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DET NORSKE VERITAS

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Final Report  
Effect of Ethanol Chemistry on SCC  
of Carbon Steel

Pipeline & Hazardous Materials Safety Administration  
U.S. Department of Transportation

Consolidated Program for Development of Guidelines for Safe and Reliable  
Pipeline Transportation of Ethanol Blends – WP#323  
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**Summary:**  
 This is the draft final report of the project on the effect of ethanol chemistry, through feedstock source and production method, (WP#323) of the Consolidated Program on Development of Guidelines for Safe and Reliable Pipeline Transportation of Ethanol Blends. The other two aspects of the consolidated program, ethanol batching and blending effects (WP#325) and monitoring (WP#327) are reported separately.

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## Executive Summary

Pipeline companies have a keen interest in assessing the feasibility of transporting fuel grade ethanol (FGE) and ethanol blends in existing pipelines. Previous field experience and laboratory research, funded by PRCI and API, has shown that steel can suffer stress corrosion cracking (SCC) when exposed to FGE in the presence of oxygen. Though cracking was prevalent under some conditions, variability in cracking susceptibility of steel was noted with different ethanol chemistries. A systematic examination of such variability was conducted in this study.

Unfortunately significant variability in the test results between the laboratories was noted. Two of the laboratories (SwRI and DNV) reported similar (but not identical) results, whereas Georgia Tech reported a highly different result. The following are the main points:

- Almost all ethanol samples supplied showed SCC in SwRI and DNV tests. However, DNV did not find SCC in one ethanol sample whereas SwRI observed minor SCC. This ethanol sample showed high OCP. This ethanol sample was later found to be contaminated with gasoline and therefore is not representative of FGE.
- The notched SSR test is being considered at present as a NACE standard test method. However, based on the experiences of the three labs in performing tests in fuel grade ethanol, the test parameters need to be better defined. Chloride concentration appears to be an important, but not the only, parameter. Notch root radius and methanol concentration may also be important. However, other factors, such as extension rates, specimen size, notch depth in addition to chloride is likely to play a critical role because Georgia Tech observed much lower maximum load to failure compared to the other two labs, even in air.
- The chemical analyses of the ethanol samples received to date do not reveal any startling differences. There are small differences in certain organic species, but these do not appear to be of significant consequence.
- The electrochemical fingerprinting method is capable of indicating differences between ethanol batches, but insufficient information exists to correlate these to chemical or SCC differences.
- Other ongoing test projects focused on developing a standardized test method should evaluate the test parameters to resolve test differences.
- Because the FGE samples represented 4 samples from producers, 2 samples from terminals that had exhibited SCC in the past, and one sample from Brazil and no significant differences in SCC tendency was observed within any of the laboratories,

a conclusion can be drawn that SCC of steel in FGE is not sensitive to feedstock differences or point of origin. Thus, the observations of SCC mainly in North American terminals (and not in Brazil or by ethanol producers) should be attributed to other factors not inherent in FGE.

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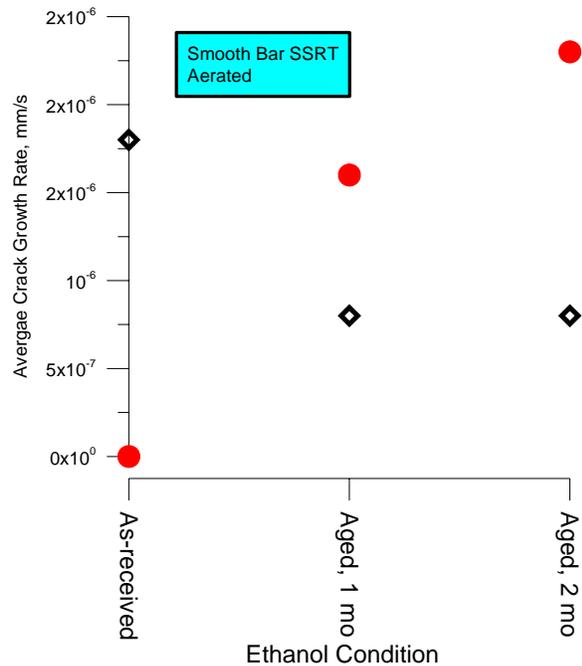
## 1.0 BACKGROUND

The U.S. Energy Independence and Security Act of 2007 established a nationwide renewable fuels standard, starting from 9 billion gallons (34 billion liters) of all biofuels in 2008 to 36 billion gallons (136 billion liters) in 2022. The most recent Renewable Fuel Standards issued by the U.S. Environmental Protection Agency [1] specifies a number of alternative biofuels, including corn-based ethanol, cellulosic ethanols, biodiesels, and other advanced biofuels that may be manufactured in the future using hitherto unknown technologies. At present, biofuel is first sent to blending terminals through tanker trucks, rail cars, and barges, where they are blended with gasoline or diesel and then sent to consumer filling stations via trucks. In the U.S. 67 percent of the ethanol is transported to blending terminals via trucks, 31 percent by rail cars, and 2 percent by barges [2]. The rail, truck, and barge transport modes are more costly and less efficient than pipeline transport for long distances. It has been estimated that for long-distance transportation of fuel, pipeline is less hazardous than trucks or rail cars based on frequency of fatalities per distance transported. While the ethanol pipeline infrastructure in the U.S. is still nascent, Brazil has a well-established ethanol pipeline already and is planning to expand this infrastructure even further.

Reliable and safe transportation of ethanol is critical to the viability of pipelines as the primary transportation mode. A 2003 survey of industry, reported in API Technical Report 939-D (2nd edition), indicates that stress corrosion cracking (SCC) has been observed primarily in user terminals exposed to ethanol products, but not in ethanol producer tanks, rail/tank car/shipping transportation, nor end-user systems (e.g., gas tanks). More recently, a short segment of pipeline transporting FGE in North America was reported to have suffered SCC. In contrast, Brazil, which has transported anhydrous and hydrous ethanol for many years, has not reported any SCC in their pipeline system. At present, there is an incomplete understanding of why the occurrence of SCC differs so significantly in different parts of the supply chain. One possible factor would be the ethanol chemistry.

Maldonado and Sridhar [3] observed that wet-milled ethanol from corn had a greater tendency to produce SCC than dry milled ethanol, despite containing seven times more water than the latter (Figure 1). They also found that one ethanol from a corn-based source did not cause any SCC and exhibited a high corrosion potential. This last observation suggests that SCC occurs at an intermediate potential range (between approximately below about -100 mV and +300 mV vs. Ag/AgCl/EtOH). The mechanistic reason for this is still being studied.





**Figure 2. SCC of steel in three lots of ethanol**

These observations prompted an investigation into the effects of ethanol chemistry on SCC. At the same time, there was also a desire to perform inter-laboratory comparison of test results for future standards development.

## 2.0 PROJECT OBJECTIVES

A Roadmap meeting, held on October 25-26, 2007, identified several gaps related to ethanol transportation in pipelines. A Consolidated Program, consisting of three projects, is being conducted to address several of these gaps:

WP#323 – Effect of ethanol source on SCC of carbon steel

WP#325 – Effect of Ethanol Blends and Batching Operations on Stress Corrosion Cracking of Carbon Steel

WP#327 – Monitoring Conditions Leading to SCC/Corrosion of Carbon Steel

The specific gaps addressed by WP#323 are shown in Table 1. The red dots indicate their priority/importance

**Table 1. Gaps addressed by the projects in this consolidated program**

Proposed Project	Gaps Addressed
WP#323	<ul style="list-style-type: none"> <li>• Limited understanding of the impact of mixing of ethanol from different sources ●●●●●●</li> <li>• No practical method for routine ethanol acceptability testing ●●●●●</li> <li>• Lack of understanding of how product composition changes during aging (with time, heat, length, etc.) ●●●●</li> <li>• Comparison between sugar and corn ethanol ●●●●</li> <li>• Current ASTM specification is based on vehicle performance. Need "API" specifications (transport based) for fuel-grade ethanol ●●●</li> <li>• Lack of knowledge about which constituents are driving factors for the characterization of ethanol ●●●</li> <li>• Defining the environment (finger printing, pH, electrodes, O<sub>2</sub>, etc.) is challenging because off-the-shelf probes do not exist ●●</li> <li>• Lack of knowledge of what contaminants cellulosic ethanol will contain ●</li> </ul>

The major objectives of this work package are to:

1. Develop an understanding of the factors that cause variation in the potency of Fuel Grade Ethanol (FGE) towards stress corrosion cracking (SCC) from different manufacturing processes (including sites, feed stocks, etc.).
2. Develop analytical methods that can be used to determine the degree of potency of a given source of ethanol in causing SCC for transportability decisions.
3. Determine whether the FGE specification needs to be modified to include transportation issues.

### 3.0 ETHANOL SAMPLES FROM PRODUCERS

Eight lots of FGE were received at DNV, Southwest Research Institute (SwRI), and Georgia Tech, as listed in Table 1. Last FGE lot (#10-1723397) was received in July 2010 and was tested for its potential to cause stress corrosion cracking (SCC) of pipeline steels. FGE samples were characterized for their chemical constituents. Slow strain rate tests (SSRT) with notched tensile samples, similar to the previously used procedures under this round robin program, were used for the latest FGE lot. These tests were conducted in general accordance with test parameters used in other labs.

**Table 2. Lot numbers used in this report and their corresponding round robin designation.**

GT Lot #	ROUND ROBIN DESIGNATION
Lot-2	1524407
Lot-3	1526470
Lot-4	1527139
Lo- 5	1531177
Lot-6	1602554
Lot-7	1602553
Lot-8	1641499
Lot-9	10-1723397

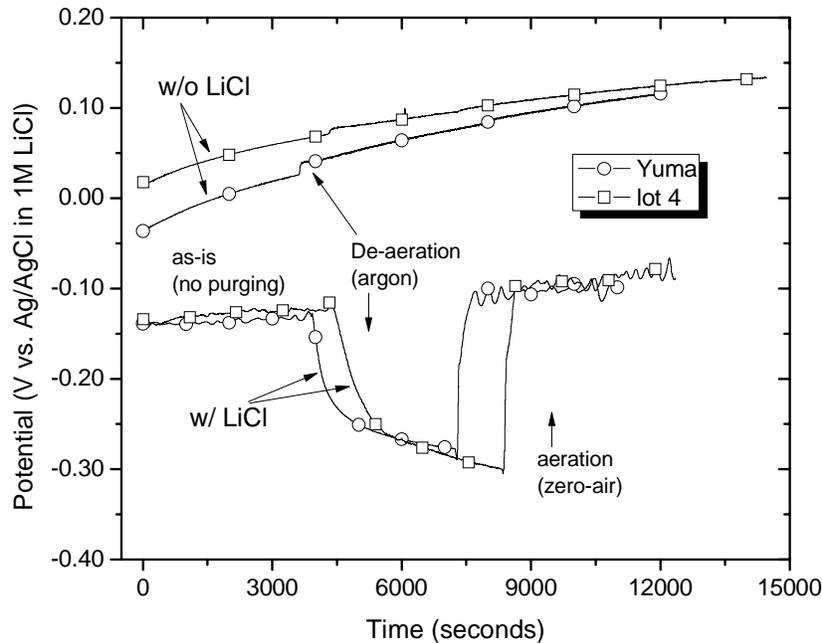
These ethanol lots were received at an intermediate distribution point, labeled only with lot numbers, and dispersed to the three laboratories for the round-robin tests. Chemical analyses were performed only by Georgia Tech. After all the tests were completed, the origins of these samples were revealed: Lots 2 through 8 represent 4 samples obtained directly from producers, 2 samples from terminals that suffered SCC in the past, and one from Brazil. The last lot (lot 9) was found to be contaminated with gasoline and cannot be considered to be representative of FGE.

## **4.0 TASK1: DETAILED CHEMICAL AND ELECTROCHEMICAL ANALYSES OF ETHANOL FROM CORN, GRAPE, SUGAR-CANE, AND CELLULOSIC SOURCES**

### **4.1 Electrochemical Finger Printing Approach**

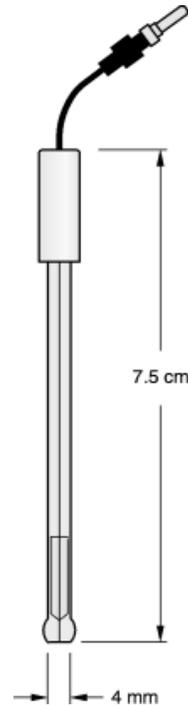
Samples of fuel grade ethanol were collected from different producers through an independent laboratory. Prior to receiving these ethanol samples, some efforts were taken to investigate whether it is feasible to use electrochemical techniques to characterize the ethanol.

Figure 3 shows the change of open circuit potential of a carbon steel sample in two different lots of fuel grade ethanol with and without the addition of Lithium Chloride (LiCl) as supporting electrolyte. The potential monitoring began at quiescent condition (i.e. no gas sparging) then was sparged with argon and zero compressed air (no CO<sub>2</sub>). The objective of the gas purging was to investigate if the Open Circuit Potential (OCP) or corrosion potential changes when the oxygen content changes in the solution. As can be seen in Figure 3, the OCP of the carbon steel sample did not show any changes when the purging gas was switched between argon and compressed air in both lots of ethanol in the absence of supporting electrolyte. The OCP, however, decreased appreciably when purging with argon and increased when purging with compressed air in the presence of LiCl as supporting electrolyte. Although the latter results were expected due to the change in the dominant cathodic reactions in the cases with and without oxygen, it was not clear why the OCP did not show any changes when dissolved oxygen concentration was changed in the absence of supporting electrolyte.



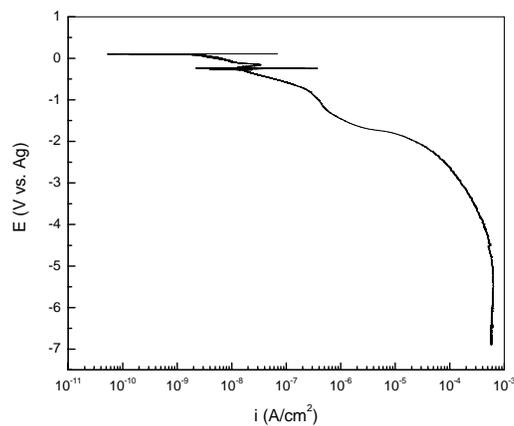
**Figure 3: The open circuit potential of carbon steel in two different lots of FGE with and without the addition of supporting electrolyte (LiCl).**

The results in Figure 3 suggest that the addition of supporting electrolyte may bias the results. Therefore, efforts were taken to perform electrochemical experiments in ethanol solutions without the addition of any supporting electrolyte. In this regard, ultramicroelectrodes (UME) have been widely used in electrochemical studies with high resistance electrolytes. A schematic of the Pt UME used in the current work is shown in Figure 4. The diameter of the UME was 10 $\mu$ m. The small surface area of the electrode resulted in smaller measured current compared to regular electrodes and thus the magnitude of the IR drop is reduced. It should be noted that IR compensation is still needed in cases where accurate potential and current are both necessary (e.g., for corrosion rate calculation). However, in a case where only limiting current is of interest, IR compensation is not necessary. By using the UME, electrochemical tests (e.g. cathodic reduction of ethanol on Pt electrode) can be performed without overloading the compliance of the potentiostat that is normally the difficulty when using regular size of electrodes.

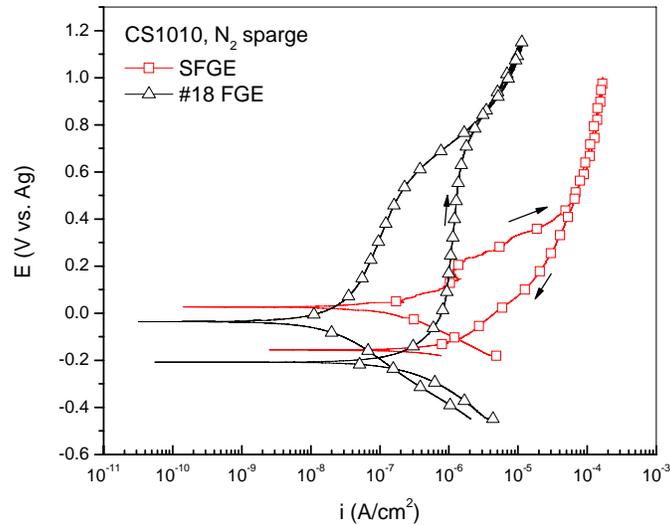


**Figure 4: The schematic of a UME.**

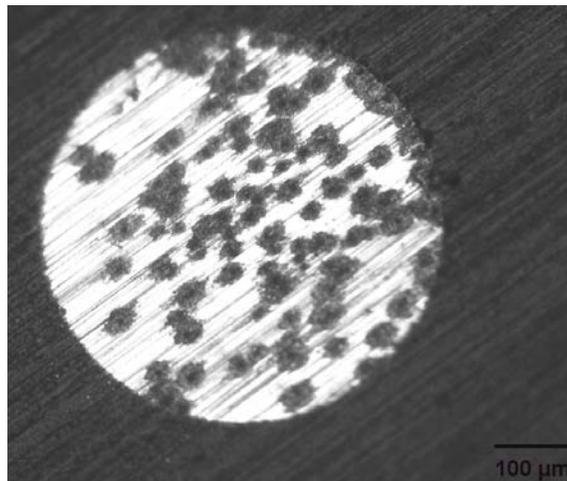
Electrochemical tests were successfully performed with the Pt UME (10 $\mu$ m diameter) and carbon steel 1010 microelectrode (0.5 mm diameter) in simulated fuel grade ethanol (SFGE) and in FGE (Figure 5 and Figure 6).



**Figure 5: A cathodic polarization curve of Pt in deaerated simulated fuel grade ethanol (scanning rate 10 mV/s)**



**Figure 6: A comparison of CPP curves of CS1010 in deaerated SFGE and FGE (scanning rate 0.2 mV/s). No supporting electrolyte was used.**

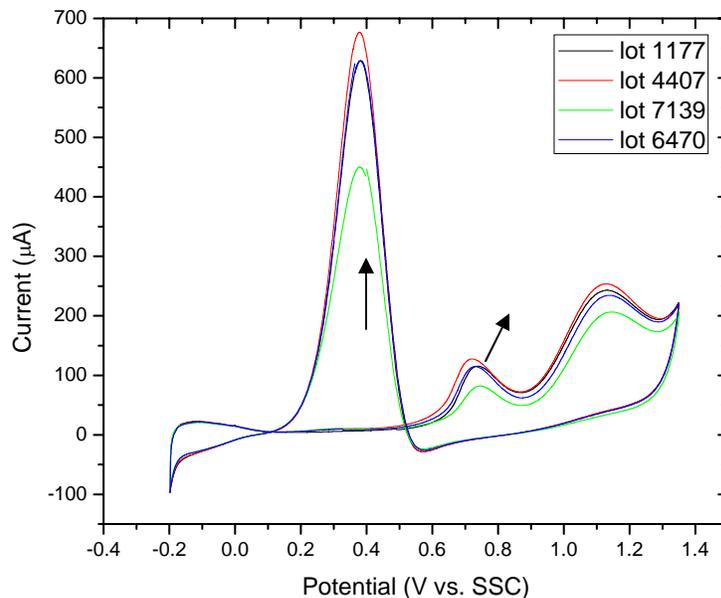


**Figure 7: Pitting corrosion after CPP testing in SFGE**

It can be seen that the SFGE exhibited much higher anodic dissolution rate than the FGE, possibly related to the higher chloride concentration of the former. Greater level of pitting was seen in SFGE (Figure 7).

Several test methods to rapidly finger print ethanol samples with respect to their SCC tendency were evaluated. The received FGE sample lots were also characterized electrochemically to investigate whether the FGE lots from different source show any difference. The first electrochemical characterization was done by oxidizing the FGE in 0.5 M H<sub>2</sub>SO<sub>4</sub> solution using

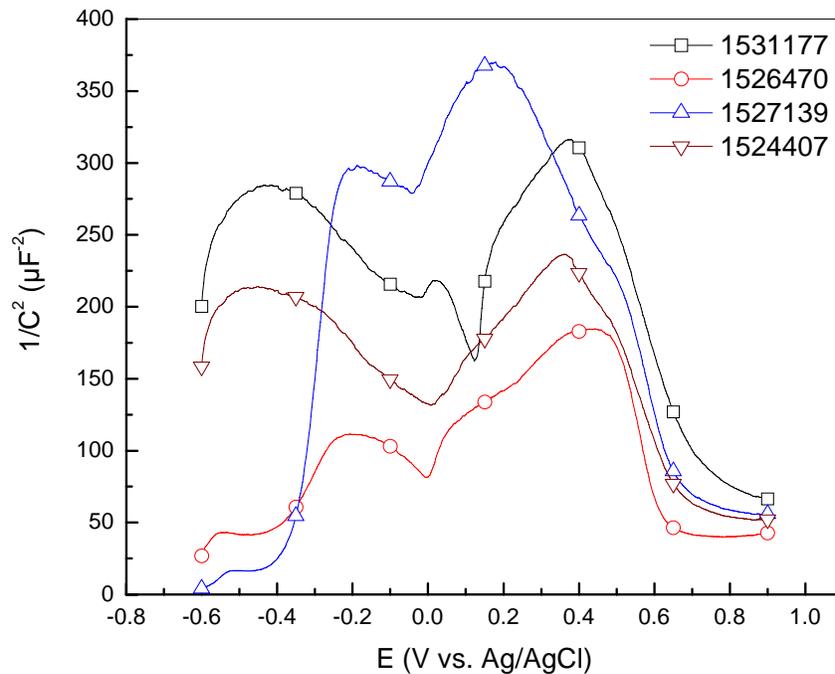
cyclic voltammetry (CV) technique. The selection of this method was based on the fact that much work on the oxidation of ethanol in  $\text{H}_2\text{SO}_4$  has been performed in the fuel cell field and the peaks associated the oxidation of ethanol were well understood. The CV curves obtained in the mixture of the FGE and  $\text{H}_2\text{SO}_4$  are compared in Figure 8. All tests were conducted in the 0.5 M  $\text{H}_2\text{SO}_4$  solution containing 1% (wt.) FGE. The scanning rate was 100 mV/s. Clearly, the CV curves showed some difference in the peak height. This suggests that there is a difference in the FGE samples.



**Figure 8. A comparison of the CV curves in the mixtures of different FGE samples with 0.5 M  $\text{H}_2\text{SO}_4$ .**

Another technique used to characterize the FGE samples was Mott-Schottky (M-S) measurement in the mixture of FGE samples in 0.5 M  $\text{H}_2\text{SO}_4$  (1% wt FGE.). This technique involves measuring the capacitance of the working electrode (Pt in this case) at different potentials. The magnitude of the capacitance reflects the extent of adsorption taking place on the Pt working electrode. The adsorption may be indicative of specific organic molecules present in different samples of FGE. The adsorption of these specific organic molecules may further indicate why certain FGE samples did not cause SCC of carbon steel.

The M-S results of the four FGE sample in 0.5 M H<sub>2</sub>SO<sub>4</sub> are compared in Figure 9. The capacitance measured in different FGE and H<sub>2</sub>SO<sub>4</sub> mixtures was different, indicating difference in the adsorption occurred on the Pt electrode. This also suggests the FGE lots did have some difference likely resulting from the biomass source or the production process by which the FGE lots were produced.



**Figure 9. The Mott-Schottky plots at 1 KHz in different FGE and 0.5 M H<sub>2</sub>SO<sub>4</sub> mixtures.**

However, the SCC susceptibility of steel does not appear to be very different amongst these lots of FGE. Thus, no correlation between the M-S peaks and SCC susceptibility could be established.

#### 4.2 Chemical Analyses of the FGE Samples

Initial water analyses for tested FGE samples are given in Table 4. Latest FGE sample (Lot-9) had less than 500 ppm of water in it. This sample also had a different smell and consistency, perhaps suggesting that this may have been an ethanol-gasoline blend. The pH<sub>e</sub> of this sample was 8.0, which was highest among all tested FGE samples. Complete chemical analysis of all FGE lots is given in Appendix A, Table 6 through Table 9. GCMS data in Table 6 shows that there were minor differences in the chemical composition but there was no significant that may

result into corrosivity differences among different FGE samples tested. GT-Lot 8 contained 2-Butenal and Terpenes, which were not seen in other FGE samples received earlier. Whereas GT-lots 5, 6, and 7 had small amounts of paraffin wax in them, this was not found in other FGE lots. At this stage, we do not have any information on the origin of these FGE lots, so the amount of constituents detected in fuel grade ethanol cannot be correlated to the process history or origin of these fuels. This information was intentionally not given to us to avoid any biases in the SCC test program. Table 7 shows the GC-MS analysis of Lot 9, confirming the initial impression that this lot was not E-95, but an ethanol-gasoline blend of unknown ratio. Therefore, the SCC results from this Lot, while they are presented, cannot be compared to the other lots.

**Table 3. Initial water content measured by Karl-Fisher method and initial pHe measured by ASTM method for different FGE lots tested in this study**

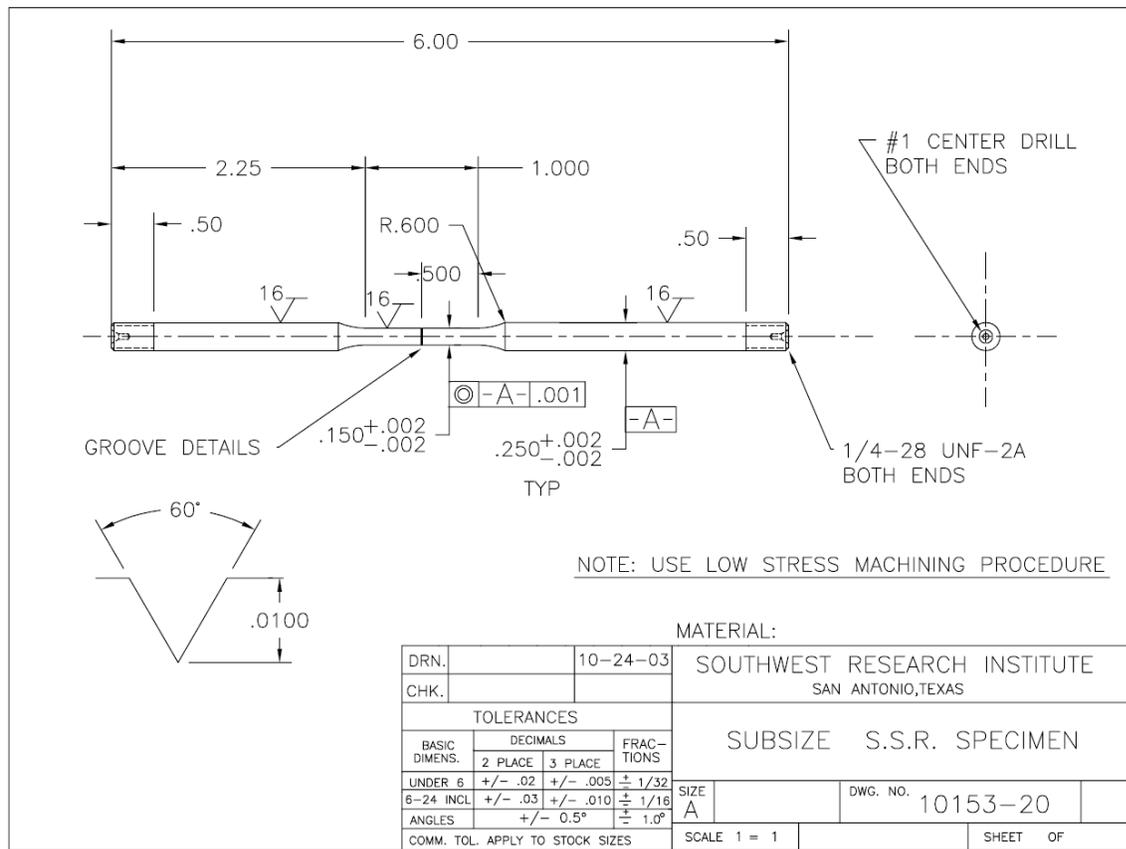
GT NAME	ROUND ROBIN DESIGNATION	INITIAL WATER CONTENT (PPM $\pm$ 125)	INITIAL PHe
200 proof Ethanol	N/A	<b>510</b>	<b>7.9</b>
Lot 1	Not in round robin study	<b>11223</b>	<b>7.7</b>
Lot 2	1524407	<b>9156</b>	<b>7.8</b>
Lot 3	1526470	<b>5529</b>	<b>7.7</b>
Lot 4	1527139	<b>6389</b>	<b>7.9</b>
Lot 5	1531177	<b>8753</b>	<b>7.1</b>
Lot 6	1602554	<b>10050</b>	<b>7.6</b>
Lot 7	1602553	<b>6471</b>	<b>7.8</b>
Lot 8	1641499	<b>6682</b>	<b>7.9</b>
Lot 9	10-1723397	<b>477</b>	<b>8.0</b>

The results of analyses of other organic and inorganic constituents are shown in Appendix A.

## 5.0 TASK 2 – SCREENING SCC TESTS IN PURE ETHANOL TO STUDY EFFECT OF MINOR CONSTITUENTS AND AGING

### 5.1 Experimental Approach

SSR tests conducted were conducted at all three labs using notched samples (Figure 10) with some notable differences in procedures.



**Figure 10. Drawing of the notched SSR test specimen**

All SSR tests were performed under aerated conditions. But there were significant differences in the test procedures between these labs:

- Georgia Tech used an elongation rate of  $5 \times 10^{-7}$  in/s, whereas DNV and SwRI used  $4 \times 10^{-7}$  in/s.
- The open circuit potential (OCP) of the SSR samples were monitored by DNV and SwRI against an Ag/AgCl/1M LiCl in EtOH reference electrode during all tests. Georgia Tech did not monitor the OCP.
- SwRI used a glass test cell vs. stainless steel test cell used by DNV Columbus.
- Both DNV and SwRI used samples machined by Metal Samples, whereas Georgia Tech used samples machined locally. For the DNV and SwRI tests, the notch on the SSR samples has a root radius of 50µm. For the Georgia Tech tests, the notch root radius was ~100 µm and the notch was ~210 µm deep. Notch depth and notch root radius of each sample was measured to be able to calculate stress concentration factor. Georgia Tech

polished the samples up to 6- $\mu$ m finish, whereas DNV and SwRI did not polish the samples. The notch was not polished by any of the organizations but the sample was ultrasonically cleaned in acetone and methanol for 20-25 minutes before SSRT.

- Georgia Tech stored the ethanol in a refrigerated environment whereas DNV and SwRI stored them in ambient temperature.
- Georgia Tech used building supply air, which may not be as pure as the air that other two labs used (Breathing Air and Zero air from cylinders). Concern was that the oil from building air supply might be act as a SCC inhibitor.

The effect of these differences were tested in various separate studies that will be discussed later.

## 5.2 Results and discussion

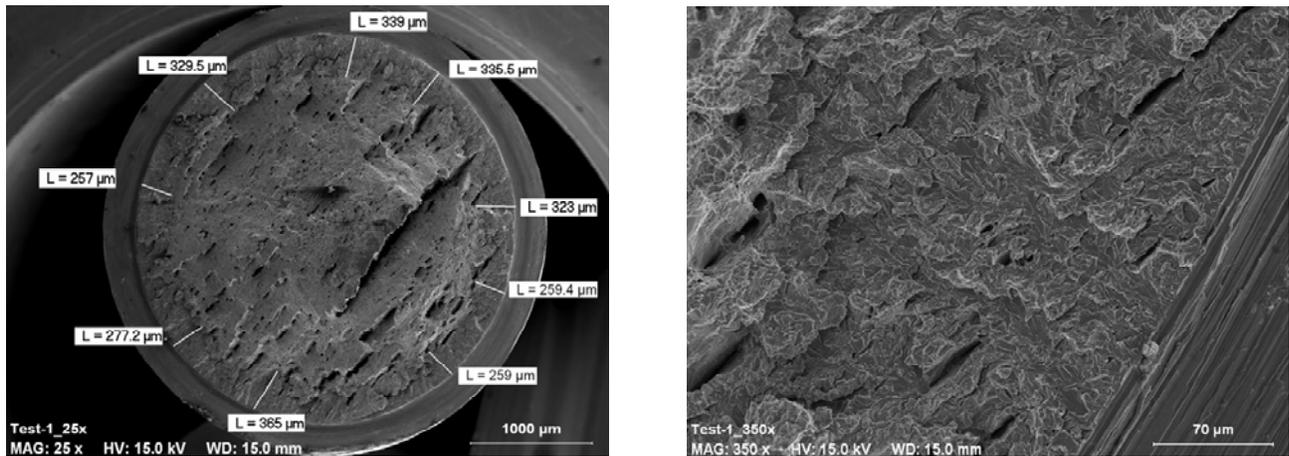
The results of the SCC testing from the three labs are summarized in Table 4.

**Table 4. summary of SSR test results from the 3 labs. The specific studies on the effect of chloride are not included in this table.**

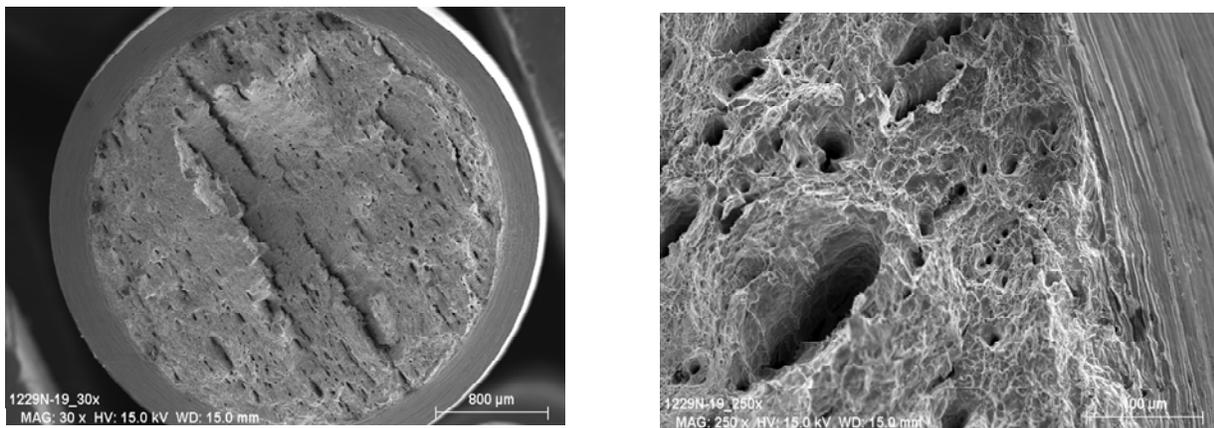
Ethanol Sample	Test Lab	Max Load, lb	Time to failure, h	Average Crack length, microns	Comment
1527139	Ga Tech	984	54	N/R	No SCC
	SwRI	1329		283	SCC
	SwRI – no OCP	1345		263	SCC
	DNV	1315	36	305	SCC
1531177	Ga Tech.	1000	61	N/R	No SCC
	SwRI	1335		148	SCC
	DNV	1330	39	314	SCC
1524407	Ga Tech	1000	59	N/R	No SCC
	SwRI	1333		246	SCC
	DNV	1370	42	299	SCC
1526470	Ga Tech	950	64	N/R	No SCC
	SwRI	1369		188	SCC
	DNV	1485	56	203	SCC

Ethanol Sample	Test Lab	Max Load, lb	Time to failure, h	Average Crack length, microns	Comment
1602553	Ga Tech	962, 972	30.4, 38	N/R	No SCC, SCC
	SwRI			215	
	DNV	1350	35	292	SCC
	DNV Glass cell	1415	49	222	SCC
1602554	Ga Tech	1000	39	0	No SCC
	SwRI	1294		251	SCC
	DNV	1340	36	345	SCC
1723398 (gasoline blend of unknown composition)	Ga Tech	1039	74.3		No SCC
	SwRI	1363		174	SCC
	DNV	1460	61	0	No SCC
	DNV Glass cell	1480		0	No SCC
Air	Ga Tech	966		N/R	No SCC
	SwRI	1414		0	No SCC
	DNV	1460	62	N/R	N/R

Examples of the fracture surface are shown in



**Figure 11. Fracture surface of steel in ethanol sample# 1527139 tested at DNV indicating the crack growth at different points of the fracture surface (average = 305 μm). High magnification view shows a mainly transgranular failure mode.**



**Figure 12. Fracture surface of steel in ethanol sample# 1723398 tested at DNV using a glass cell indicating the no SCC features. High magnification view shows a mainly ductile, microvoid coalescence failure mode.**

It can be seen from Table 2 and Table 5 that the Georgia Tech test results are at considerable variance from those of DNV and SwRI, not only in terms of the observations of SCC but also in terms of maximum loads and time to failure. There are some differences between the test results of SwRI and DNV, notably for the ethanol sample 1723398, where DNV did not observe any SCC and SwRI observed minor SCC. However, this ethanol sample had unknown amounts of gasoline and should be ignored from further consideration.

An interesting observation is that the maximum loads in Georgia Tech tests are significantly lower than those for SwRI and DNV tests, even for air test where there is no SCC. This suggests

that mechanical factors, such as specimen dimension, notch depth, etc. play a critical role in test results, although they need further evaluation.

## **6.0 TASK 3 – STATISTICALLY DESIGNED TEST MATRIX TO DETERMINE EFFECT OF CHLORIDE AND NOTCH ROOT RADIUS**

### **6.1 Experimental Approach**

As mentioned previously, a number of procedural differences were noted between the three laboratories. Georgia Tech conducted tests using purer air, ethanol stored at room temperature, and an extension rate matching that of DNV and SwRI, but did not find any SCC in these test procedures. The only condition under which they find SCC is when they intentionally added chloride to their ethanol sample or placed a reference electrode in the test cell close to the test specimen which leaked chloride. In contrast, SwRI conducted SSR tests in which they removed the reference electrode (and therefore the source of chloride contamination), but did not find that it altered SCC occurrence (see Table 4, ethanol 1527139). Similarly, DNV conducted tests without introducing reference electrode and did not find any change in SCC occurrence. Furthermore, DNV acquired an ethanol sample used by Georgia Tech and stored it at the same temperature as Georgia Tech, but did not find this to alter the consequent occurrence of SCC. However, DNV found that chloride concentration can affect the occurrence of SCC in un-notched SSR tests. Therefore, it was suspected that notch acuity may have an important conjoint action with chloride. Furthermore, methanol is added to ethanol to simulate FGE.

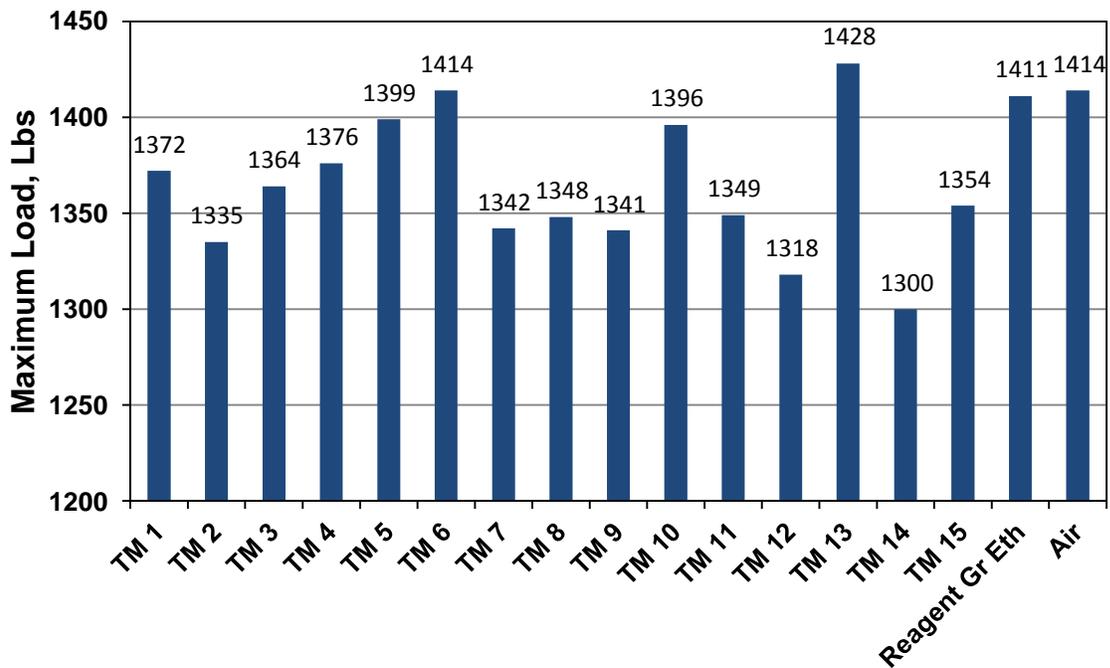
SSR testing was performed by SwRI based on a statistical matrix to determine effects three factors at three levels. The chloride content levels were 0 ppm, 5 ppm and 10 ppm. The notch radius levels were 75  $\mu\text{m}$ , 90  $\mu\text{m}$  and 100  $\mu\text{m}$  with a  $\pm 4\mu\text{m}$  tolerance. The methanol levels were 0, 0.25% and 0.5vol%. All tests have been completed and the maximum load responses from the statistical design matrix are presented in Table 1. All SSR tests were held at room temperature while under continuous air purge (Zero air – 21%O<sub>2</sub>/N<sub>2</sub>). SEM analysis was performed on the fracture surfaces after the exposure to determine the presence and extent of cracking. The test matrix is shown in Table 5 (along with the salient results).

### **6.2 Results and discussion**

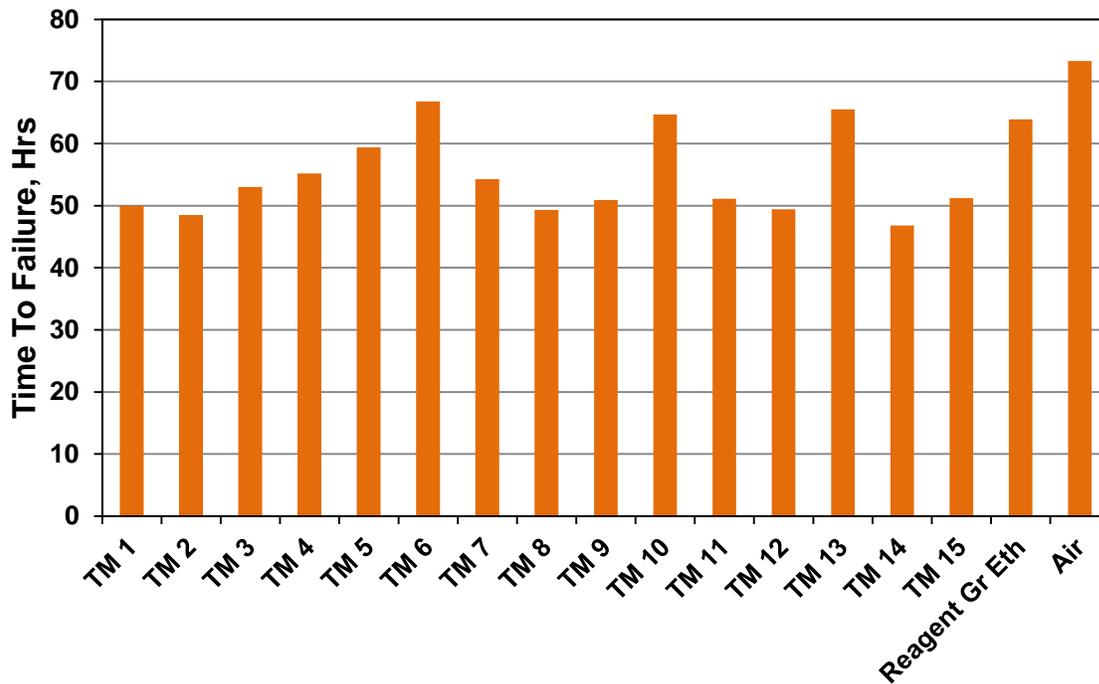
The maximum load values from the SSR testing is presented in Figure 13. A baseline air test and a pure 100% reagent grade ethanol tests were also performed for comparison. The air and reagent grade ethanol tests had 1414 and 1411 lbs, respectively. It was confirmed that no cracking occurred on the air sample after testing by SEM. Several other samples also displayed high max loads; most notably Test 5, Test 6, Test 10 and Test 13. All of these tests had maximum loads that were close to or above 1400 lbs. These same samples also had the highest time to failure as seen in Figure 14.

**Table 5. Box-Behnken Statistical Design SSR Test Matrix**

Test Run	Factor 1	Factor 2	Factor 3	Response
Order	Chloride, ppm	NotchRadius	Methanol, vol%	MaxLoad
1	5	90	0.25	1372
2	5	75	0	1335
3	5	75	0.5	1364
4	10	100	0.25	1376
5	0	75	0.25	1399
6	0	90	0	1414
7	5	100	0.5	1342
8	5	90	0.25	1348
9	10	90	0	1341
10	0	100	0.25	1396
11	5	100	0	1349
12	10	90	0.5	1318
13	0	90	0.5	1428
14	10	75	0.25	1300
15	5	90	0.25	1354
Rea Gr Eth	0	86	0	1411



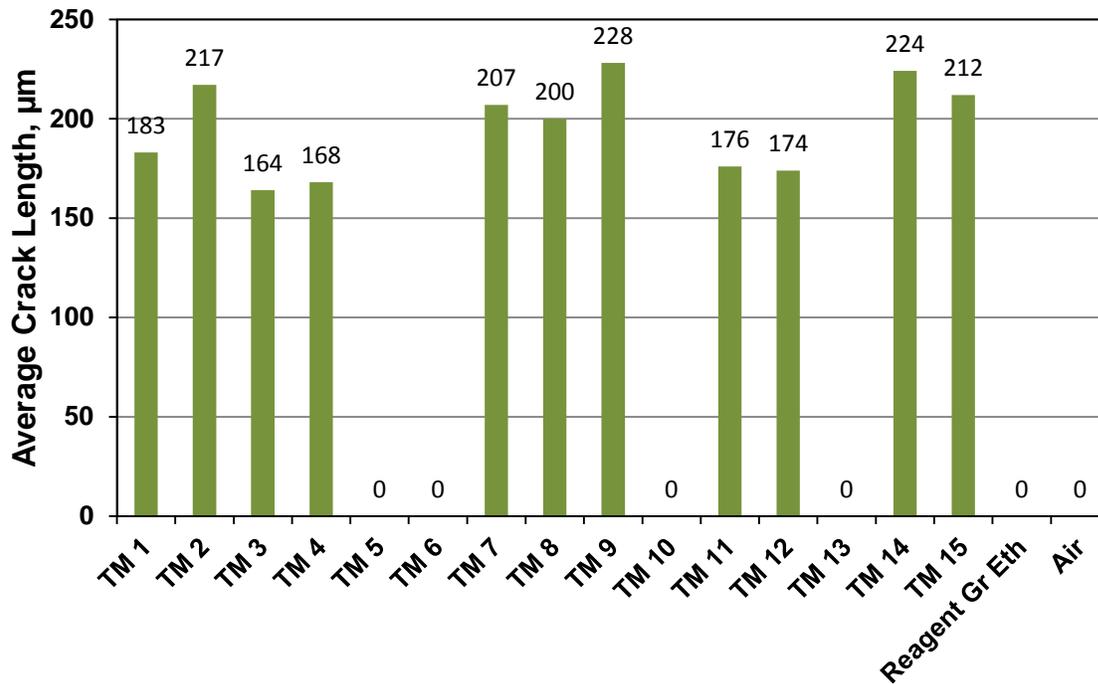
**Figure 13. Results of partial factorial matrix of tests at SwRI**



**Figure 14. Time to Failure during SSR testing. A baseline comparison is made with air and reagent grade ethanol.**

The SEM analysis revealed that no SCC occurred for Tests 5, 6, 10 and 13 and samples from these runs only showed ductile behavior. The remaining tests showed some level of transgranular cracking with average crack lengths between 164  $\mu\text{m}$  to 228  $\mu\text{m}$  (see Figure 3). All tests without chloride had the highest maximum loads. They corresponded to Tests 5 and 6, 10 and 13. The chloride contents of 5 ppm and 10 ppm all showed cracking behavior, even when the notch radius was high (100  $\mu\text{m}$ ). Thus, the primary factor in this matrix of testing was chloride level.

The notch radius and methanol concentration have a secondary effect. However, it was noted that at chloride levels of 5 ppm there was no real discernable difference between the 75 and 100  $\mu\text{m}$  notch root radius samples. Even when there was no chloride (0 ppm) the 100  $\mu\text{m}$  radius max load was not appreciably different than the 75  $\mu\text{m}$  radius sample (effect between Test 5 and 10).



**Figure 15. Average Crack Length for Tests 1 through 15. Tests 5, 6, 10 and 13 did not show cracking behavior.**

## 7.0 SUMMARY AND RECOMMENDATIONS

- The chemical analyses of the ethanol samples received to date do not reveal any startling differences. There are small differences in certain organic species, but these do not appear to be of significant consequence.
- The electrochemical fingerprinting method is capable of indicating differences between ethanol batches, but insufficient information exists to correlate these to chemical or SCC differences.
- The round robin tests between 3 labs showed considerable differences in the results. One of the labs did not observe any SCC on steel tested in different ethanol samples, whereas the results of SwRI and DNV were consistent with each other.
- The results from SwRI are consistent with the Georgia Tech results in that they show that chloride is an important factor in the SCC in ethanol (assuming that sufficient aeration is present). The notch root radius and methanol had secondary effects.

- However, mechanical factors, such as specimen size, notch depth, and extension rate, in addition to chloride is likely to play a critical role because Georgia Tech observed much lower maximum load to failure compared to the other two labs, even in air.
- Because the FGE samples represented 4 samples from producers, 2 samples from terminals that had exhibited SCC in the past, and one sample from Brazil and no significant differences in SCC tendency was observed within any of the laboratories, a conclusion can be drawn that SCC of steel in FGE is not sensitive to feedstock differences or point of origin. Thus, the observations of SCC mainly in North American terminals (and not in Brazil or by ethanol producers) should be attributed to other factors not inherent in FGE.

## 8.0 REFERENCES

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## APPENDIX A: CHEMICAL ANALYSES OF FGE

Table 6. GC-MS Results showing organic constituents in FGE GT Lots 1- 8

	retention time (min)	control (200 proof EtOH) µg/ml	Lot1  No number µg/ml	Lot 2  1524407 µg/ml	Lot 3  1526470 µg/ml	Lot 4  1527139 µg/ml	Lot 5 (added 08/10/09) 1531177 µg/ml	Lot 6 (added 9/3/09) 1602554 µg/ml	Lot 7 (added 9/3/09) 1602533 µg/ml	Lot 8  1641499 µg/ml
<b>Alcohol</b>										
2-butenal	7.15	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	389.355
3-methyl butanol	7.57	0.00	1296.01	433.66	372.73	617.55	0.00	0.00	0.00	0.00
<b>Subtotal, µg</b>		<b>0.00</b>	<b>1296.01</b>	<b>433.66</b>	<b>372.73</b>	<b>617.55</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>389.355</b>
<b>Fatty acid esters</b>										
C10:COOH, ethyl ester	32.57	0.00	0.00	1.52	0.00	0.00	0.00	0.00	0.00	0.00
C12:COOH, ethyl ester	44.307	0.00	0.31	1.16	0.00	0.00	0.00	0.00	0.00	0.00
C14:COOH, ethyl ester	48.46	0.00	0.66	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C16:COOH		0.00	0.00	0.00	0.00	0.00	0.06	0.00	0.67	0.00
C16:COOH, ethyl ester	51.152	0.00	16.65	2.49	0.00	0.13	1.57	0.00	1.78	0.00
C18:COOH, methyl ester	52.353	0.00	0.00	0.00	0.00	1.03	0.00	0.00	0.00	0.00
9,12-octadecadienoic acid, ethyl ester	53.046	0.00	7.94	0.86	0.00	0.00	0.18	0.00	0.00	0.00
9-octadecenoic acid, ethyl ester	53.127	0.00	1.04	0.16	0.00	0.00	0.13	2.29	2.95	0.00
<b>Subtotal, µg</b>		<b>0.00</b>	<b>26.61</b>	<b>6.18</b>	<b>0.00</b>	<b>1.16</b>	<b>1.94</b>	<b>2.29</b>	<b>5.39</b>	<b>0.00</b>
C18:COOH	53.556	0.00	2.17	1.45	7.45	0.17	0.00	0.00	0.00	1.21
<b>Subtotal, µg</b>		<b>0.00</b>	<b>2.17</b>	<b>1.45</b>	<b>7.45</b>	<b>0.17</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>1.21</b>
2,2-bis[(4-hydroxy)phenyl]propane	53.729	0.00	0.00	3.16	7.73	0.00	0.00	0.00	0.00	0.00



	retention time (min)	control (200 proof EtOH) µg/ml	Lot1  No number µg/ml	Lot 2  1524407 µg/ml	Lot 3  1526470 µg/ml	Lot 4  1527139 µg/ml	Lot 5 (added 08/10/09) 1531177 µg/ml	Lot 6 (added 9/3/09) 1602554 µg/ml	Lot 7 (added 9/3/09) 1602533 µg/ml	Lot 8  1641499 µg/ml
<b>Subtotal, µg</b>		0.00	0.00	3.16	7.73	0.00	0.00	0.00	0.00	0.00
<b>Hydrocarbon</b>										
C22H46	53.36	0.48	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C23H48	54.301	0.79	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C24H50	55.256	1.29	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C25H52	56.237	1.48	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C26H54	57.244	1.61	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C27H56	58.336	1.38	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C28H58	59.58	1.08	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C29H60	61	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
<b>Subtotal, µg</b>		8.11	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
<b>Rosin Acids</b>										
Pimaric acid	54.385	0.00	3.07	0.86	5.30	0.07	0.06	0.69	1.05	0.53
Sandaracopimaric acid	54.617	0.00	1.83	0.61	2.99	0.10	0.04	0.56	0.98	0.39
Isopimaric acid	54.937	0.00	2.63	0.75	3.40	0.07	0.07	0.64	1.07	0.42
Palustric acid	55.206	0.00	3.96	1.15	5.92	0.08	0.07	0.29	0.34	0.27
DHA	55.617	0.65	2.27	0.99	4.79	0.08	0.07	0.53	1.22	0.62
Abietic acid	55.966	0.00	3.29	1.31	6.92	0.14	0.08	0.67	1.41	0.68
<b>Subtotal, µg</b>		0.65	17.05	5.66	29.31	0.54	0.39	3.38	6.07	2.91
<b>Terpenes?</b>										
2,2-bis[(4-hydroxy)phenyl]propane										3.68
unidentified terpene										0.46



	retention time (min)	control (200 proof EtOH) µg/ml	Lot1 No number µg/ml	Lot 2 1524407 µg/ml	Lot 3 1526470 µg/ml	Lot 4 1527139 µg/ml	Lot 5 (added 08/10/09) 1531177 µg/ml	Lot 6 (added 9/3/09) 1602554 µg/ml	Lot 7 (added 9/3/09) 1602533 µg/ml	Lot 8 1641499 µg/ml
unidentified terpene										0.89
unidentified terpene										0.19
<b>Subtotal, µg</b>										<b>3.68</b>
<b>Paraffin wax components</b>										
2,2-bis[4'-hydroxyphenyl]propane		0.00	0.00	0.00	0.00	0.00	0.25	0.49	2.92	0.00
Plasticizer?		0.00	0.00	0.00	0.00	0.00	0.04	10.30	0.97	0.00
Plasticizer?		0.00	0.00	0.00	0.00	0.00	0.04	0.00	0.47	0.00
<b>Subtotal, µg/ml</b>		<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.33</b>	<b>10.79</b>	<b>4.36</b>	<b>0.00</b>
<b>Total ° µg</b>		<b>8.76</b>	<b>1341.85</b>	<b>450.12</b>	<b>417.22</b>	<b>619.43</b>	<b>2.66</b>	<b>16.46</b>	<b>15.82</b>	<b>397.17</b>

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**Table 7. GC-MS Results showing organic constituents in FGE GT Lots 1- 9**

	<b>FGE Lot 9 10-1723397</b>
Identified Constituents	Content µg/ml
<b>Gasoline components</b>	
1-methylethyl benzene	1433.89
Propyl benzene	4110.60
1-ethyl-3-methyl benzene	10456.80
1,2,3 trimethyl benzene	2261.87
1-ethyl-2-methyl benzene	2078.62
1,2,4-trimethyl benzene	12759.78
1,3,5-trimethyl benzene	3347.41
Indane	1256.11
1-methyl-3-propyl benzene	2689.76
1-methyl-2-propyl benzene	1468.16
4-ethyl-1,2-dimethyl benzene	4335.20
1-methyl-4-propyl benzene	654.42
1-methyl-3(1-methylethyl) benzene	3029.22
1-methyl-2(1-methylethyl) benzene	825.90
1,2,3,4-tetramethyl benzene	3340.11
5-methyl indane	823.78
1-methyl-4-(1-methylpropyl) benzene	457.30
2,3-dihydro-5-methyl indene	1716.69
4-methylphenyl acetone	274.74
Phthalene	290.87
2(4'-methylphenyl) propanal	275.30
1,4-diethyl-2-methyl benzene	189.92
Naphthalene	2096.89
1-methyl-1-butenyl benzene	109.06

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Indene	517.28
Ethyl-1,2,4-trimethyl benzene	504.84
Dodecane	188.59
2,3-dihydro-4,7-dimethyl indene	528.11
2-methyl naphthalene	1268.12
Tridecane	171.44
1-methyl naphthalene	652.84
2-ethyl naphthalene	287.70
2,7-dimethyl naphthalene	1007.42
Pentadecane	130.56
2,5-dimethyl-1,6-methanol annulene	38.09
Trimethyl naphthalene	266.97
Hexadecane	49.74
Octadecane	22.92
<b>Subtotal, µg/ml</b>	<b>65917.02</b>
<b>Fatty Acids</b>	
Palmitic acid	42.80
Oleic acid	80.98
Stearic acid	53.73
<b>Subtotal, µg/ml</b>	<b>177.52</b>
<b>Rosin acid</b>	
Pimaric acid	76.54
Isopimaric acid	62.13
Sandanacopimaric acid	63.69
Palustric acid	24.14
DHA	69.13
abietic acid	40.63
<b>Subtotal, µg/ml</b>	<b>336.26</b>
<b>Total, µg/ml</b>	<b>66430.79</b>

Table 8. ICP Data showing metal concentration in GT Lots 1-5.

Element	Density (g/ml)	Lot 1	Lot 2	Lot 3	Lot 4	Control	Lot 5 (added 8/26/09)	Lot 6 (added 8/26/09) no number	Lot 7 (added 8/26/09)	Lot 8 (added 12/15/09)	FGE LOT 9
		ADM ug/L (ppb)	1524407 ug/L (ppb)	1526470 ug/L (ppb)	1527139 ug/L (ppb)	200 proof ug/L (ppb)	1531177 ug/L (ppb)	1641499	1641499	10-1723397	
S	2.07	394.4	301.6	171.8	177.2	77.1	509.0	587.0	643.3	185	953
P	1.82	125.9	296.8	300.2	235.3	150.9	121.2	162.0	161.3	133	194
Na	0.97	104.0	109.8	124.3	117.5	96.4	415.4	394.5	415.5	139	1811
Si	2.33	65.6	44.2	83.5	51.4	27.8	76.5	66.0	50.3	386	103
Se	4.79	38.2	53.5	63.9	34.8	34.4	22.3	29.8	29.7	24.0	35.7
Ca	1.55	31.4	41.8	49.1	78.0	36.9	68.4	56.1	66.7	27.0	161
Sn	7.31	25.7	12.4	20.6	24.6	14.0	15.9	20.6	14.4	9.00	25.4
Al	2.70	18.2	5.3	4.5	15.2	3.6	5.8	23.5	8.2	4.00	17.0
K	0.86	15.5	21.1	14.5	23.8	5.6	30.4	62.1	32.5	33.0	321
Mg	1.74	10.3	9.7	7.3	10.6	7.3	15.0	14.0	6.1	5.00	13.9
Fe	7.87	7.1	17.2	17.9	27.8	5.5	21.3	41.3	41.9	8.00	61.4
Zn	7.13	7.0	10.2	7.0	12.4	3.6	10.2	22.3	10.8	6.00	69.3
Cu	8.96	4.2	8.0	5.1	6.9	6.3	9.7	5.2	70.5	4.00	18.5
Cd	8.65	3.0	1.0	3.0	3.0	0.9	0.9	1.2	1.2	1.00	1.92
Co	8.90	1.9	1.4	1.3	5.1	1.3	1.3	1.8	1.7	1.00	2.10
Mn	7.30	0.3	0.2	0.1	0.3	0.1	1.9	1.0	0.8	0.164	0.72
Ti	4.54	0.3	0.6	0.4	0.6	0.4	0.1	1.4	0.6	0.493	0.48
As	5.73	16.8	<18.0	16.9	16.8	16.9	17.0	22.7	22.6	19.0	27.1
B	2.34	0.7	<0.7	0.7	0.7	0.7	4.2	0.9	1.0	1.00	1.05
Ba	3.50	0.5	<0.6	0.5	3.0	0.5	0.9	1.0	1.0	1.00	1.44
Be	1.85	0.0	<0.0	0.0	0.0	0.0	0.0	0.1	0.1	0.0444	0.0648
Cr	7.19	0.8	<0.9	0.8	0.8	0.8	0.8	1.1	1.1	1.00	3.84
Mo	10.22	3.1	3.4	3.1	3.1	3.1	3.2	4.2	4.2	3.00	5.05
Ni	8.90	1.6	1.7	1.6	1.6	2.2	3.0	2.1	2.1	2.00	3.84
Pb	11.35	10.6	15.3	10.7	13.3	30.8	10.7	14.3	14.3	12.0	17.1
Sb	6.69	10.0	10.7	10.0	10.0	10.0	10.1	13.4	13.4	11.0	16.1
Sr	2.54	0.0	0.2	0.1	0.1	0.0	0.1	0.2	0.2	0.0543	0.24
Tl	11.85	22.4	24.0	22.5	22.4	22.5	22.6	30.2	30.1	25.0	36.1
V	6.11	0.3	0.4	0.3	0.3	0.3	0.4	0.8	1.0	0.385	3.60
Li	0.53						0.3	0.4	0.4		0.58
<b>Total (ug/L)</b>		<b>920.1</b>	<b>1010.6</b>	<b>941.9</b>	<b>896.6</b>	<b>560.1</b>	<b>1398.9</b>	<b>1581.3</b>	<b>1646.8</b>	<b>1041</b>	<b>3905</b>

**Table 9. Chloride, sulfate and acetate content in FGE lot-9**

Sample Name	Instrument Injection Concentration (ug/g)			Sample Concentration Factor	Sample Concentration (ug/g)		
	Chloride	Sulfate	Acetate		Chloride	Sulfate	Acetate
FGE GT Lot9	1.697	0.493	3.804	16.98	0.1	0.03	0.22
FGE GT Lot9 (Duplicate)	2.251	0.508	68.922	15.99	0.14	0.03	4.31

# DNV Energy

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