	0.98	0.18	0.53	
% Change in Elongation		-0.40	-0.48	-0.43	

Elongation Results - Dry					
CO	No Soak	Fuel C	E10	E20	
Extension at max load	8.734	7.750	6.876	7.250	
Standard Deviation	1.43	0.88	0.92	1.02	
% Change in Elongation		-0.11	-0.21	-0.17	

Elongation Results - Wet					
ECO	No Soak	Fuel C	E10	E20	
Extension at max load	12.448	11.247	8.309	7.486	
Standard Deviation	1.71	0.85	0.95	0.56	
% Change in Elongation		-0.10	-0.33	-0.40	

Elongation Results - Dry					
ECO	No Soak	Fuel C	E10	E20	
Extension at max load	12.448	11.890	10.256	11.198	
Standard Deviation	1.71	0.09	1.16	0.77	
% Change in Elongation		-0.05	-0.18	-0.10	

Elongation Results - Wet					
CR	No Soak	Fuel C	E10	E20	
Extension at max load	8.134	4.502	4.501	5.235	
Standard Deviation	0.48	0.25	0.32	0.50	
% Change in Elongation		-0.45	-0.45	-0.36	

Elongation Results - Dry						
CR	No Soak	Fuel C	E10	E20		
Extension at max load	8.134	7.472	7.443	7.247		
Standard Deviation	0.48	0.34	0.51	0.27		
% Change in Elongation		-0.08	-0.09	-0.11		

## APPENDIX P (CONTINUED)

## Minnesota State University, Mankato E20 Elastomer Study Elongation Results Averages of 5 samples per fuel

Elongation Results - Wet					
NBR-medium	No Soak	Fuel C	E10	E20	
Extension at max load	17.054	12.453	9.571	9.879	
Standard Deviation	1.34	0.07	0.86	1.08	
% Change in Elongation		-0.27	-0.44	-0.42	

Elongation Results - Dry						
NBR-medium	No Soak	Fuel C	E10	E20		
Extension at max load	17.054	15.901	14.854	15.410		
Standard Deviation	1.39	0.05	1.02	0.48		
% Change in Elongation		-0.07	-0.13	-0.10		

Elongation Results - Wet					
NBR-high	No Soak	Fuel C	E10	E20	
Extension at max load	8.636	5.065	4.652	4.927	
Standard Deviation	0.50	0.37	0.56	0.51	
% Change in Elongation		-0.41	-0.46	-0.43	

Elongation Results - Dry						
NBR-high	No Soak	Fuel C	E10	E20		
Extension at max load	8.636	7.344	7.071	7.138		
Standard Deviation	0.50	0.36	0.33	0.77		
% Change in Elongation		-0.15	-0.18	-0.17		

Elongation Results - Wet					
OZO	No Soak	Fuel C	E10	E20	
Extension at max load	9.512	6.488	5.525	5.816	
Standard Deviation	0.75	0.31	0.29	0.61	
% Change in Elongation		-0.32	-0.42	-0.39	

Elongation Results - Dry						
OZO	No Soak	Fuel C	E10	E20		
Extension at max load	9.512	6.547	6.501	6.909		
Standard Deviation	0.75	0.25	0.37	0.47		
% Change in Elongation		-0.31	-0.32	-0.27		

Elongation Results - Wet					
FKM	No Soak	Fuel C	E10	E20	
Extension at max load	7.865	7.141	6.089	6.105	
Standard Deviation	0.16	0.67	0.46	0.36	
% Change in Elongation		-0.09	-0.23	-0.22	

Elongation Results - Dry						
FKM	No Soak	Fuel C	E10	E20		
Extension at max load	7.865	7.362	7.372	7.842		
Standard Deviation	0.16	0.57	0.61	0.48		
% Change in Elongation		-0.06	-0.06	0.00		