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FINAL REPORT

Determine the Requirements for Existing Pipeline, Tank, and Terminal Systems to Transport Ethanol Without Cracking

PRCI Project Phase 2 SCC-4-4 Report
Contract PR186-073503
DNV Columbus, Inc. – Project 811 7376 1

Prepared for the

Corrosion Technical Committee of

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Prepared by the following Research Agency:

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RESEARCH SUMMARY

Title:	Determine the Requirements for Existing Pipeline, Tank, and Terminal Systems to Transport Ethanol Without Cracking
Contractor:	DNV Columbus, Inc. (formerly CC Technologies, Inc.)
Principal Investigators:	John A. Beavers, FNACE, Ph.D.
Report Period:	Final Report for Phase 2
Objectives:	The objectives of SCC 4-4 were to: (1) Develop data necessary to make engineering assessments of the feasibility of transporting FGE and FGE blends in existing pipelines. The transportation may be in a dedicated pipeline or in a batching mode, (2) Identify ethanol blends that can be transported in existing pipelines without significant modification of the system and operations (Case 1), blends that require significant modifications (Case 2) and blends that cannot be transported in existing pipelines, but could be moved in specially designed systems (Case 3), and (3) Characterize the time to initiation of SCC in a range of potent ethanol environments and identify safe operating and or batching practices that prevent the initiation and growth of SCC.
Scope:	In order to achieve the project objectives, the work scope was divided in two Phases: **Phase 1**. (Screening Tests on Transportability of Blends) consists of four tasks; Task 1-1 (Screening SCC Tests to Determine FGE Blends that can be Transported in Existing Pipeline without Significant Modification), Task 1-2 (Evaluation of the Effect of Ethanol Blends on Static Elastomeric Seals), Task 1-3 (Evaluation of the Effect of Ethanol Blends on Dynamic Elastomeric Seals), and Task 1-4 (Reporting and Program Management). **Phase 2**. (Time to Failure Tests under Static and Dynamic Loads for Assessing Batching Operations) consists of three tasks: Task 2-1 (Static Load Tests), Task 2-2 (Cyclic Load Tests), and Task 2-3 (Reporting and Management). This report summarizes the results of Phase 2.

Technical Perspective:	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. Despite the accumulation of a considerable amount of laboratory data on SCC susceptibility of pipe steel in FGE, significant gaps remain in our understanding of the technical issues associated with pipeline transportability of FGE and its gasoline blends: • It is unclear whether a safe limit in terms of gasoline-FGE blending that would not cause SCC under any circumstances exists. This blending limit may depend on ethanol chemistry. • SCC initiation and growth rates in FGE and blends are not known. The tests conducted to date are essentially screening type tests focused on identifying the effects of environmental variables on a cracking/no cracking pass/fail basis. The initiation and growth rate data may permit better estimation of acceptable batching procedures or FGE residence times. • The effect of metallurgical changes due to welding or casting on SCC is not known. • The implementation of various SCC mitigation methods in the field has not been developed. For example, the effectiveness of oxygen scavengers may depend on dosage, introduction mode, gas/liquid phase ratio, flow rate, presence of solids, residence time, etc. • The interactions between FGE/blends and drag reduction agents, scale, internal coatings, or corrosion products/deposits have not been characterized. • The effect of ethanol on other non-metallic components, such as seals and gaskets, is not fully understood. • The effect of various mitigation methods on end-use components (engines and tailpipe emissions, gas tanks, etc.) is not known. • A systematic methodology incorporated in a guideline document, by which risks of accepting a particular ethanol chemistry and blend can be assessed, is needed. Such a guidelines document is one of the eventual goals of the research. PRCI SCC 4-4 addressed some of these gaps as described in the Objectives and Scope. Other programs being planned by PRCI and others will address the remaining gaps with the ultimate goal of collating the results of all planned efforts to develop a coherent methodology for sound decision-making.

Technical Approach:	All testing in Phase 2 of the PRCI program was performed with pre-cracked compact tension specimens machined from an X46 line pipe steel having a DSAW long seam weld. Variables in the testing included location of the pre-crack with respect to the seam weld (in the weld metal, in the heat-affected zone (HAZ), or in the base metal), ethanol-gasoline blend ratio, type of loading (cyclic loading and constant displacement), and batching. The pre-cracked specimens were oriented such that the through-wall pre-crack propagated in the axial pipe direction. The crack growth in the tests was monitored continuously using the electric potential drop (EPD) technique. The Phase 2 tests were performed at room temperature in a 4 L stainless steel test cell. The test solutions (simulated fuel grade ethanol (SFGE) and SFGE gasoline blends) were actively sparged with breathing grade air at a flow rate of approximately 4 ml/minute. An ethanol bubbler trap was used on the outlet of the test cell to exclude moisture. Specimens were tested in SFGE containing 5 ppm Cl and in SFGE-gasoline blends. The SCC tests were performed under freely corroding conditions and the corrosion potential was periodically monitored in each test using a Ag/AgCl EtOH reference electrode. A piece of rusted pipe steel was placed in the test cell and galvanically connected to the test specimen to more closely simulate the native corrosion potential of a mill scaled/rusted pipe wall. The rusted steel to specimen area ratio was approximately 5 to 1. The specimen and rusted steel piece were electrically isolated from the specimen grips and test cell in the test machine. For the constant displacement tests, the specimen was strained at a constant displacement rate, using a SSR test frame, and the displacement was stopped when there was evidence of crack extension from the EPD measurements. The specimen remained in the loading frame under this constant displacement (static) loading until crack growth stopped. For the cyclic load tests, the ratio of the minimum to maximum load (R ratio) ranged from 0.6 to 0.8 and the cyclic frequency was 1.2×10^{-4} Hz (one cycle every 2.3 hours). These conditions are typical of cyclic pressure fluctuations on liquid petroleum pipelines. The maximum load in the tests was selected to simulate the driving force on a crack that just survived a hydrostatic test. For each specimen, cracking was initiated in an aggressive SFGE and propagated for approximately one-month period. The test conditions were changed periodically to evaluate the effect of the variables in the test matrix on crack growth.

Results:	The results of the Phase 1 SSR tests and Phase 2 cyclic load tests were generally consistent. In the DSAW line pipe steel tested in Phase 2, the base metal, heat affected zone, and weld metal were all susceptible to SCC in SFGE and the differences are not significant from an integrity perspective. In the static load tests, the threshold stress intensity factor for SCC initiation, K_{thSCC}, in the base metal was approximately 30 ksi $in^{1/2}$ in SFGE. In the cyclic load tests with blends, no SCC was observed in gasoline and E-10 but significant SCC was observed in E-20 and higher ethanol blends. Crack growth also arrested in E-15, but a relatively long time was required for crack arrest in this blend. One somewhat surprising observation was the high crack growth rates in the E-50 blend in the cyclic load tests. This blend was a more potent SCC agent than SFGE; an effect that was not apparent in the Phase 1 SSR tests. The results of the batching tests were not promising. For the long (twelve-day) batch cycle, the average crack growth rate could be reasonable estimated based on the exposure time to the SFGE. This rate is too high to be considered a reasonable mitigation method. For the short (twenty-four hour) batch cycle, even short times of exposure to SFGE resulted in measurable SCC crack growth. This behavior indicates that SCC initiation times are short for sharp cracks.
Project Implications:	• Pipelines made of common line pipe steels (e.g., Grade B and X-42 to X-60) are likely to be susceptible to ethanol SCC and any differences in susceptibility probably are not significant from an integrity perspective (Phase 1 Results). • While differences in susceptibility were noted for some weld types, in general, the base metal, heat affected zone, and weld metal were all susceptible to SCC in SFGE (Phase 1 Results). • For sharp cracks, SCC initiation times are short once the line pipe steel is exposed to FGE or FGE blends capable of promoting SCC. • Once cracks initiate, crack growth rates are high in comparison with other forms of pipeline SCC. • Batching does not appear to be a viable method for SCC mitigation. • The only blends that can be safely transported in existing pipelines without significant modification of the system or operations (Case 1) are those containing less than 15% ethanol. • All other blends require significant modifications of the system or operations (Case 2), or specially designed systems (Case 3). • Case 2 could include deaeration of the SFGE, or the addition of
Project Mgr.:	John Beavers

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13. ABSTRACT

The potential exists for stress corrosion cracking (SCC) of carbon steel pipelines transporting fuel grade ethanol (FGE) and FGE- gasoline blends. The objectives of SCC 4-4 were to:

1. Develop data necessary to make engineering assessments of the feasibility of transporting FGE and FGE blends in existing pipelines,
2. Identify ethanol blends that can be transported in existing pipelines without significant modification of the system and operations (Case 1); blends that require significant modifications (Case 2) and blends that cannot be transported in existing pipelines, but could be moved in specially designed systems (Case 3), and
3. Characterize the time to initiation of SCC in a range of potent ethanol environments and identify safe operating and or batching practices that prevent the initiation and growth of SCC.

The results of the research (Phase 1 and Phase 2) demonstrated that:

1. Pipelines made of common line pipe steels (e.g., Grade B and X-42 to X-60) are likely to be susceptible to ethanol SCC and any differences in susceptibility are probably not relevant from an integrity perspective.
2. While differences in susceptibility were noted for some weld types, in general, the base metal, heat affected zone, and weld metal were all susceptible to SCC in SFGE.
3. For sharp cracks, SCC initiation times are short once the line pipe steel is exposed to FGE or FGE blends capable of promoting SCC.
4. Once cracks initiate, crack growth rates are high in comparison with other forms of pipeline SCC.
5. Batching does not appear to be a viable method for SCC mitigation.
6. The only blends that can be safely transported in existing pipelines without significant modification of the system or operations (Case 1) are those containing less than 15% ethanol.
7. All other blends require significant modifications of the system or operations (Case 2), or specially designed systems (Case 3).
8. Case 2 could include deaeration of the SFGE, or the addition of inhibitors.
9. Case 3 is the subject of ongoing research (SCC 4-5).

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Pipeline Research Council International, Inc.
Falls Church, Virginia

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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. Additionally, the effects of residence time of FGE or its blends on SCC (i.e. crack initiation time and growth rate) had not yet been determined. Finally, the effects of ethanol on other materials used in the pipelines, such as elastomeric seals and internal coatings, needed to be evaluated. Thus, the major objectives of the program are to:

1. Develop data necessary to make engineering assessments of the feasibility of transporting FGE and FGE blends in existing pipelines. The transportation may be in a dedicated pipeline or in a batching mode.

2. Identify ethanol blends that can be transported in existing pipelines without significant modification of the system and operations (Case 1), blends that require significant modifications (Case 2) and blends that cannot be transported in existing pipelines, but could be moved in specially designed systems (Case 3).

3. Characterize the time to initiation of SCC in a range of potent ethanol environments and identify safe operating and or batching practices that prevent the initiation and growth of SCC.

A program consisting of two phases was performed to address these objectives.

<u>Phase 1:</u> In the first phase, screening tests were conducted to identify ethanol blends that are unlikely to cause internal SCC.

Slow strain rate (SSR) tests of smooth and notched specimens were used to rapidly determine susceptibility to SCC. Variables in the testing included long seam weld type, location of the notch with respect to the seam weld (in the weld metal, in the heat-affected zone (HAZ), or in the base metal), ethanol chemistry, and the ethanol-gasoline blend ratio In the majority of tests, a simulated FGE was blended with various proportions of gasoline to determine the maximum ethanol/gasoline ratio that will not cause SCC in these tests. The simulated ethanol is a 200-proof ethanol in which water, chloride, methanol, acetic acid, and denaturant are added within the ASTM D-4806 specification. Several SSR tests also were performed to compare the potency of the simulated FGE and one lot of corn-based FGE. In Phase 1, a literature survey and laboratory testing also was performed to evaluate static and dynamic elastomeric seals.

The following were the conclusions of the Phase 1 SSR testing.

- No SCC was observed in aerated 10% ethanol (by volume) blend (E-10), prepared with SFGE.
- Significant SCC was observed with aerated ethanol-gasoline blends having ethanol concentrations of 20% (by volume) or higher.
- Significant SCC was observed with both simulated and one lot of corn-based FGE but the simulated FGE was a slightly more potent SCC agent.
- The base metal of all of the steels evaluated (X42, X46, X52, and X60) and cast steel exhibited measurable susceptibility to SCC and the differences probably are not significant from an integrity standpoint.
- Crack growth rates for the seamless, the cast steel, and the low frequency electric resistance welded (LFERW) line pipe steels were somewhat lower than for the double submerged arc welded (DSAW) and two other ERW line pipe steels but all steels exhibited relatively deep cracks.
- No major effect of weld metallurgy on SCC behavior was observed in SSR tests of base metal, weld metal, and heat affected zone specimens from girth welds and DSAW long seam welds. The crack depths in the tests were similar for the three different metallurgies, although the weld metals appeared to be somewhat more resistant to cracking.
- The absence of SCC in several tests with high frequency electric resistance welded (HFERW) and LFERW specimens, where the notch was located at or near the bond line of the long seam weld, was attributed to the poor mechanical properties of the bond line.
- In two SSR tests with one LFERW pipe steel, the bond line did appear to be more resistant to SCC in the ethanol-gasoline blends than the base metal of that steel.

Phase 2: In the second phase, crack growth tests under static and cyclic loads were conducted in ethanols/blends that caused cracking in SSR tests to identify safe operating and or batching practices that prevent the initiation and growth of SCC.

All testing in Phase 2 of the PRCI program was performed with pre-cracked compact tension specimens machined from an X46 line pipe steel having a DSAW long seam weld. Variables in the testing included location of the pre-crack with respect to the seam weld (in the weld metal, in the heat-affected zone (HAZ), or in the base metal), ethanol-gasoline blend ratio, type of loading (cyclic loading and constant loading), and batching. The pre-cracked specimens were oriented such that the through-wall pre-crack propagated in the axial pipe direction. The crack growth in the tests was monitored continuously using the electric potential drop (EPD) technique. The Phase 2 tests were performed at room temperature in a 4 L stainless steel test cell. The test solutions (SFGE and SFGE gasoline blends) were actively sparged with breathing grade air at a flow rate of approximately 4 ml/minute. An ethanol bubbler trap was used on the outlet of the test cell to exclude moisture.

Specimens were tested in SFGE containing 5 ppm Cl and in SFGE-gasoline blends. The SCC tests were performed under freely corroding conditions and the corrosion potential was periodically monitored in each test using an Ag/AgCl EtOH reference electrode. A piece of rusted pipe steel was placed in the test cell and galvanically connected to the test specimen to more closely simulate the native corrosion potential of a mill scaled/rusted pipe wall. The rusted steel to specimen area ratio was approximately 5 to 1. The specimen and rusted steel piece were electrically isolated from the specimen grips and test cell in the test machine.

For the constant displacement tests, the specimen was strained at a constant displacement rate, using a SSR test frame, and the displacement was stopped when there was evidence of crack extension from the EPD measurements. The specimen remained in the loading frame under this constant displacement (static) loading until crack growth stopped. For the cyclic load tests, the ratio of the minimum to maximum load (R ratio) ranged from 0.6 to 0.8 and the cyclic frequency was 1.2×10^{-4} Hz (one cycle every 2.3 hours). These conditions are typical of cyclic pressure fluctuations on liquid petroleum pipelines. The maximum load in the tests was selected to simulate the driving force on a crack that just survived a hydrostatic test. For each specimen, cracking was initiated in an aggressive SFGE and propagated for approximately a one-month period. The test conditions were changed periodically to evaluate the effect of the variables in the test matrix on crack growth.

The results of the Phase 1 SSR tests and Phase 2 cyclic load tests were generally consistent. In the DSAW line pipe steel tested in Phase 2, the base metal, heat affected zone, and weld metal were all susceptible to SCC in SFGE and the differences are not significant from an integrity perspective.

In the cyclic load tests with blends, no SCC was observed in gasoline and E-10 but significant SCC was observed in E-20 and higher ethanol blends. Crack growth also arrested in E-15, but a relatively long time was required for crack arrest in this blend. One somewhat surprising observation was the high crack growth rates in the E-50 blend in the cyclic load tests. This blend was a more potent SCC agent than SFGE; an effect that was not apparent in the Phase 1 SSR tests.

The results of the batching tests were not promising. For the long (twelve-day) batch cycle, the average crack-growth rate could be reasonably estimated based on the exposure time to the SFGE. This rate is too high to be considered a reasonable mitigation method. For the short (twenty-four hour) batch cycle, even short times of exposure to SFGE resulted in measurable SCC crack growth. This behavior indicates that SCC initiation times are short for sharp cracks.

Referring to the overall project objectives, the following are the conclusions of SCC 4-4:

- Pipelines made of common line pipe steels *(e.g., Grade B and X-42 to X-60)* are likely to be susceptible to ethanol SCC and any differences in susceptibility are probably not significant from an integrity perspective.
- While differences in susceptibility were noted for some weld types, in general, the base metal, heat affected zone, and weld metal were all susceptible to SCC in SFGE.
- The threshold stress intensity factor for SCC initiation, K_{thSCC}, in the base metal is approximately 30 ksi in$^{1/2}$ in SFGE.
- For sharp cracks, SCC initiation times are short once the line pipe steel is exposed to FGE or FGE blends capable of promoting SCC.
- In the cyclic load tests, the crack growth rates in SFGE followed a Weibull distribution with the 50th percentile for the distribution of 5.55×10^{-8} mm/s. This rate is about three times higher than maximum rates measured for near neutral pH SCC of underground pipelines.
- Batching does not appear to be a viable method for SCC mitigation.
- The only blends that can be safely transported in existing pipelines without significant modification of the system or operations (Case 1) are those containing less than 15% ethanol.
- All other blends require significant modifications of the system or operations (Case 2), or specially designed systems (Case 3).
- Case 2 could include deaeration of the SFGE, or the addition of inhibitors, and is being studied, in detail, as a part of SCC 4-3.
- Case 3 is the subject of ongoing research (SCC 4-5).

All of the SCC 4-4 Phase 2 research was performed with the simulated fuel grade ethanol. This was done to avoid experimental difficulties associated with degradation or changes in FGE in the long-term cyclic load tests. In the Phase 1 work, the potency of the SFGE was compared with one lot of corn-based FGE and the results were similar. Therefore, while there is no evidence to indicate that the use of actual FGE would affect any of the conclusions of the Phase 2 work, some additional verification would be worthwhile.

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1.0 BACKGROUND

Ethanol has been used for the last several years as an environmentally friendly alternative to methyl tertbutyl ether (MTBE), which is an oxygenate additive to gasoline, to increase octane levels and to facilitate the combustion process. The need to find alternatives to imported oil and gas has spurred the increased use of ethanol as an alternative fuel source. Further, ethanol is being promoted as a potential trade-off for CO_2 emissions from the burning of fossil fuels since CO_2 is consumed by the plants used as the ethanol source. Legislation has been passed by Congress that mandates a significant increase in fuel ethanol usage over the next twenty years. The widespread usage of ethanol will require efficient and reliable transportation from diverse ethanol producers to distribution terminals. Pipelines are, by far, the most cost-effective means of transporting large quantities of liquid hydrocarbons over long distances. For transporting ethanol, both existing pipeline infrastructure and new pipeline construction are being contemplated.

As early as 1995, stress corrosion cracking (SCC) of storage tank bottoms and associated terminal equipment that contained fuel grade ethanol (FGE) was noted. A subsequent survey of the industry, funded by the American Petroleum Institute (API) and the Renewable Fuels Association (RFA), indicated that SCC had been observed mainly in user terminals and not in ethanol producer tanks, in rail/tank car/shipping transportation, or in end-user systems (e.g., gas tanks) [1]. Detailed laboratory studies, also funded by API and RFA [2], demonstrated that, in ASTM D-4806 [3] FGE, oxygen is the most important factor causing SCC, followed in importance by pre-existing scale on steel, chloride, and methanol. The corrosion inhibitor typically added to FGE, Octel DCI-11, did not have any effect on exacerbating or mitigating SCC (note: Octel DCI-11 was specified mainly to mitigate corrosion of automotive components and was not designed to mitigate SCC.) The grade of wrought carbon steel appears to be less important, although some of the metallurgical changes engendered by welding seem to have a significant adverse effect on SCC. A parallel study, funded by PRCI [4], has shown that some oxygen scavengers and corrosion inhibitors may mitigate SCC. A number of proprietary studies are also being conducted on gasoline blends, oxygen scavengers, etc.

There are variations in the SCC potency of FGE depending on the chemistry of ethanol, production method (wet vs. dry milling), and even between production batches from a single source. The data accumulated thus far indicate that SCC occurs when the corrosion potential of steel is between about -100 mV and +300 mV versus an Ag/AgCl/Ethanol reference electrode. Carbon steel in some producer ethanol exceeds the upper bound value of this corrosion potential and does not show SCC. In all ethanols (regardless of chemistry or impurities), removal of dissolved oxygen mitigates SCC. Surprisingly, it has been found in recent research that removal of oxygen may or may not be associated with a decrease in the corrosion potential in all instances.

2.0 STATEMENT OF NEED

Despite the accumulation of a considerable amount of laboratory data on SCC susceptibility of pipe steel in FGE, significant gaps remain in our understanding of the technical issues associated with pipeline transportability of FGE and its gasoline blends:

- It is unclear whether a safe limit in terms of gasoline-FGE blending that would not cause SCC under any circumstances exists. This blending limit may depend on ethanol chemistry.
- SCC initiation and growth rates in FGE and blends are not known. The tests conducted to date are essentially screening type tests focused on identifying the effects of environmental variables on a cracking/no cracking pass/fail basis. The initiation and growth rate data may permit better estimation of acceptable batching procedures or FGE residence times.
- The effect of metallurgical changes due to welding or casting on SCC is not known.
- The implementation of various SCC mitigation methods in the field has not been developed. For example, the effectiveness of oxygen scavengers may depend on dosage, introduction mode, gas/liquid phase ratio, flow rate, presence of solids, residence time, etc.
- The interactions between FGE/blends and drag reduction agents, scale, internal coatings, or corrosion products/deposits have not been characterized.
- The effect of ethanol on other non-metallic components, such as seals and gaskets, is not fully understood.
- The effect of various mitigation methods on end-use components (engines and tailpipe emissions, gas tanks, etc.) is not known.
- A systematic methodology incorporated in a guideline document, by which risks of accepting a particular ethanol chemistry and blend can be assessed, is needed. Such a guidelines document is one of the eventual goals of the proposed program.

PRCI SCC 4-4 addressed some of these gaps. Other programs being planned by PRCI and others will address the remaining gaps with the ultimate goal of collating the results of all planned efforts to develop a coherent methodology for sound decision-making.

3.0 OBJECTIVES

The major objectives of the PRCI SCC 4-4 program were to:

1. Develop data necessary to make engineering assessments of the feasibility of transporting FGE and FGE blends in existing pipelines. The transportation may be in a dedicated pipeline or in a batching mode.
2. Identify ethanol blends that can be transported in existing pipelines without significant modification of the system and operations, (Case 1); blends that require significant modifications, (Case 2); and blends that cannot be transported in existing pipelines, but could be moved in specially designed systems, (Case 3).
3. Characterize the time to initiation of SCC in a range of potent ethanol environments and identify safe operating and or batching practices that prevent the initiation and growth of SCC.

4.0 SCOPE OF WORK

A multi-phased program was performed, structured to obtain sufficient information in a six- to twelve-month period to develop an engineering assessment of the transportability of FGE and its gasoline blends in the existing pipeline infrastructure. Phase 1 (Screening Tests on Transportability of Blends) consisted of four tasks:

Task 1-1 (Screening SCC Tests to Determine FGE Blends that can be Transported in Existing Pipeline without Significant Modification),

Task 1-2 (Evaluation of the Effect of Ethanol Blends on Static Elastomeric Seals),

Task 1-3 (Evaluation of the Effect of Ethanol Blends on Dynamic Elastomeric Seals) and,

Task 1-4 (Reporting and Program Management).

Phase 2 (Time to Failure Tests under Static and Dynamic Loads for Assessing Batching Operations) consisted of three tasks: Task 2-1 (Static Load Tests); Task 2-2 (Cyclic Load Tests); and Task 2-3 (Reporting and Management).

The Phase 1 work was completed and the Task Reports were issued. This report summarizes the results of Phase 2 of the program.

5.0 SUMMARY OF TASK 1-1 RESULTS

The slow strain rate (SSR) test technique was used as a screening tool to determine FGE blends that can be transported in existing pipelines without significant modification. Several initial SSR tests were performed with un-notched base metal specimens to establish the optimum chloride concentration of the simulated FGE (SFGE) for the subsequent tests. The main matrix of tests in Task 1-1 was performed with SSR specimens containing notches in the gage section. Variables in the matrix included steel grade, long seam weld type, location of the notch with respect to the seam weld (in the weld metal, in the heat-affected zone (HAZ), or in the base metal), ethanol chemistry, and the ethanol-gasoline blend ratio. Steel grades evaluated included X42, X46, X52, X60 and cast steel. Weld types evaluated included electric resistance welds (ERW), double submerged arc welds (DSAW) and girth welds.

The majority of the tests were performed using the SFGE containing 5 ppm Cl. This concentration of Cl was chosen to produce a relatively aggressive SFGE that accurately mimics the intergranular/mixed mode fracture behavior typically observed in field failures. Several tests also were performed with one lot of corn-based FGE. A strain rate of 1×10^{-6} sec^{-1} was used for all of the SSR tests with smooth specimens while a displacement rate of 9.53×10^{-6} mm/s (3.75×10^{-7} inches/s) was used for all of the SSR tests with notched specimens. As a comparison, a standard tensile test duration is several minutes; whereas, a slow strain rate test duration is three to five days depending on the severity of cracking.

The tests were performed in stainless steel test cells at room temperature. The cell was actively sparged with breathing air at a flow rate of approximately 4 ml/minute. Specimens were tested under freely corroding conditions and the corrosion potential was monitored in each test using an Ag/AgCl/EtOH reference electrode. In all tests, a piece of rusted pipe steel was placed in the test cell and galvanically connected to the test specimen to more closely simulate the native corrosion potential of a mill scaled/rusted pipe wall. The rusted steel to specimen area ratio was approximately 5 to 1. The rusted carbon steel samples were prepared by exposing mill scaled samples of an X52 line pipe steel to aerated deionized water for approximately two weeks.

After testing, the specimens were examined and optically photographed. Some specimens were broken open by cooling then in liquid nitrogen and fracturing them with a hammer blow. The fracture surfaces were examined in the scanning electron microscope (SEM) and the maximum depth of SCC was measured. Other parameters that were recorded for each test included the maximum load and the time to failure.

Below are the conclusions from the SSR tests performed on different line pipe steels and weld types in Task 1-1 of Project PRCI SCC 4-4. It should be recognized that the SSR test technique is a very aggressive SCC test technique; the observation of SCC in a SSR test does not necessarily indicate that SCC will occur in service.

- No SCC was observed in SSR tests in aerated 10% (by volume) ethanol blend (E-10), prepared with SFGE.
- Significant SCC was observed in SSR tests in aerated ethanol-gasoline blends having ethanol concentrations of 20% (by volume) or higher.
- Significant SCC was observed with both simulated and one lot of corn-based FGE but the simulated FGE was a slightly more potent SCC agent.
- The base metal of all of the steels evaluated exhibited measurable susceptibility to SCC and the differences probably are not significant from an integrity standpoint.
- Crack growth rates for the seamless, the cast steel, and the LFERW line pipe steels were somewhat lower than for the DSAW and two other ERW line pipe steels but all steels exhibited relatively deep cracks.
- No major effect of weld metallurgy on SCC behavior was observed in SSR tests of base metal, weld metal, and heat affected zone specimens from girth welds and DSAW long seam welds, although the weld metals appeared to be somewhat more resistant to cracking.
- The absence of SCC in several tests with HFERW and LFERW specimens, where the notch was located at or near the bond line of the long seam weld, was attributed to the poor mechanical properties of the bond line.
- In two SSR tests with one LFERW pipe steel, the bond line did appear to be more resistant to SCC in the ethanol-gasoline blends than the base metal of that steel.

6.0 PHASE 2 EXPERIMENTAL APPROACH

All testing in Phase 2 of the PRCI program was performed with pre-cracked compact tension specimens machined from an X46 line pipe steel. The pipe had a double submerged arc welded (DSAW) longitudinal seam weld (1238 line pipe steel) and was manufactured in approximately 1960. Table 1 shows the composition of the steel. A schematic and picture of the test specimen are shown in Figure 1. The specimens were oriented such that the through-wall pre-crack propagates in the axial pipe direction. The use of a pre-cracked, compact tension geometry is justified because of the likely presence of undetected flaws in pipelines. The crack growth in the tests was monitored continuously using the electric potential drop (EPD) technique, in accordance with the procedures found in ASTM E647-00 [5]. With this technique, a direct current of 20 amperes was passed through the specimen and the change in resistance of the specimen, as a result of crack extension, was monitored. The resistance was converted to a crack length using the Johnson equation found in ASTM E647 [5].

The Phase 2 tests were performed at room temperature in a 4 L stainless steel test cell. Photographs of the test cell are shown in Figures 2 and 3. The test solutions (SFGE and SFGE gasoline blends) were actively sparged with breathing grade air at a flow rate of approximately 4 ml/minute. An ethanol bubbler trap was used on the outlet of the test cell to exclude moisture.

All of the SCC 4-4 Phase 2 research was performed with a simulated fuel grade ethanol (SFGE). This was done to avoid experimental difficulties associated with degradation or changes in FGE in the long-term cyclic load tests. In the Phase 1 work, the potency of the SFGE was compared with one lot of actual corn-based FGE and the results were similar. Table 2 shows the recipe for preparation of the SFGE and Table 3 show the target composition. Note that the SFGE contained 5 ppm Cl and other additives and contaminants found in the ASTM D4806-09 specification. (3) The SCC tests were performed under freely corroding conditions and the corrosion potential was periodically monitored in each test using a Ag/AgCl EtOH reference electrode. A piece of rusted pipe steel was placed in the test cell and galvanically connected to the test specimen to more closely simulate the native corrosion potential of a mill scaled/rusted pipe wall. The rusted carbon steel samples were prepared by exposing mill scaled samples of an X52 line pipe steel to aerated deionized water for approximately two weeks. The rusted steel to specimen area ratio was approximately 5 to 1. The specimen and rusted steel piece were electrically isolated from the specimen grips and test cell in the test machine.

At the end of the crack growth tests, selected specimens were electric discharge machined (EDM) in half. One-half was metallographically prepared and examined. The other half was broken open by cooling it in liquid nitrogen and fracturing it with a hammer blow. The fracture surface was examined optically and in the scanning electron microscope (SEM).

6.1 Task 2-1 (Static Load Tests)

The purpose of the Task 2-1 tests was to estimate the threshold stress intensity factor for SCC (K_{thSCC}) and ensure that all of the Task 2-2 tests were performed at K_{max} values above K_{thSCC}. This parameter also can be used to compare the relatively potency of different blends or lots of FGE and estimate what size flaw in a pipeline will be prone to grow by an SCC mechanism. With this test technique, the compact tension specimen was strained at a constant displacement rate, using a slow strain rate (SSR) test frame. The displacement was stopped at a specified maximum $K_{applied}$, or when there was evidence of crack extension from the EPD measurements. The specimen was left in the loading frame under this constant displacement (static) loading. If SCC growth occurred during the constant displacement rate step, then the crack would extend and the load would drop during the constant displacement step. This process continued until K_{thSCC} was reached. Once no additional load drop or increase in EPD was measured for one to two months, the specimen was removed from the test frame and examined as described above. The K_{thSCC} value was calculated based on the final load and crack length.

For the Task 2-1 tests, a displacement rate of approximately 6×10^{-7} mm/s was used. This rate approached the slowest rate for the SSR test frames. The hold time for the constant displacement rate step was 20 days for Specimen 4-4 Base 3 and 60 days for 4-4 Base 5. The time was extended for the second test to determine whether the threshold dropped significantly with exposure time under constant displacement.

6.2 Task 2-2 (Cyclic Load Tests)

The majority of tests in Phase 2 were performed on compact tension specimen under cyclic loading conditions. These load conditions are designed to simulate the loading conditions on a just- surviving crack in a pipeline that has been previously hydrostatically tested. The imposition of a cyclic load is important since it produces continuous micro-plastic deformation that enhances SCC growth and simulates the ripple load effect from pressure fluctuations on an operating pipeline. The ratio of the minimum to maximum load (R ratio) in the tests ranged from 0.6 to 0.8 and the cyclic frequency was 1.2×10^{-4} Hz (one cycle every 2.3 hours). These conditions are typical of cyclic pressure fluctuations on liquid petroleum pipelines. For each specimen, cracking was initiated in an aggressive SFGE and propagated for approximately one-month period. The test conditions were changed periodically to evaluate the effect of the variables in the test matrix on crack growth. This crack growth experimental technique was developed by DNV Columbus over a 15-year period in research on external SCC of petroleum pipelines. In some tests, it was necessary to temporarily increase the cyclic frequency, to approximately 7.6×10^{-3} Hz (one cycle every 2.2 minutes), or apply an unload-reload cycle to initiate cracking.

Variables in the testing included the location of the crack with respect to the long seam weld (in the weld metal, in the heat affected zone (HAZ), and in the base metal), the exposure environment (SFGE or SFGE-gasoline blends), and the effect of batch cycles.

7.0 PHASE 2 EXPERIMENTAL RESULTS

Table 4 summarizes the results of all of the Phase 2 crack growth tests. The first column in the table is the specimen number. The term Base in the specimen number refers to the location of the pre-crack in the specimen with respect to the weld; Base = pre-crack located in the base metal, HAZ = pre-crack located in the heat affected zone of the weld, and Weld = pre-crack located in the weld metal. The second column in the table is the time period over which the test was conducted using a specific test condition. As described above, the test conditions wwere changed periodically for each specimen in order to asses the effects of the parameters on SCC behavior. The third column is the R ratio, which is the ratio of the minimum to maximum load. Testing was performed at R ratios of 0.6 and 0.8. Initial tests were performed at an R ratio of 0.8 but cracking did not initiate in some tests so it was decided to run the majority of the later tests under more aggressive cycling, with an R ratio of 0.6. The fourth column in the table is the test environment. All tests were started with the simulated FGE, and; in some tests, the environment was changed during the test. The fifth and sixth columns are the maximum K and range in K (ΔK), in ksi $in^{1/2}$ for the test period. The seventh column is the amount of crack growth measured from the EPD during the test period and the eighth column is the resulting crack growth rate. The last column contains comments about the test period.

7.1 Task 2-1 (Static Load Tests)

Two tests were performed to establish the threshold stress intensity factor for ethanol SCC; 4-4 Base 3 and 4-4 Base 5. As described above, a different experimental approach was used for these tests than that used for the cyclic load tests. In the Task 2-1 tests, the compact tension specimen was strained at a constant displacement rate, using a SSR test frame and the displacement was stopped when there was evidence of crack extension from the EPD measurements. The specimen remained in the loading frame under this constant displacement (static) loading until crack growth stopped.

Crack length versus time data for 4-4 Base 3 are shown in Figure 4. The figure shows that there was a large jump in the measured crack length after about five days of straining. This likely was the result of some initial tearing of the pre-existing fatigue crack, or a significant increase in the plastic zone size. There was then a steady increase in the crack length for the next ten days, with an average crack-growth rate of 5.48×10^{-8} mm/s. The crosshead was stopped on Day 16 of the test. The crack continued to extend for another four days, at an average crack-growth rate of 2.41×10^{-8} mm/s. The total amount of SCC crack extension was estimated to be approximately 120 µm, based on the EPD data. There was no evidence of additional growth for the remaining 15 days of the test.

Figure 5 is a low magnification SEM photograph of the fracture surface following breaking open half of the specimen in liquid nitrogen. The machined notch, fatigue pre-crack and brittle rapid fracture regions are shown. At this magnification, the SCC zone cannot be resolved. Figure 5 is a higher magnification SEM photograph of the fracture surface between the fatigue pre-crack and the rapid fracture region. Intergranular facets characteristic of SCC are evident. The width of the SCC zone is 100 to 120 µm at this location, which is very close to the SCC growth region estimated from the EPD readings.

The threshold stress intensity factor for SCC, K_{thSCC}, was calculated based on the final crack length and load, giving a value of 30.3 ksi $in^{1/2}$. A similar procedure was used in the longer-term test, 4-4 Base 5 and a K_{thSCC} value of 33.5 ksi $in^{1/2}$ was estimated based on the final crack length and load. Note that all of the cyclic load tests were performed at K_{max} values above these threshold K values.

7.2 Task 2-2 (Cyclic Load Tests)

7.2.1 Effect of Metallurgy

Eight tests were initiated with base metal specimens in SFGE (1,2,4,6,7,9,10, and 11). For these specimens, there were 32 test periods in which crack growth was monitored in SFGE. These periods varied in length from two to twelve weeks. Table 4 and Figure 7 summarize the crack growth rates for these tests. Crack growth rates for base metal specimens in SFGE ranged between 0 mm/s and 1.43×10^{-7}mm/s (4.48 mm/y), the latter is nearly an order of magnitude higher rate than that typically measured for external near neutral pH SCC of pipelines.

The average crack growth rate for the base metal specimens was 5.92×10^{-8} mm/s and the median crack growth rate was 5.14×10^{-8} mm/s. The zero (crack growth rate) data were excluded from the data set and the remainder of the data was statistically analyzed. A Probability Plot of the data is shown in Figure 8. The fit to a Weibull distribution was excellent, passing the Anderson-Darling goodness of fit test at the 95% confidence level. The 50th percentile for the distribution was 5.55×10^{-8} mm/s, the 95% Upper Confidence Limit (UCL) was 7.27×10^{-8} mm/s, and the 95% Lower Confidence Limit (LCL) was 4.24×10^{-8} mm/s.

One HAZ specimen was tested and there were five separate test periods, over 475 days, in which the specimen was exposed to SFGE. Between these periods, the specimen was exposed to E-50, gasoline, batching with gasoline, and one inhibitor. Table 4 and Figure 9 summarize the crack growth rates for these test periods. Crack growth rates ranged between 1.7×10^{-9} mm/s and 3.72×10^{-8} mm/s (1.17 mm/y). The average crack-growth rate was 1.43×10^{-8} mm/s and the median crack growth rate was 1.41×10^{-8} mm/s. These values are considerably lower than those measured for the base metal specimens but the sample size was considerably smaller and the R ratio was higher than for most base metal tests. An analysis of the means was performed comparing the crack growth rate data for the HAZ specimen and the base metal specimens and the differences was not significant at a 95% confidence level (alpha = 0.05). Given the small sample size, the data were analyzed at a 90% confidence level (alpha = 0.1) and the difference was significant at this lower confidence level.

Three weld metal specimens were tested and there were six separate test periods, over 84 days, in which the specimens were exposed to SFGE. Between these periods, the specimens were exposed to gasoline, or unload – reload transients were applied to the specimens to initiate cracking. No cracking could be initiated in one of the specimens (Weld 1) in spite of the fact that two unload reload transients were applied and the solution was changed.

The other two specimens exhibited cracking and a very high crack growth rate (1.18×10^{-7} mm/s) was observed for one of those specimens. This rate is at the upper end of rates observed for the base metal specimens, as show in Figure 7. An analysis of the means was performed comparing the crack growth rate data for the weld specimens and the base metal specimens and the differences were not significant at a 90% or 95% confidence level.

These two weld specimens also exhibited crack growth in the gasoline, following initiation in the SFGE. In the case of specimen Weld - 2, the cracking did not arrest. It is highly likely that the crack growth in the gasoline phase was the result of crack growth under cyclic loading in the inhomogeneous weld, since this type of behavior was never observed with HAZ or base metal specimens. However, this theory could not be confirmed from the fractography because of the mixed mode of the SCC region. Unfortunately, the quasi-cleavage in the mixed mode SCC cracking could not be distinguished from the quasi-cleavage associated with crack growth under cyclic loading.

7.2.2 Effect of Blend Ratio

The results of the crack growth tests were generally consistent with the results of the SSR tests performed in Task 1 of PRCI SCC 4-4. Evidence of crack growth was observed in blends containing 20% (by volume) and higher concentrations of ethanol (prepares with SFGE and gasoline); whereas, continued crack growth was not observed in E-15, E-10, or in gasoline. Typical test data are shown in Figures 10 and 11, for sample Base 4, which is a base metal sample. In this test, the environment was changed from SFGE to E-20 on day seventy-three. Prior to the change, the crack growth rate (in SFGE) was 7.26×10^{-8} mm/s, which is about four times faster than the highest rates reported for external near neutral pH SCC. Over the next 53 days, the average crack-growth rate was only slightly lower, at 5.89×10^{-8} mm/s. On Day 126, the environment was changed to E-10 and there was no measurable growth over the next 73 days. On Day 199, the environment was changed back to SFGE and cracking re-initiated. On Day 218, the environment was changed to E-15 and cracking again arrested but it took approximately 50 days to arrest in the E-15 blend. On Day 298, the environment was changed back to SFGE and crack re-initiated within about one week.

Figure 12 shows the average crack-growth rate as a function of the ethanol concentration in the ethanol-gasoline blends. These data show that the crack growth rate in E-20 is comparable to that in E-95 while the rate is actually measurably higher in E-50. There are three observations of this effect. Figure 13 shows data for Sample 4-4 Base 2 in which the test solution was changed from E-95 to E-50 on Day 96. The increase in the crack growth rate (slope) is evident. Similar behavior was observed with Sample 4-4 Base 14, as shown in Figure 14. Table 4 contains the calculated crack growth rates for these two test specimens. These data show that the crack growth rates in E-50 were almost identical (3.3×10^{-7} mm/s for 4-4 Base 2 and 3.5×10^{-7} mm/s for 4-4 Base 14) while they were somewhat different for the exposure periods in E-95 (8.9×10^{-8} mm/s for 4-4 Base 2 and 4.3×10^{-8} mm/s for 4-4 Base 14).

7.2.3 Effect of Batching

Three batch cycles were evaluated in the project; two short cycles with a twenty-four hour period and one long cycle with a twelve-day period:

Cycle 1: One hour SFGE: 23 hours gasoline

Cycle 2: One hour gasoline: 23 hours SFGE

Cycle 3: Five Days SFGE: 7 Days gasoline

One long cycle test was performed with Sample (4-4 HAZ-1). One short cycle test, with 23 hours of SFGE exposure, was performed with one sample (4-4 Base 7). Three short cycle tests with 1 hour of SFGE exposure were performed (Samples 4-4 Base 6, 4-4 Base 7, and 4-4 Base 11). The results are summarized in Table 4.

A fair amount of disruption in the EPD data was observed in all tests, as a result of the batching process but the behavior in the tests could be estimated from the peaks of the EPD data. Figure 15 shows the average crack growth rates observed for the five tests. This figures shows that measurable crack growth was observed for the single tests with Cycles 2 and 3 and two of the three tests with Cycle 1. Data for the long cycle test are shown in Figure 16. Batch Cycle 3 was started on Day 237 and the growth rate declined only slightly from that which was observed in SFGE. A reasonable estimate of the average crack growth could be made for the long batch cycle based on the relative ratio of exposure time in FGE and in gasoline, assuming that no SCC growth occurs in the gasoline phase.

This approach does not appear to be applicable to the short batch cycle. Surprisingly, a fair amount of growth was observed for two of the three tests with Batch Cycle 1 in which there was very little exposure time to SFGE. Data for Specimen 4-4 Base 7 are shown in Figure 17. Batch Cycle 1 was started on Day 234 and cracking appeared to have arrested over the next 30 days. However, cracking reinitiated and the average crack growth rate over the Batching period (Day 234 to Day 315) was 2.37×10^{-8} mm/s, which is lower than the rate observed in SFGE, but never the less, quite significant.

8.0 DISCUSSION AND CONCLUSIONS

The major objectives of the PRCI SCC 4-4 program were to:

1. Develop data necessary to make engineering assessments of the feasibility of transporting FGE and FGE blends in existing pipelines. The transportation may be in a dedicated pipeline or in a batching mode.

2. Identify ethanol blends that can be transported in existing pipelines without significant modification of the system and operations (Case 1), blends that require significant modifications (Case 2), and blends that cannot be transported in existing pipelines, but could be moved in specially designed systems (Case 3).

3. Characterize the time to initiation of SCC in a range of potent ethanol environments and identify safe operating and or batching practices that prevent the initiation and growth of SCC.

The scope of work consisted of SSR screening tests in Phase 1 of the research and cyclic load tests in Phase 2 of the research. While both test techniques are aggressive from a loading perspective, the cyclic load test conditions are more realistic of pipeline operation. Factors evaluated in the research included pipeline steel grade, welds, FGE-gasoline blends, and batching. The majority of testing was performed with a simulated FGE.

The results of the SSR and cyclic load tests were generally consistent. In DSAW line pipe steel, the base metal, heat affected zone, and weld metal were all susceptible to SCC in SFGE. In the Phase 1 SSR tests, the base metal of all of the steels examined exhibited measurable susceptibility to SCC and the differences probably are not significant from an integrity standpoint. With the possible exception of the high ferrite bond line of one ERW line pipe steel, there is no evidence to indicate that any line pipe steel or weld is resistant to ethanol SCC.

In the tests with blends, no SCC was observed in gasoline and E-10 but significant SCC was observed in E-20 and higher ethanol blends. In the cyclic load tests, crack growth also arrested in E-15, but a relatively long time was required for crack arrest in this blend. One somewhat surprising observation was the high crack growth rates in the E-50 blend in the cyclic load tests. This blend was a more potent SCC agent than SFGE; an effect that was not apparent in the Phase 1 SSR tests.

The results of the batching tests were not promising. For the long (twelve-day) batch cycle, the average crack growth rate could be reasonably estimated based on the exposure time to the SFGE. This rate is too high to be considered a reasonable mitigation method. For the short (twenty-four hour) batch cycle, even short times of exposure to SFGE, resulted in measurable SCC crack growth. This behavior indicates that initiation times are short for sharp cracks.

Referring to the overall project objectives, the following are the conclusions of SCC 4-4:

- Pipelines made of common line pipe steels *(e.g., Grade B and X-42 to X-60)* are likely to be susceptible to ethanol SCC and any differences in susceptibility are probably not significant from an integrity perspective.
- While differences in susceptibility were noted for some weld types, in general, the base metal, heat affected zone, and weld metal were all susceptible to SCC in SFGE.
- The threshold stress intensity factor for SCC initiation, K_{thSCC}, in the base metal is approximately 30 ksi $in^{1/2}$ in SFGE.
- For sharp cracks, SCC initiation times are short once the line pipe steel is exposed to FGE or FGE blends capable of promoting SCC.
- In the cyclic load tests, the crack growth rates in SFGE followed a Weibull distribution with the 50th percentile for the distribution of 5.55×10^{-8} mm/s. This rate is about three times higher than maximum rates measured for near neutral pH SCC of underground pipelines.
- The only blends that can be safely transported in existing pipelines without significant modification of the system or operations (Case 1) are those containing less than 15% (by volume) ethanol.
- All other blends require significant modifications of the system or operations (Case 2), or specially designed systems (Case 3).
- Case 2 could include deaeration of the SFGE, or the addition of inhibitors, and is being studied, in detail, as a part of SCC 4-3.
- Case 3 is the subject of ongoing research (SCC 4-5).
- Batching does not appear to be a viable method for SCC mitigation.

9.0 FOLLOW-ON WORK

All of the SCC 4-4 Phase 2 research was performed with the simulated fuel grade ethanol. This was done to avoid experimental difficulties associated with degradation or changes in FGE in the long-term cyclic load tests. In the Phase 1 work, the potency of the SFGE was compared with one lot of actual corn-based FGE and the results were similar. Therefore, while there is no evidence to indicate that the use of actual FGE would affect any of the conclusions of the Phase 2 work, some additional verification would be worthwhile.

The observation of higher crack growth rates in E-50 compared to lower and higher ethanol blends was unexpected. This may have implications in batching operations where transmix may contain blend ratios in the range of E-50. The effectiveness of inhibitors in E-50 is being evaluated in SCC 4-3. However, the fundamental reason for the higher crack growth rate in E-50 is not understood.

10.0 REFERENCES

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Table 1. Chemical composition of X46 line pipe steel used in the Phase 2 testing.

ELEMENT		X46 DSAW	API 5L X46 *
C	(Carbon)	0.191	0.28 Max
Mn	(Manganese)	0.97	1.25 Max
P	(Phosphorus)	0.006	0.04 Max
S	(Sulfur)	0.017	0.05 Max
Si	(Silicon)	0.017	
Cu	(Copper)	0.016	
Sn	(Tin)	0.002	
Ni	(Nickel)	0.016	
Cr	(Chromium)	0.029	
Mo	(Molybdenum)	0.000	
Al	(Aluminum)	0.003	
V	(Vanadium)	0.001	
Nb	(Niobium)	0.001	
Zr	(Zirconium)	0.000	
Ti	(Titanium)	0.000	
B	(Boron)	0.000	
Ca	(Calcium)	0.000	
Co	(Cobalt)	0.006	
Fe	(Iron)	Balance	Balance

(*) API 5LX, Welded, Cold Expanded, Electric Furnace, 9th Edition, February 1960.

Table 2. Additives used to prepare SFGE.

200-Proof Ethanol	Water	Methanol	Denaturant	Chlorides	Acetic Acid
3785 ml	40 ml	20 ml	150 ml gasoline	0.0265 g NaCl	0.2 ml

Table 3. Target composition of SFGE.

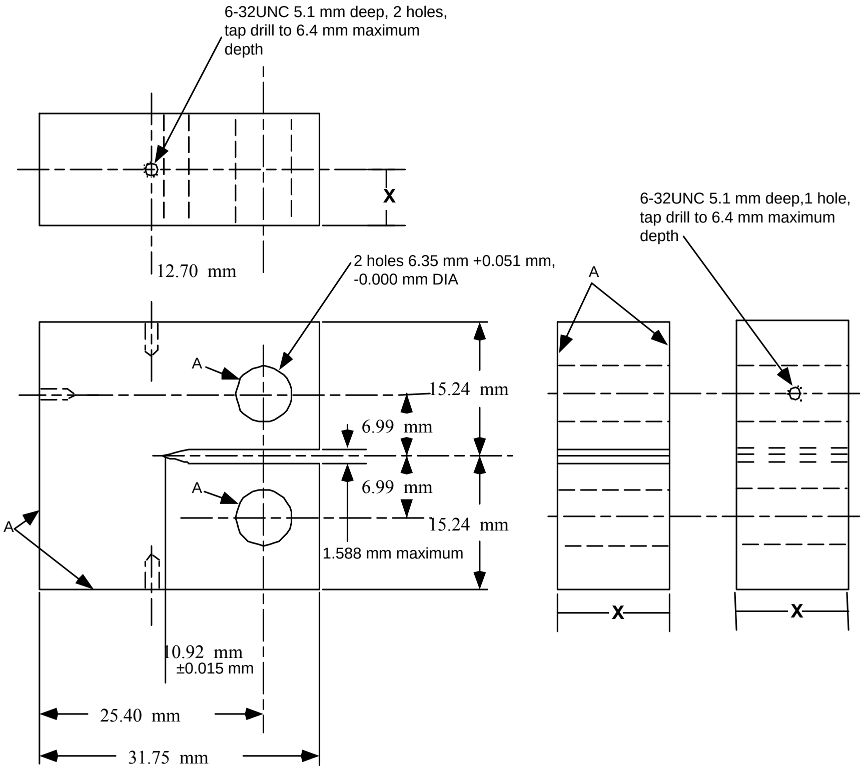
Requirement	ASTM D 4806-09 Limits		PRCI SFGE
	Minimum	Maximum	
Ethanol, vol. %	92.1	–	
Methanol, vol. %	–	0.50	0.50
Solvent-washed gum, mg/100 ml	–	5.0	–
Water content, vol. %	–	1.26	1.0
Denaturant content, vol. %	1.96	5.00	3.75
Inorganic chloride, ppm (mg/L)	–	12.5 (10)	5 ppm
Copper, mg/kg	–	0.1	–
Acidity (as Acetic Acid CH_3COOH), mass % (mg/L)	–	0.007 (56)	(50)
pH_e	6.5	9.0	–

Table 4. Summary of results of Phase 2 crack growth tests.

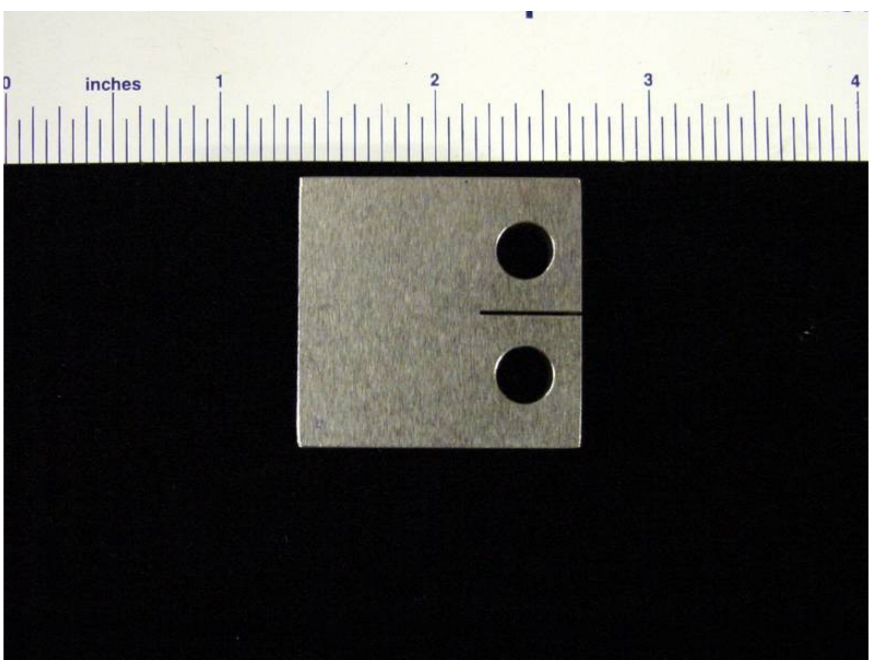
Specimen Number	Time Period, days	R Ratio	Test Environment	K_{max} Start-End, Ksi in$^{1/2}$	ΔK Start-End Ksi in$^{1/2}$	Crack Growth, inches	Crack Growth Rate, mm/s	Note
4-4 Base 1	4 – 39	0.8	SFGE	35.7 – 36.8	7.2 – 7.4	0.0089	7.46E-08	Good Cracking
4-4 Base 1	39 – 60	0.8	Gasoline	36.8 – 36.9	7.4 – 7.4	0.0000	0	No Obvious Crack Growth
4-4 Base 1	60 – 96	0.8	SFGE	36.8 – 36.9	7.4 – 7.6	0.0013	1.07E-08	SCC reinitiated at a slower rate.
4-4 Base 1	96 – 115	0.8	SFGE	37.0 – 36.8	7.6 – 7.3	0.0060	7.52E-08	Unloaded/Reloaded at Day 96
4-4 Base 1	116 – 132	0.64	SFGE	36.8 – 37.0	13.4 – 13.7	0.0057	1.04E-07	Day 116 Switch to R=0.6
4-4 Base 1	132 – 194	0.64	Gasoline	37.0 – 37.3	13.1 – 13.3	0.0000	0.00E+00	Day 132 Changed to 100% Gasoline
4-4 Base 1	194 – 211	0.64	Gasoline	36.6 – 36.8	13.9 – 13.9	0.0000	0.00E+00	Unload/Reload
	211							Test Over
4-4 Base 2	0 – 40	0.8	SFGE	35.7 – 35.9	6.6 – 6.6	0.0022	1.62E-08	Minor SCC Growth
4-4 Base 2	40 – 59	0.6	SFGE	35.9 – 35.9	13.2 – 13.5	0.0000	0.00E+00	Changed to R=0.6 at Day 40
4-4 Base 2	60 – 96	0.6	SFGE	35.9 – 36.8	13.5 – 14.25	0.0105	8.93E-08	Unload-Reload at Day 59
4-4 Base 2	96 – 119	0.6	50% Blend	37.2 – 40.5	14.3 – 14.9	0.0256	3.33E-07	Changed to 50/50 Blend at Day 96
4-4 Base 2	119 – 180	0.6	E10	39.7 – 40.6	15.5 – 16.0	0.0063	3.04E-08	Day 119 Change to E-10 – Day 144-180 CGR was 0
4-4 Base 2	180 – 205	0.61	E10	41.0 – 40.6	16.0 – 16.0	0.0000	0.00E+00	Unload/Reload – End Test Day 205
	205							Test Over
4-4 Base 3	0 – 16	NA	SFGE	0 – 32.6	32.6	0.0030	5.45E-08	Constant Displacement Rate, Loading Stopped at Day 16
4-4 Base 3	16 – 20	NA	SFGE	32.6	0.0	0.0017	2.41E-08	Displacement Held Day 16 to Day 35
4-4 Base 3	20 – 35	NA	SFGE	32.6	0.0	0.0000	0.00E+00	Displacement Held Day 16 to Day 35
	35							Test Over
4-4 Base 4	0 – 29	0.8	SFGE	35.6 – 35.7	6.5 – 6.5	0.0006	2.03E-08	Noisy Crack Growth Data
4-4 Base 4	29 – 37	0.8	SFGE	35.9 – 35.9	7.3 – 7.3	0.0000	0.00	Unload-Reload at 29 Days
4-4 Base 4	37 – 73	0.63	SFGE	36.4 – 37.1	13.6 – 13.9	0.0088	7.26E-08	Changed to R=0.6 at Day 37
4-4 Base 4	73 – 126	0.62	E20	37.1 – 38.4	13.9 – 14.4	0.0109	5.98E-08	Changed to E-20
4-4 Base 4	126 – 199	0.63	E10	38.3 – 38.2	14.4 – 14.4	0.0000	0.00E+00	Changed to E-10
4-4 Base 4	199 – 218	0.63	SFGE	37.9 – 38.9	14.2 – 14.5	0.0072	1.11E-07	Changed to SFGE
4-4 Base 4	218 – 298	0.63	E15	38.9 – 39.3	14.5 – 14.7	0.0037	1.36E-08	Changed to E-15; CGR was 0 last 45 days
4-4 Base 4	298 – 324	0.63	SFGE	39.3 – 39.9	14.7 – 14.9	0.0045	5.16E-08	Changed to SFGE, Day 298
4-4 HAZ 1	0 – 84	0.8	SFGE	36.5 – 37.8	7.1 – 7.4	0.0105	3.72E-08	Good Cracking but Slower than Base-1
4-4 HAZ 1	84 – 115	0.8	E50	37.8 – 37.8	7.4 – 7.4	0.0003	2.75E-09	Changed to E-50 at 84 Days
4-4 HAZ 1	115 – 132	0.8	E50	37.6 – 38.2	7.3 – 7.5	0.0052	9.17E-08	Unload/Reload Day 115
4-4 HAZ 1	132 – 172	0.81	Gasoline	38.0 – 38.0	7.3 – 7.4	0.0000	0.00	Change to 100% Gasoline
4-4 HAZ 1	172 – 236	0.81	SFGE	38.3 – 38.7	7.4 – 7.4	0.0034	1.56E-08	Changed to SFGE

Specimen Number	Time Period, days	R Ratio	Test Environment	K_{max} Start-End, Ksi in$^{1/2}$	ΔK Start-End Ksi in$^{1/2}$	Crack Growth, inches	Crack Growth Rate, mm/s	Note
4-4 HAZ 1	236 – 304	0.81	Batching	38.7 – 38.8	7.6 – 7.5	0.0017	7.37E-09	Batching 7 days Gasoline 5 Days SFGE
4-4 HAZ 1	304 – 329	0.81	SFGE	38.8 – 39.0	7.5 – 7.5	0.0012	1.41E-08	Changed to SFGE
4-4 HAZ 1	329 – 403	0.81	Inhibitor	39.0 – 39.0	7.5 – 7.5	0.0005	1.99E-09	500 ppm 30% NH_4OH
4-4 HAZ 1	403 – 455	0.81	SFGE	39.1 – 39.1	7.5 – 7.6	0.0003	1.70E-09	Changed to SFGE
4-4 HAZ 1	455 – 517	0.8	SFGE	37.4 – 37.4	7.6 – 7.6	0.0002	2.94E-09	Unload-Reload Lowered Load Day 455
4-4 HAZ 1	517							Test Over
4-4 Base 5	0 – 20	NA	SFGE	0 – 32.8		0.0024	3.54E-08	Constant Displacement Rate, Loading Stopped at Day 19
4-4 Base 5	19 – 80	NA	SFGE	32.6		0.0000	0.00E+00	Displacement Held Day 19 to Day 70
	80							Test Over
4-4 Base 6	0 – 17	0.61	SFGE	33.0 – 34.3	13.0 – 12.9	0.0055	9.64E-08	
4-4 Base 6	17 – 30	0.60	SFGE	34.3 – 36.5	12.9 – 13.3	0.0007	1.58E-08	Day 17 Unload-Reload
4-4 Base 6	30 – 42	0.60	SFGE	36.3 – 36.6	14.3 – 14.8	0.0015	3.74E-08	Day 30 Unload-Reload
4-4 Base 6	42 – 66	0.60	SFGE	36.6 – 36.9	14.8 – 14.9	0.0027	3.27E-08	Changed Solution
4-4 Base 6	66 – 77	0.60	SFGE	36.8 – 37.6	14.5 – 14.8	0.0072	1.97E-07	Changed to Fast Cycle Frequency
4-4 Base 6	77 – 135	0.60	SFGE	36.8 – 38.1	15.0 – 15.2	0.0044	2.20E-08	Returned to Standard Cycle Frequency
4-4 Base 6	135 – 154	0.60	SFGE	38.1 – 38.8	15.3 – 15.5	0.0056	9.03E-08	Batching 23 hrs Gasoline 1 hr SFGE
	154							Test Over
4-4 Base 7	0 – 16	0.61	SFGE	35.4 – 35.8	13.7 – 13.9	0.0051	9.40E-08	
4-4 Base 7	16 – 30	0.62	SFGE	35.8 – 36.6	13.8 – 13.9	0.0040	8.29E-08	Day 16 Unload-Reload
4-4 Base 7	30 – 42	0.61	SFGE	36.3 – 36.6	13.9 – 14.1	0.0026	6.50E-08	Day 30 Unload-Reload
4-4 Base 7	42 – 66	0.61	SFGE	36.6 – 36.8	14.1 – 14.2	0.0032	3.88E-08	Changed Solution
4-4 Base 7	66 – 77	0.61	SFGE	36.8 – 37.2	14.2 – 14.2	0.0053	1.43E-07	Changed to Fast Cycle Frequency
4-4 Base 7	77 – 136	0.62	SFGE	37.2 – 37.9	14.2 – 14.3	0.0053	2.65E-08	Returned to Standard Cycle Frequency
4-4 Base 7	136 – 170	0.62	Batching	37.9 – 38.9	14.3 – 14.7	0.0093	7.83E-08	Batching 1 hr Gasoline 23 hrs SFGE
4-4 Base 7	170 – 234	0.62	SFGE	38.9 – 39.4	14.7 – 14.9	0.0049	2.28E-08	Batching Stopped
4-4 Base 7	234 – 316	0.62	Batching	39.5 – 40.1	15.0 – 15.2	0.0066	2.37E-08	Batching 23 hrs Gasoline 1 hr SFGE
4-4 Base 7	316 – 356	0.62	Gasoline	40.1 – 40.1	15.2 – 15.2	0	0.00E+00	Changed to Gasoline, Day 356
4-4 Base 8								Test Over No Data
4-4 Base 9	45 – 93	0.63	SFGE	38.0 – 38.7	14.1 – 14.6	0.0061	3.74E-08	
4-4 Base 9	93 – 195	0.63	Inhibitor	38.7 – 39.5	14.6 – 14.8	0.0082	2.36E-08	500 ppm DEA
4-4 Base 9	195 – 243	0.63	SFGE	39.5 – 40.3	14.8 – 15.1	0.0066	4.04E-08	Flushed cell New SFGE, Day 195
4-4 Base 9	243 – 281	0.61	SFGE	38.6 – 39.1	15.2 – 15.3	0.0034	2.63E-08	Lowered Load, Day 243

Specimen Number	Time Period, days	R Ratio	Test Environment	K_{max} Start-End, Ksi in$^{1/2}$	ΔK Start-End Ksi in$^{1/2}$	Crack Growth, inches	Crack Growth Rate, mm/s	Note
4-4 Base 10	26 – 68	0.66	LTV-200	33.5 – 33.4	11.3 – 11.4	0	0	LTV (mineral oil)
4-4 Base 10	72 – 80	0.61	SFGE	33.2 – 35.7	13.1 – 14.1	0.0246	9.04E-07	Changed to Fast Cycling, Day 72
4-4 Base 10	80 – 131	0.61	SFGE	36.6 – 37.2	14.2 – 14.5	0.0073	4.30E-08	Changed to Regular Cycling, Day 80
4-4 Base 10	131 – 148	0.61	SFGE	37.2 – 37.2	14.5 – 14.5	0.0006	1.04E-08	Added 500 ppm 30% NH_4OH, Day 131
4-4 Base 10	148		SFGE					Changed to SFGE, Day 148
4-4 Base 11	22 – 68	0.62	SFGE	34.1 – 34.9	13.0 – 13.3	0.0098	5.01E-08	Establishing Cracking
4-4 Base 11	68 – 126	0.62	Batching	34.9 – 34.0	13.3 – 13.2	0	0.00E+00	Batching 23 hrs Gasoline 1 hr SFGE
4-4 Base 11	126 – 141	0.61	SFGE	34.0 – 34.0	13.2 – 13.2	0	0.00E+00	Changed to SFGE, Day 141
4-4 Base 11	141 – 159	0.62	SFGE	34.0 – 33.7	13.2 – 12.9	0.0002	3.17E-09	Unload-Reload, Day 141
4-4 Base 12	0 – 121	0.61	SFGE	34.9 – 37.9	13.7 – 15.1	0.0315	7.65E-08	Start
4-4 Base 12	121 – 132	0.6	SFGE	37.9 – 37.9	15.1 – 15.0	0	0.00E+00	Added Inhibitor MCC 062909-1, Day 121
4-4 Base 13	0 – 32	0.58	SFGE	32.7 – 33.1	13.7 – 13.8	0.0066	6.06E-08	Establishing Cracking
4-4 Base 14	65 – 105	0.65	SFGE	34.0 – 34.4	11.9 – 12.0	0.0058	4.32E-08	Establishing Cracking
4-4 Base 14	105 – 127	0.65	E-50	34.4 – 37.0	12.0 – 12.8	0.0262	3.50E-07	Changed to E-50, Day105
4-4 Weld 1	0 – 17	0.7	SFGE	36.4 – 36.0	11.3 – 10.9	0.0000	0.00E+00	Establishing Cracking
4-4 Weld 1	17 – 30	0.7	SFGE	37 – 36.6	11.4 – 10.8	0.0000	0.00E+00	Day 17 Unload-Reload
4-4 Weld 1	30.0 – 42	0.7	SFGE	36.6 – 36.9	10.9 – 10.9	0.0000	0.00E+00	Day 30 Unload-Reload
4-4 Weld 1	42 – 64	0.71	SFGE	36.9 – 36.6	10.9 – 10.5	0.0000	0.00E+00	Changed Solution
4-4 Weld 1	64 – 77						0	Changed to Fast Cycling Day 64
4-4 Weld 2	0 – 33	0.62	SFGE	34.2 – 39.2	12.9 – 14.7	0.0422	3.79E-07	Establishing Cracking
4-4 Weld 2	33 – 61	0.63	Gas	39.2 – 41.8	14.7 – 15.6	0.0178	1.87E-07	Changed to Gasoline, Day 33
4-4 Weld 2	61 – 84	0.62	Gas	40.9 – 40.9	15.4 – 15.4	0	0.00E+00	Lowered Load, Day 61
4-4 Weld 2								Test Over
4-4 Weld 3	8 – 32	0.64	SFGE	32.7 – 33.6	11.9 – 12.2	0.0096	1.18E-07	Establishing Cracking
	32 – 50	0.64	Gasoline	33.6 – 33.6	12.2 – 12.2	0.0002	3.27E-09	Changed to Gasoline on Day 32
4-4 Weld 3	50 – 59	0.64	Gasoline	33.6 – 33.7	12.2 – 12.3	0.0015	4.79E-08	Crack Growth Rate Increased, Beginning Day 32
4-4 Weld 3	59 – 72	0.64	Gasoline	33.7 – 33.7	12.3 – 12.3	0.0000	0.00E+00	Crack Growth Rate Decreased to 0 Day 59-72
4-4 Weld 3	32 – 72	0.64	Gasoline	33.6 – 33.7	12.3 – 12.3	0.0017	1.25E-08	Gasoline Period
4-4 Weld 3	72 – 84	0.64	SFGE	33.7 – 34.0	12.3 – 12.3	0.0027	6.61E-08	Changed to SFGE on Day 72, Running



(a) Schematic



(b) Photograph

Figure 1. Compact tension specimen.

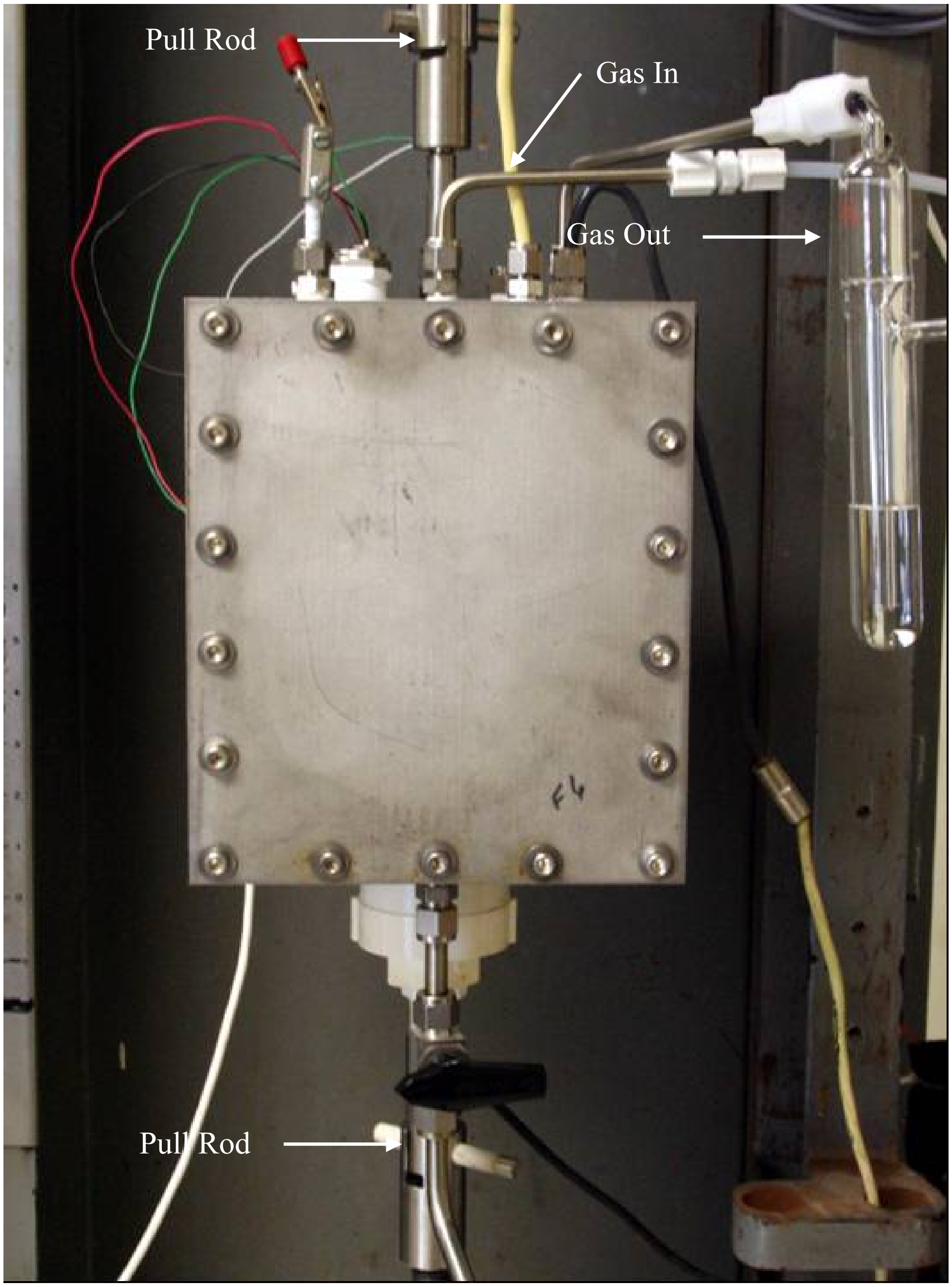


Figure 2. Photograph of test cell used for crack-growth testing.

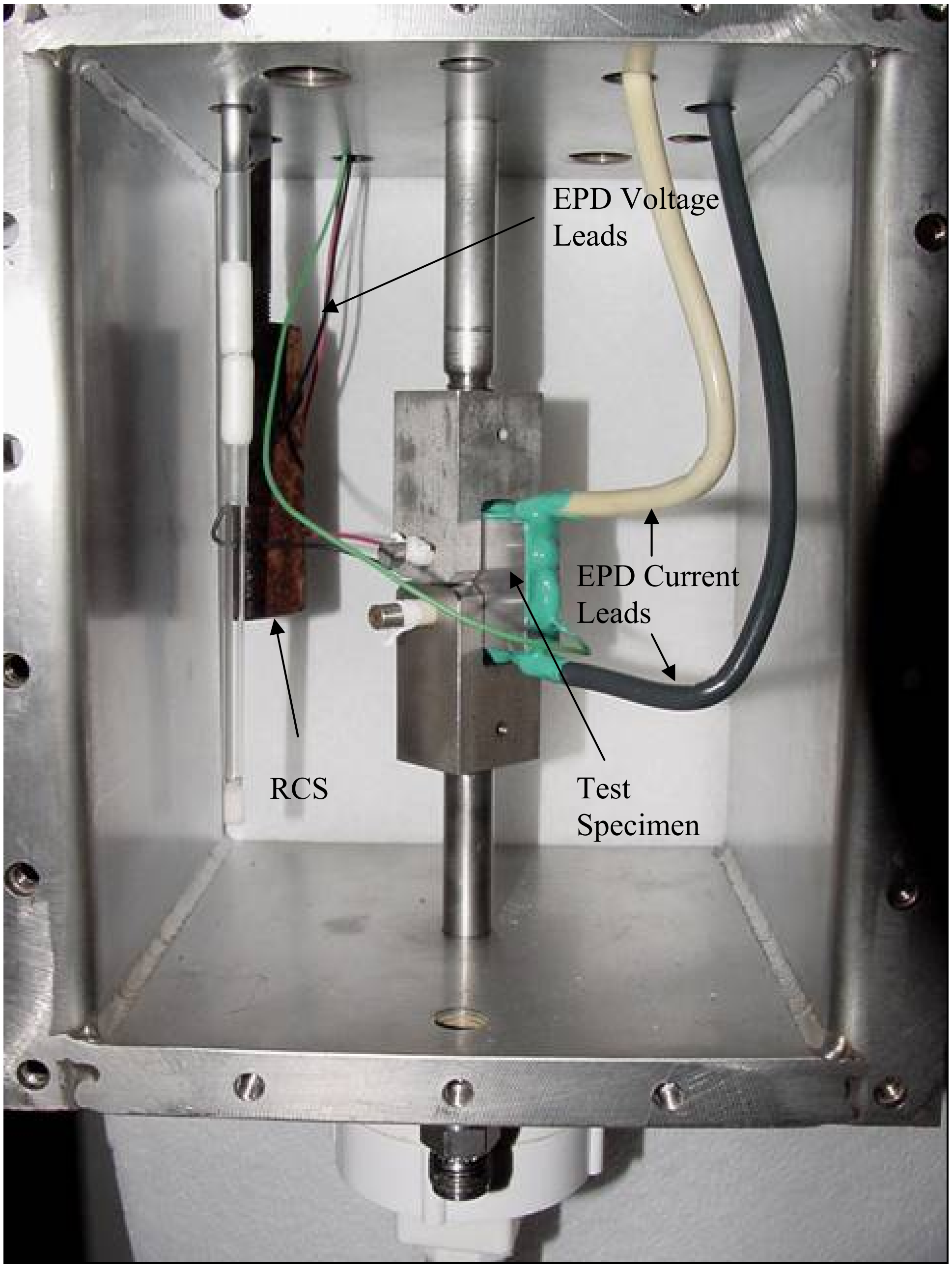


Figure 3. Photograph of the inside of the test cell used for crack-growth testing, showing the test specimen and cell internals; RCS –Rusted Carbon Steel Sample, EPD – Electric Potential Drop.

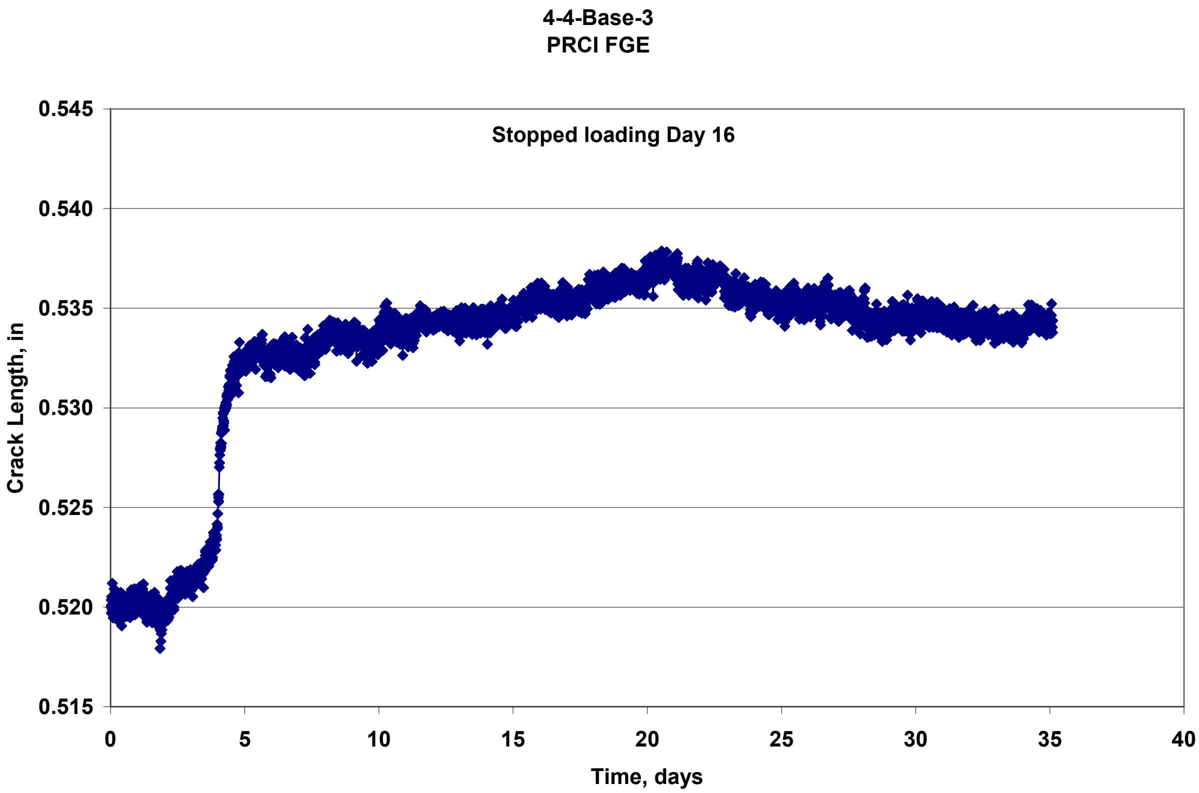


Figure 4. Crack length as a function of time for Specimen 4-4 Base 3.

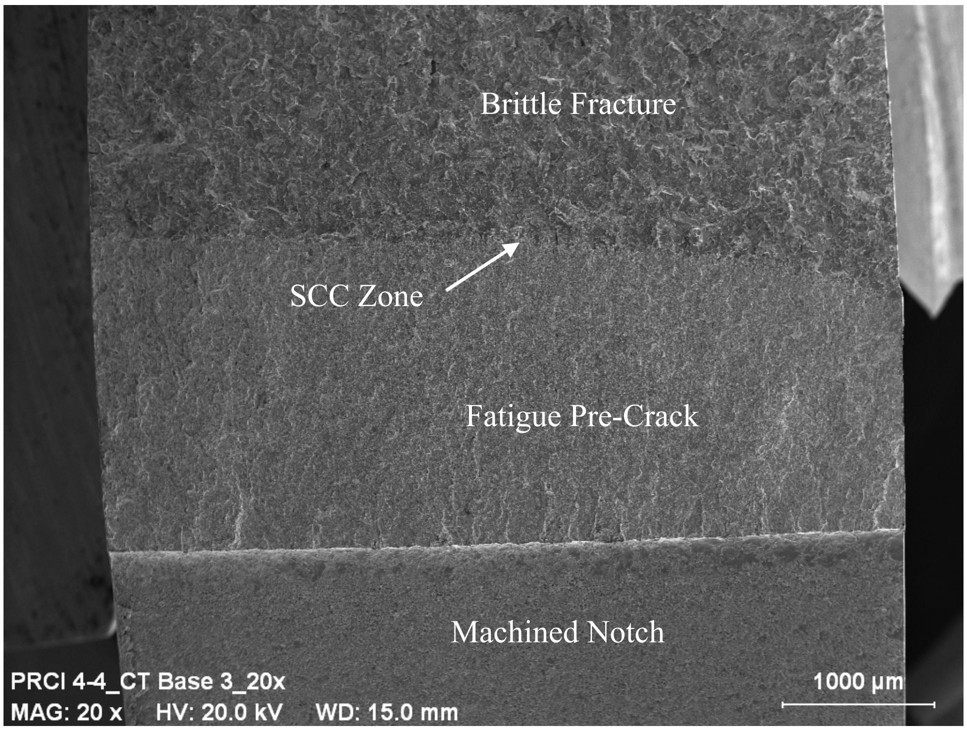


Figure 5. Low magnification SEM photograph of fracture surface of Specimen 4-4 Base 3.

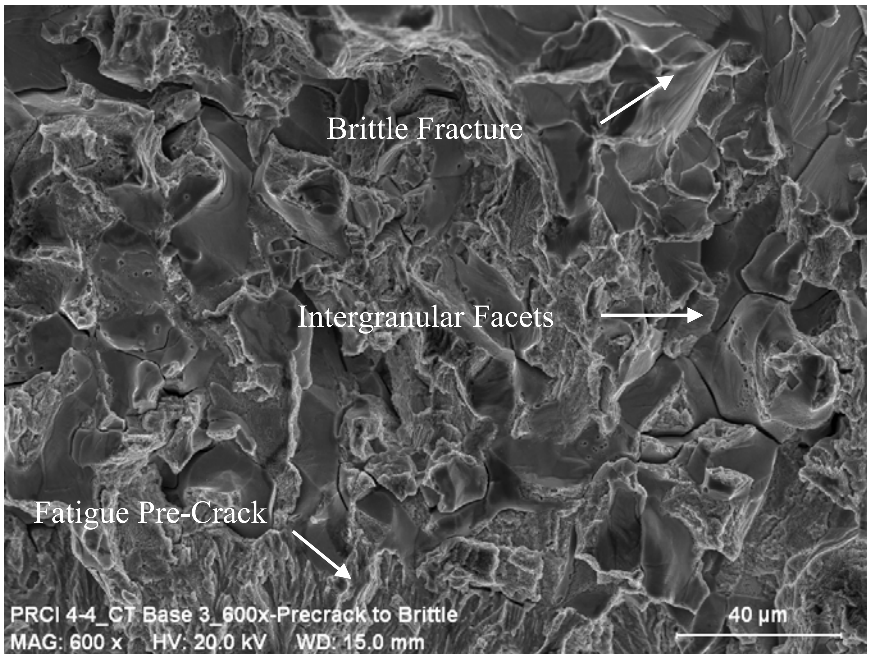


Figure 6. SEM photograph of fracture surface of Specimen 4-4 Base 3 showing the SCC zone.

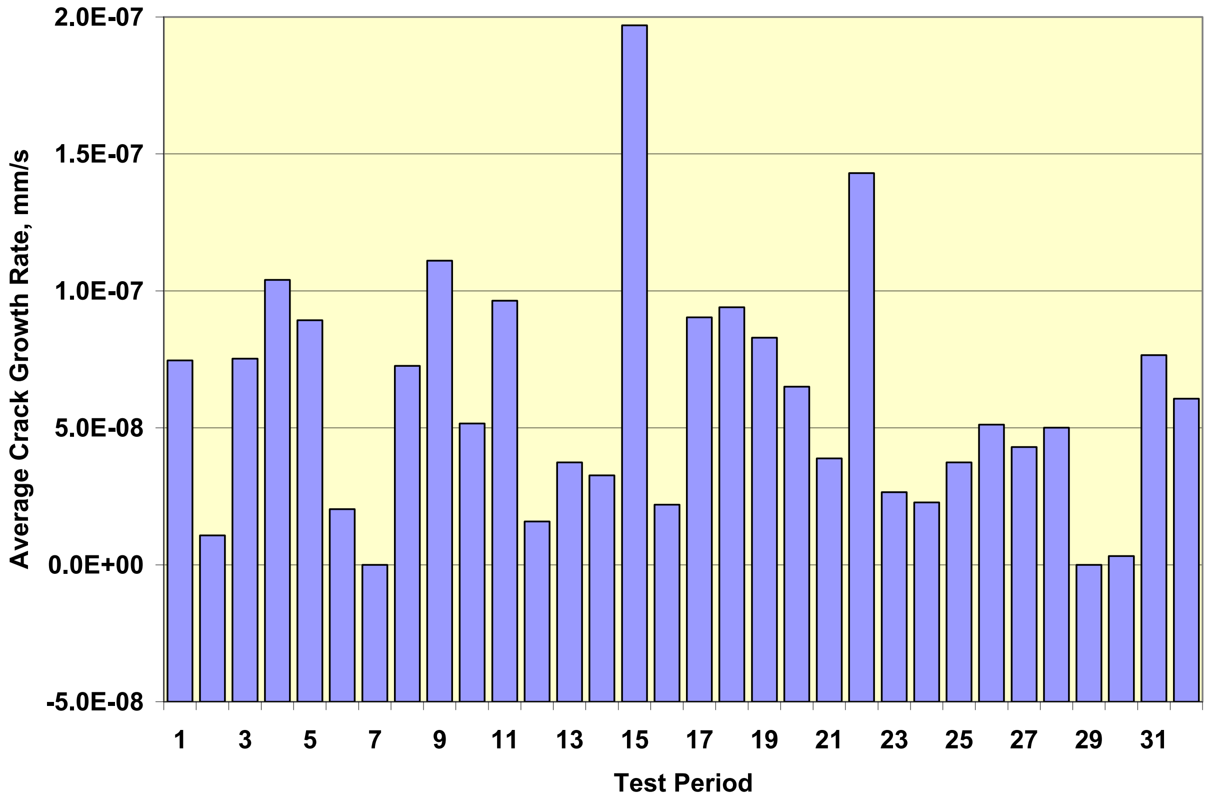


Figure 7. Crack growth rates for tests performed on base metal specimens in SFGE. The periods indicate segments of time for different specimens in which the samples were exposed to SFGE. They vary in length from approximately 2 to 12 weeks; see Table 4.

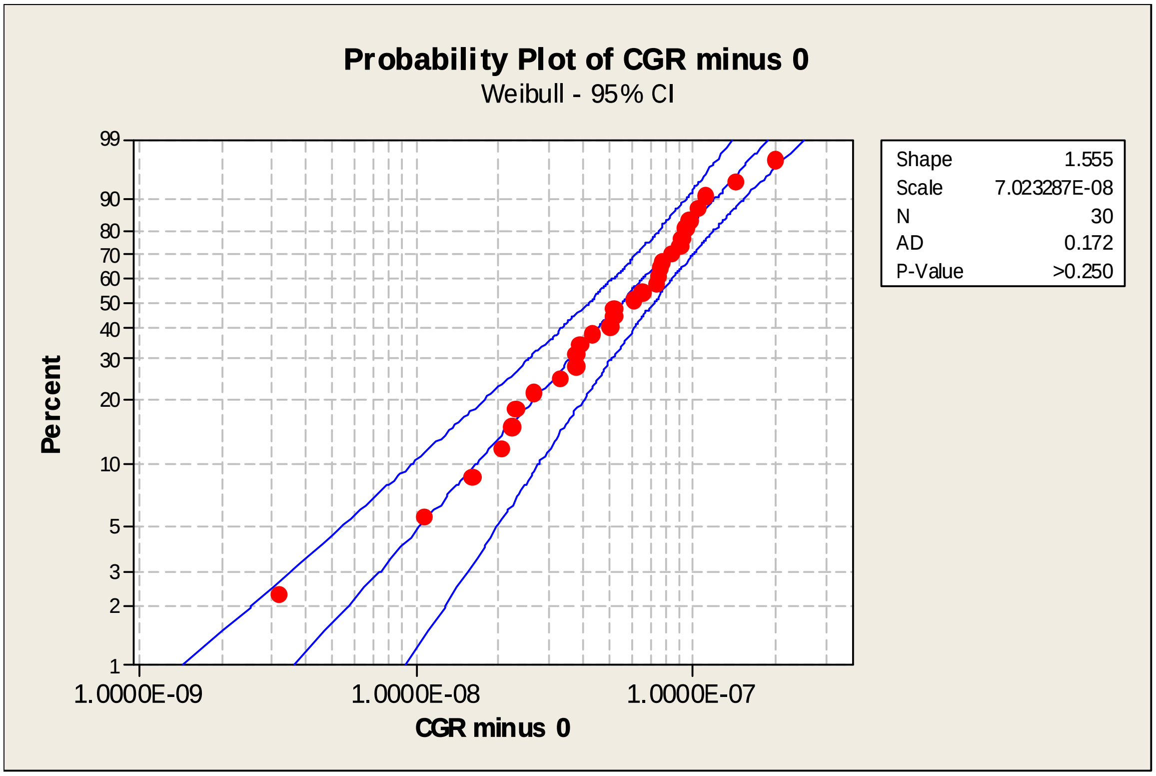


Figure 8. Probability plot of crack-growth rates for tests performed on base metal specimens in SFGE.

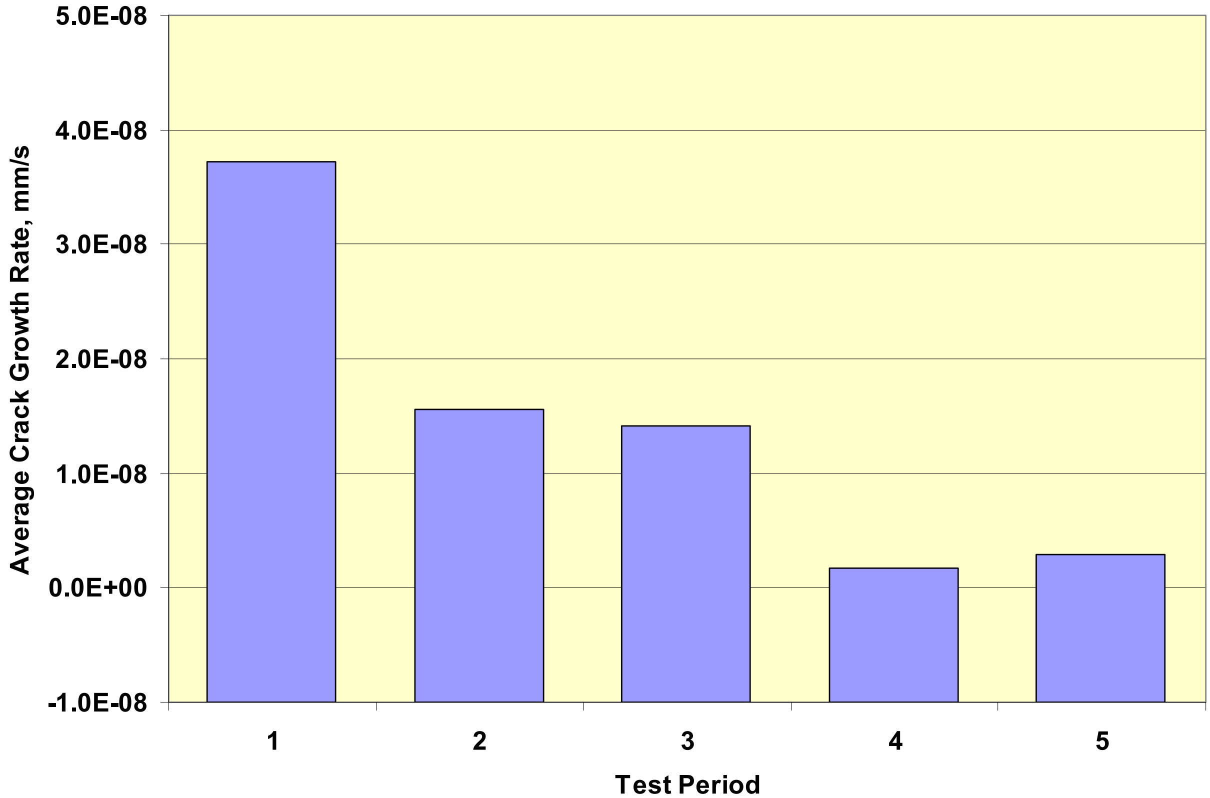


Figure 9. Crack growth rates for test periods in which Specimen 4-4-HAZ 1 was exposed to SFGE. The periods indicate segments of time in which the specimen was exposed to SFGE. They vary in length from 25 to 84 days; see Table 4.

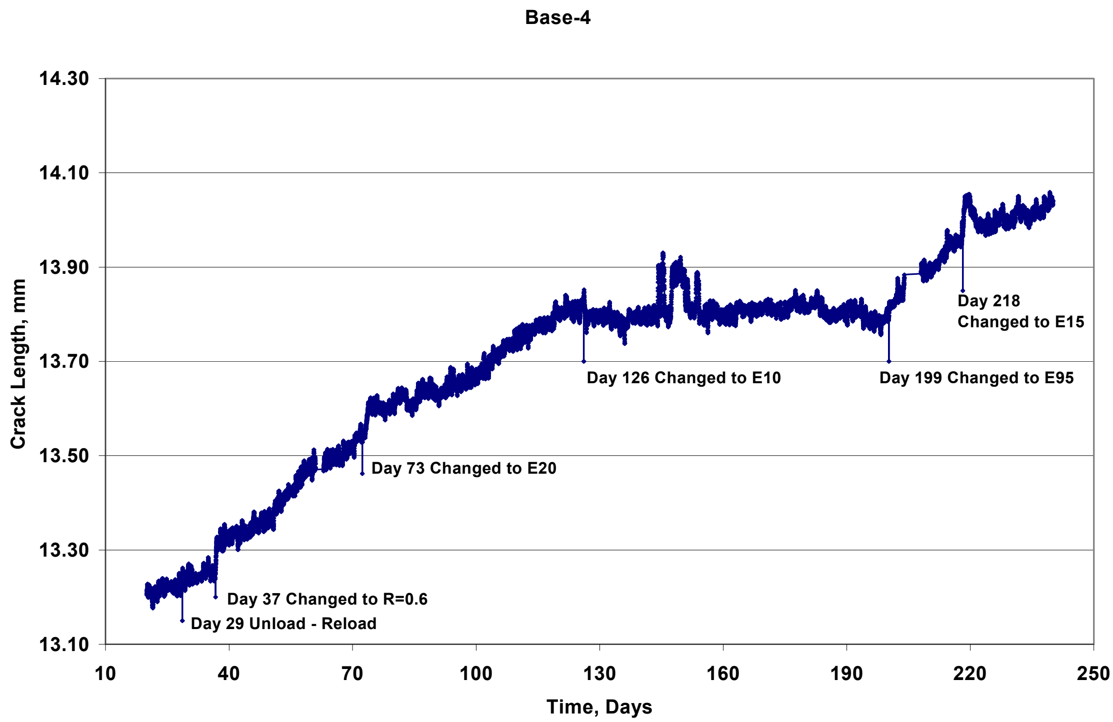


Figure 10. Crack length as a function of time for Specimen 4-4 Base 4 (0 to 240 hours).

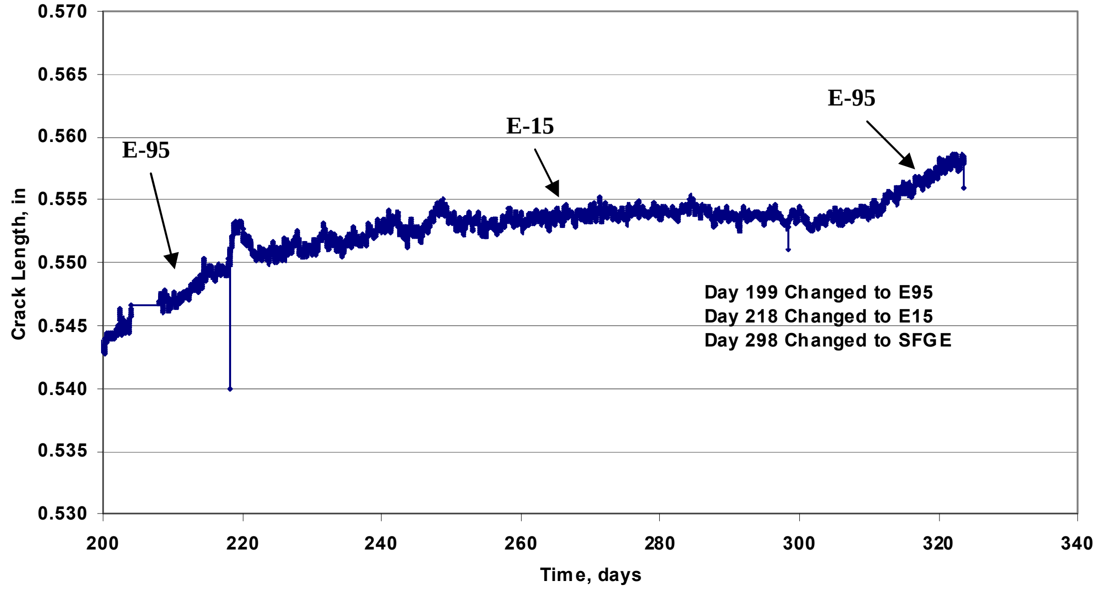


Figure 11. Crack length as a function of time for Specimen 4-4 Base 4 (200 to 325 hours).

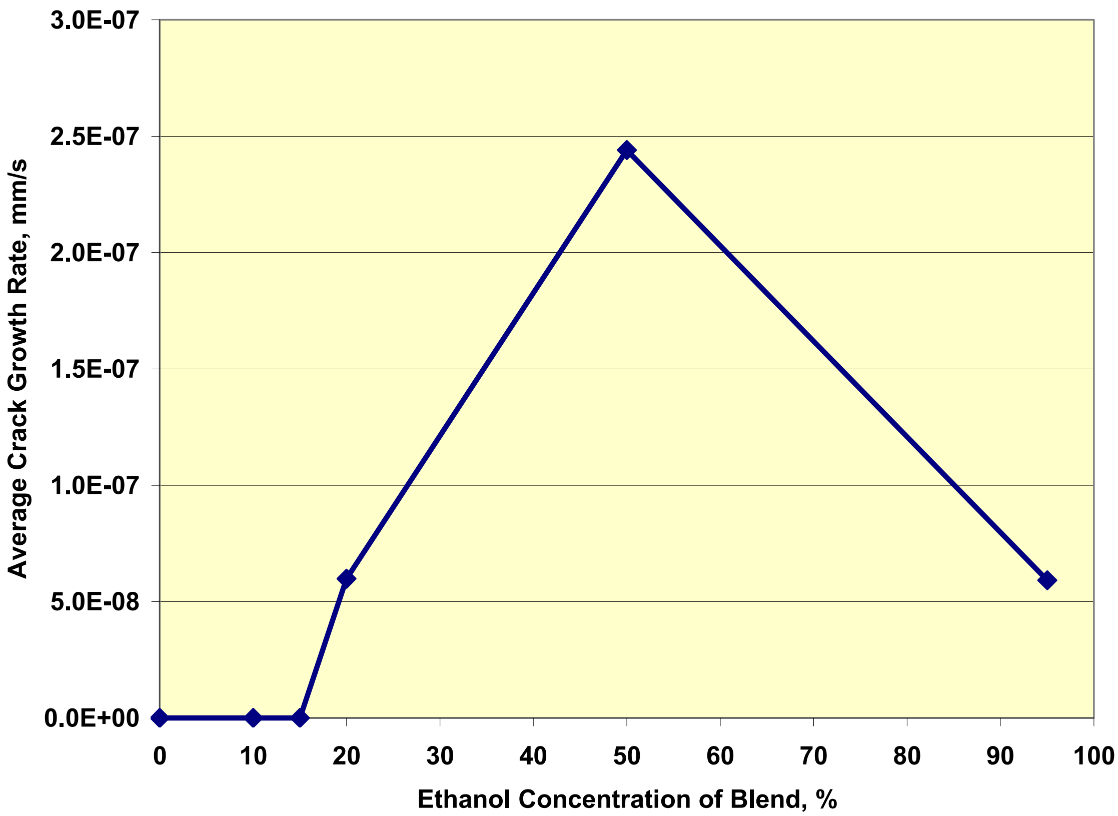


Figure 12. Average crack growth rate as a function of percent ethanol in the blend.

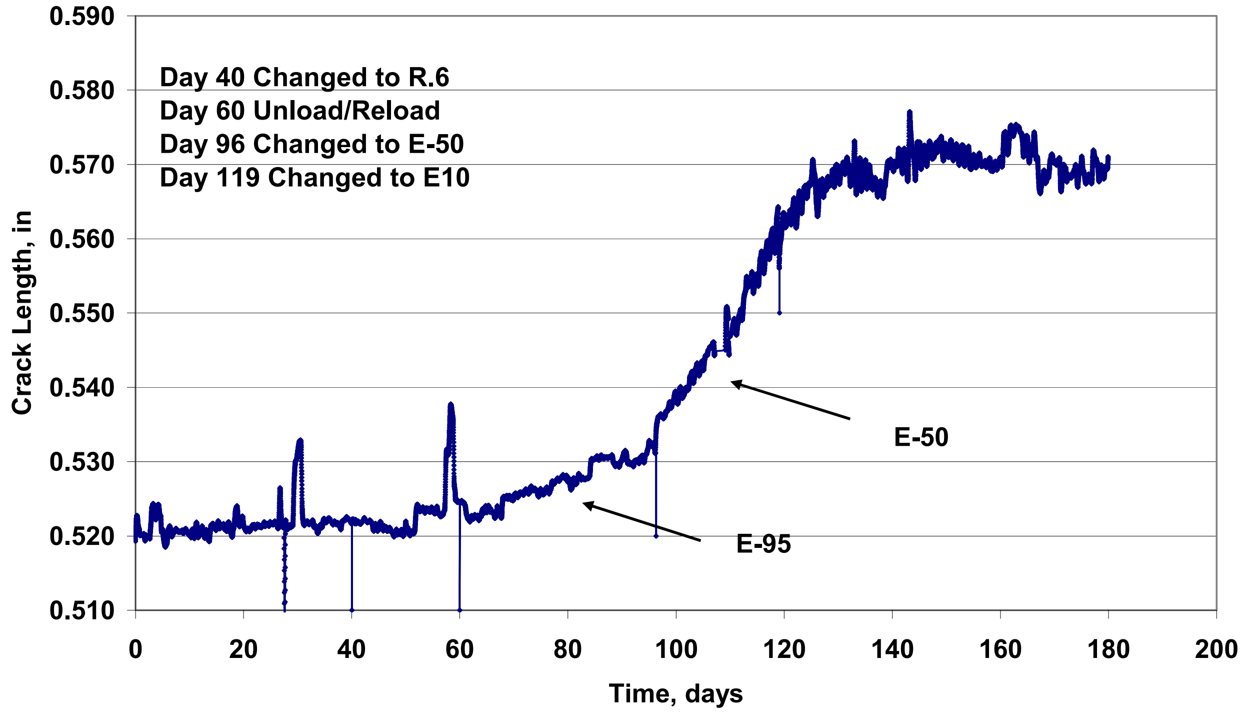


Figure 13. Crack length as a function of time for Specimen 4-4 Base 2 (0 to 190 hours).

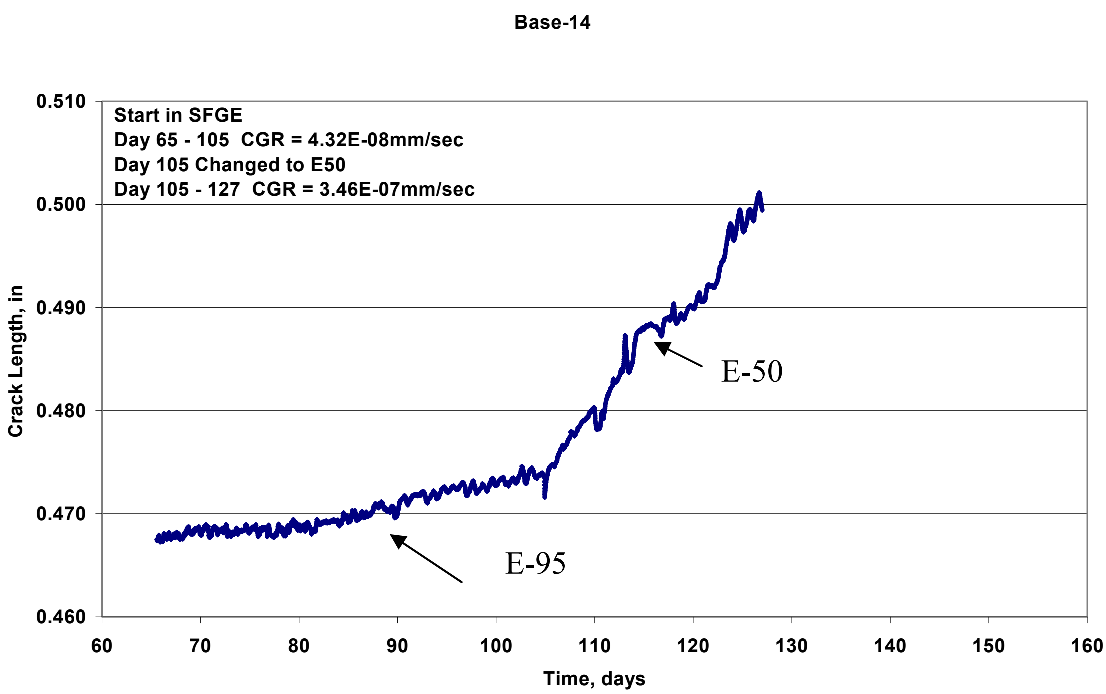


Figure 14. Crack Length as a function of time for Specimen 4-4 Base 14 (65 to 130 hours).

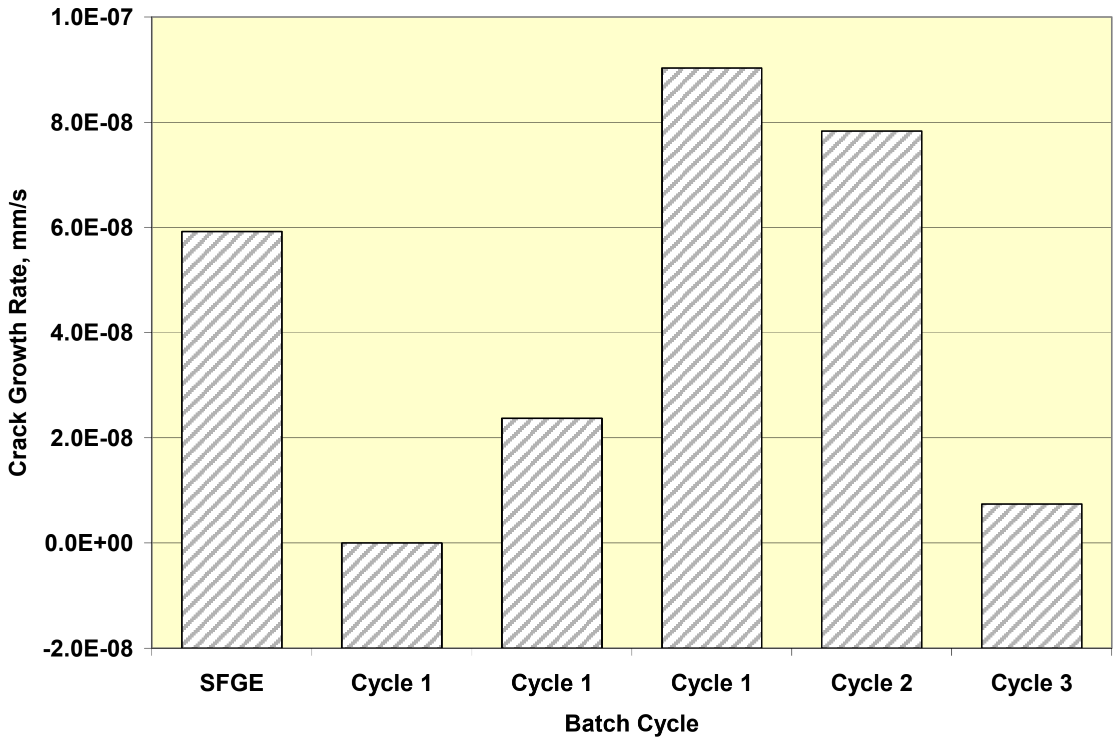


Figure 15. Time average crack-growth rate for various batch cycles. Value for SFGE (no batching) is an average for a number of observations.

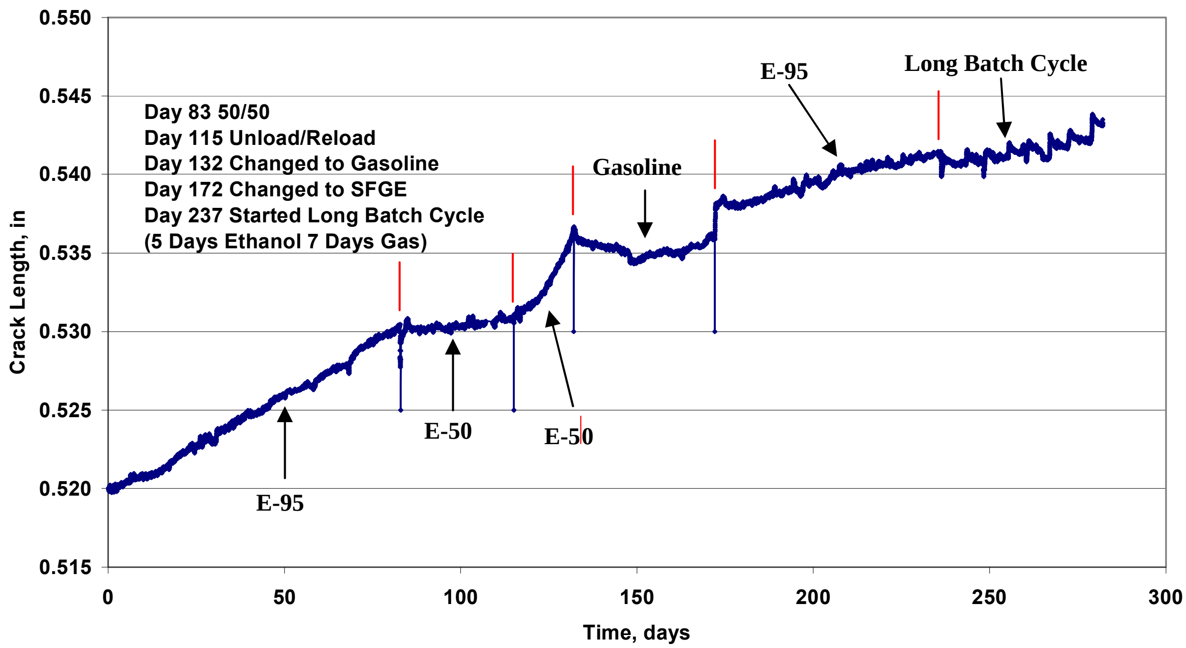


Figure 16. Crack length as a function of time for Specimen 4-4 HAZ 1. Long batch cycle (5 days SFGE – 7 Days Gasoline) started on Day 237.

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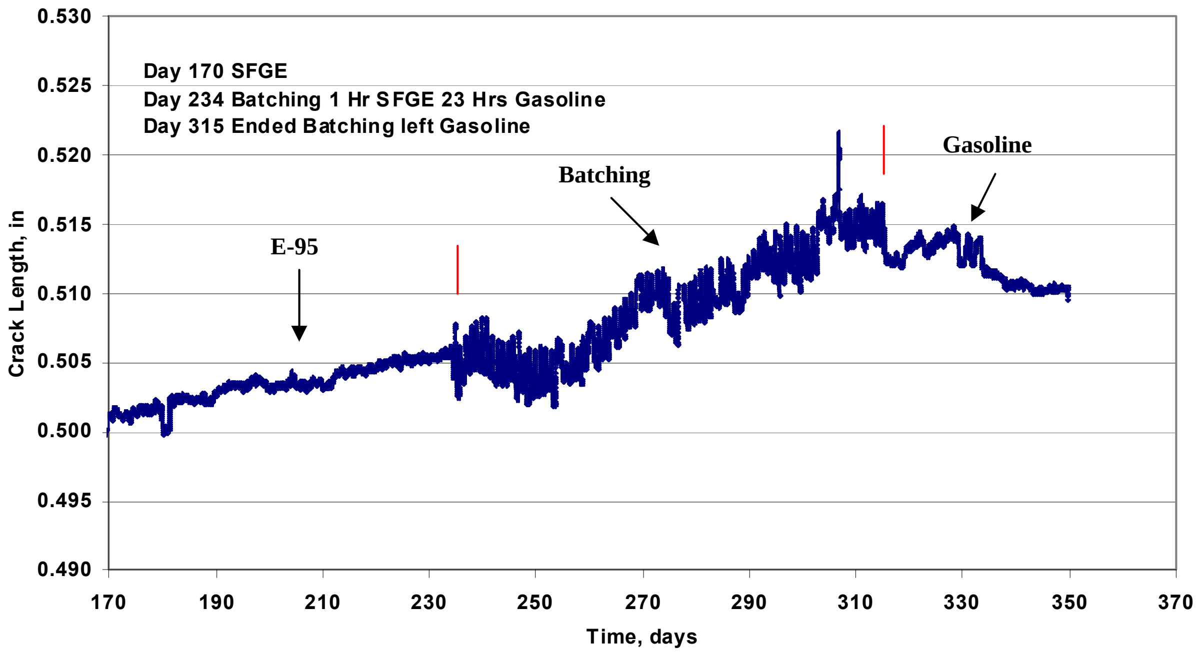

Figure 17. Crack length as a function of time for Specimen 4-4 Base 7. Short batch cycle (1 hour SFGE – 23 hours Gasoline) started on Day 234.

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