

Identification, Repair, and Mitigation of Cracking of Steel Equipment in Fuel Ethanol Service

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Identification, Repair, and Mitigation of Cracking of Steel Equipment in Fuel Ethanol Service

1 Scope

Usage of fuel ethanol as an oxygenate additive in gasoline blends is increasing both in the United States and internationally. This document discusses stress corrosion cracking (SCC) of carbon steel tanks, piping, and equipment exposed to fuel ethanol as a consequence of being in the distribution system, at ethanol distribution facilities, or end user (EU) facilities where the fuel ethanol is eventually added to gasoline. Such equipment includes but is not limited to:

- storage tanks,
- piping and related handling equipment, and
- pipelines

that are used in distribution, handling, storage, and blending of fuel ethanol. However, data for pipelines in ethanol service is limited and caution should be used when applying guidelines from this document that have been derived mainly from applications involving piping and tanks in ethanol storage and blending facilities. SCC of other metals and alloys is beyond the scope of this document, as is the corrosion of steel in this service.

It is realized that SCC of steel in fuel ethanol is a topic where knowledge of the subject is actively growing through documentation of recent experience and through research in progress. This document deals with handling of cracks in existing equipment and reduction of SCC occurrence in new equipment as a result of exposure to fuel ethanol per ASTM D4806 (or other international specifications), ethanol fuel blends, and pipeline transmixes involving fuel ethanol and conventional hydrocarbon fuels (gasoline, diesel, or jet fuel). It includes guidelines for carbon steel construction materials, including their fabrication, inspection, and repair to help assure safe and reliable operations.

This document is based on current engineering practices and insights from recent industrial experience and research. Older equipment may not conform exactly to the information contained herein, but this does not imply that such equipment is being operated in an unsafe or unreliable manner. It is also recognized that facilities may vary and may need to be modified depending on specific operating conditions, inspection, and maintenance experience. Each user company is ultimately responsible for its own safe and reliable operations.

The steels referred to in this document are defined by the ASTM or API designation systems or equivalent steel grades contained in other recognized codes or standards. Welded construction is considered the primary method of fabrication in equipment exposed to fuel ethanol.

Terminology used herein is given in Section 3.

2 Normative References

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

API Specification 5L, *Specification for Line Pipe*

API Publication 327, *Aboveground Storage Tank Standards: A Tutorial*

API 570, *Piping Inspection Code: Inspection, Repair, Alteration, and Rerating of In-service Piping Systems*

API Recommended Practice 574, *Inspection Practices for Piping System Components*

API Standard 579-1/ASME FFS-1, *Fitness-for-Service*

API Recommended Practice 580, *Risk-Based Inspection*

API Recommended Practice 582, *Recommended Practice and Welding Guidelines for the Chemical, Oil, and Gas Industries*

API Standard 620, *Design and Construction of Large, Welded, Low-pressure Storage Tanks*

API Standard 650, *Welded Tanks for Oil Storage*

API Standard 652, *Lining of Aboveground Petroleum Storage Tank Bottoms*

API Standard 653, *Tank Inspection, Repair, Alteration and Reconstruction*

API Publication 939-D, *Stress Corrosion Cracking of Carbon Steel in Fuel Grade Ethanol—Review, Experience Survey, Field Monitoring, and Laboratory Testing*

API Standard 1160, *Managing System Integrity for Hazardous Liquid Pipelines*

API Recommended Practice 1626, *Storing and Handling Ethanol and Gasoline-ethanol Blends at Distribution Terminals and Filling Stations*

API Standard 2015, *Requirements for Safe Entry and Cleaning of Petroleum Storage Tanks*

API Standard 2016, *Guidelines and Procedures for Entering and Cleaning Petroleum Storage Tanks*

API Standard 2217A, *Guidelines for Work in Inert Confined Spaces in the Petroleum and Petrochemical Industries*

API Standard 2610, *Design, Construction, Operation, Maintenance and Inspection of Terminal and Tank Facilities*

API Publication 4261, *Alcohols and Ethers: A Technical Assessment of Their Application as Fuels and Fuel Components*

ASME B31.4 ¹, *Pipeline Transportation Systems for Liquid Hydrocarbons and Other Liquids*

ASTM D4806 ², *Standard Specification for Denatured Fuel Ethanol for Blending with Gasolines for Use as Automotive Spark-Ignition Engine Fuel*

ASTM D6423, *Standard Test Method for Determination of pH_e of Ethanol, Denatured Fuel Ethanol, and Fuel Ethanol*

NACE TM0111 ³, *Slow Strain Rate Test Method for Evaluation of Ethanol Stress Corrosion Cracking in Carbon Steels*

NFPA 326 ⁴, *Standard for the Safeguarding of Tanks and Containers for Entry, Cleaning, or Repair*

RFA Publication 960501 ⁵, *Fuel Ethanol, Industry Guidelines, Specifications and Procedures*

STI SP001 ⁶, *Standard for the Inspection of Aboveground Storage Tanks*

U.S. DOE ⁷, (Alternative Fuels Data Center) *Handbook for Handling, Storage and Dispensing E85*

¹ ASME International, Two Park Avenue, New York, New York 10016-5990, www.asme.org.

² ASTM International, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428, www.astm.org.

³ NACE International (formerly the National Association of Corrosion Engineers), 1440 South Creek Drive, Houston, Texas 77084-4906, www.nace.org.

⁴ National Fire Protection Association, 1 Batterymarch Park, Quincy, Massachusetts 02169-7471, www.nfpa.org.

⁵ Renewable Fuels Association, 425 Third Street, Suite 150, Washington, DC 20024, www.ethanolrfa.org.

⁶ Steel Tank Institute, 944 Donata Court, Lake Zurich, Illinois 6004, www.steeltank.com.

⁷ U.S. Department of Energy, 1000 Independence Avenue, SW, Washington, DC 20585, energy.gov.



3 Terms, Definitions, Acronyms, and Abbreviations

3.1 Terms and Definitions

For the purposes of this document, the following definitions apply.

3.1.1

coating

Any thin material, liquid or powder, which applied over a structure, forms a continuous film to protect against corrosion.

3.1.2

cold working

Plastic deformation of a metal under conditions of temperature and strain that induce strain hardening and residual stress, usually, but not necessarily conducted at ambient temperature.

3.1.3

corrosion inhibitor

A chemical formulation that is added to an environment to reduce the severity of corrosion.

3.1.4

denature

The process of adding a substance (i.e. denaturant) for the purpose of making it unfit for human consumption.

NOTE In the case of fuel ethanol, natural gasoline is intentionally added. See Table 1 for limits of denaturant addition to fuel ethanol per ASTM D4806.

3.1.5

ethanol-containing fuels

In the United States, conventional gasoline is blended with up to 10 % by volume fuel ethanol (referred to as E10). It is also blended with 51 % to 83 % ethanol, depending on geography and season. This blend is referred to as E85.

3.1.6

fuel ethanol

fuel grade ethanol

FGE

Nominally anhydrous (<1 % volume water) denatured ethanol intended to be blended with natural gasoline at 1 % to 10 % volume for use as a spark ignition automotive engine fuel. In regions outside the United States, fuel ethanol may be available with lower water (anhydrous ethanol) or higher water content, up to 4.9 %v (6.1 %m) water, which is referred to as "hydrous ethanol."

NOTE Fuel ethanol is typically produced from organic sources such as corn, grain, grapes, sugarcane, and other forms of starch or sugar-based feedstocks.

3.1.7

intergranular cracking

Cracking produced in a susceptible metal that propagates along the grain boundaries within the material.

3.1.8

mixed-mode cracking

A term related to fracture by SCC whereby it has features of both intergranular cracking and transgranular cracking.

3.1.9

normalizing

Heating a ferrous metal to a suitable temperature above its transition temperature to form austenite, holding for a suitable time, and then cooling in still air (or atmosphere) to a temperature below the transition temperature.

3.1.10**plastic deformation**

Permanent deformation produced by stressing beyond the elastic limit of the material into the plastic region, creating a permanent set in the material.

3.1.11**postweld heat treatment****PWHT**

Any thermal treatment performed to a weldment following the process of welding, usually with the intent of reducing residual stresses or reducing hardness in the weldment.

3.1.12**residual stress**

The stress that exists in a metal or alloy that arises from either plastic deformation, welding, and/or thermal expansion.

3.1.13**stress corrosion cracking****SCC**

Environmental cracking in a susceptible metal produced by the simultaneous application of a tensile stress and exposure to particular corrosive environments.

3.1.14**stress relieving**

A form of PWHT involving heating a metal with the specific intent to reduce residual stress and then cooling slowly enough to minimize the development of new stresses.

3.1.15**tempering**

A form of heat treatment or PWHT involving heating for the purpose of decreasing the hardness of weldments and the strength of a material while generally increasing the toughness.

3.1.16**transgranular cracking**

Cracking produced in a metal that propagates across the grains within the material.

3.1.17**transmix**

Also known as “interface,” the product removed from a pipeline system between two separate product batches (e.g. fuel ethanol and gasoline) that, in some cases, cannot be blended with the first or second product.

3.1.18**weldment**

The portion of a component on which welding has been performed, including the weld metal, the heat-affected zone (HAZ), and the adjacent parent metal.

3.2 Acronyms and Abbreviations

ACFM	alternating current field measurement
DFMT	dry fluorescent magnetic testing
EC	eddy current
EFT	external floating roof tank
EU	end user
FGE	fuel grade ethanol

HAZ	heat-affected zone
HIC	hydrogen induced cracking
IFT	internal floating roof tank
IG	intergranular
ILI	in-line inspection
MIC	microbiologically influenced corrosion
MTBE	methyl tert-butyl ether
NDE	nondestructive examination
NDT	nondestructive testing
PA	phased array
PRCI	Pipeline Research Council International
PT	penetrant testing
PTBE	pounds per thousand barrels of ethanol
PWHT	postweld heat treatment
RT	radiographic testing
SCC	stress corrosion cracking
SRB	sulfate reducing bacteria
TMCP	thermomechanically controlled processing
TOFD	time of flight diffraction
U.S. DOT	U.S. Department of Transportation
UT	ultrasonic testing
WFMT	wet fluorescent magnetic particle testing

4 Background on SCC in Fuel Ethanol

4.1 Description of Fuel Ethanol

Ethanol as a fuel additive (i.e. fuel ethanol) has been used in the United States for more than 25 years. In the early 1990s, the U.S. Congress passed the Clean Air Act that required that oxygenate be added to gasoline in specific regions of the country. Commonly used oxygenates include ethanol and methyl tert-butyl ether (MTBE). Recently, MTBE has been found to contaminate groundwater and many states have banned its use. Additionally, the federal government has passed energy legislation (Energy Policy Act of 2005) that among other things, phases out the use of MTBE, promotes the use of ethanol in gasoline, eliminates some oxygenate requirements, and phases in increasing requirements for the use of renewable fuels like ethanol and other formulations. Consequently, the use of fuel ethanol as an additive and/or extender to gasoline is increasing. Additional considerations for use and distribution of fuel ethanol and alcohol fuels are available in API 1626 and API 4261.

Modern production of ethanol from starch or sugar-based feedstocks (grain, fruit, sugarcane, and other biomass) is similar to processes that have been used for centuries, but which have been considerably refined in recent years. There are two production processes for grains: wet milling and dry milling. The main difference between the two is in the initial treatment of the grain. In the final process, the starch and any remaining water from the mash is converted to sugars that can then be fermented to produce ethanol that is recovered by distillation. Once produced and ready for distribution, the ethanol is denatured (typically by the addition of unleaded gasoline) and a corrosion inhibitor is added.

4.2 Relevant Standards for Fuel Ethanol

In the United States, fuel ethanol is currently governed by ASTM D4806, which gives the compositional and physical limits for fuel ethanol. These are shown in Table 1.

Table 1—Quality Specification for Fuel Ethanol per ASTM D4806

Property	Units	Specification	ASTM Designation
Ethanol	%v min	92.1	D5501
Methanol	%v max	0.5	—
Solvent-washed gum	mg/100 mL max	5.0	D381
Water content	%v max	1.0	E203
Denaturant content	%v min %v max	1.96 5.00	D4806
Inorganic chloride content	ppm (mg/L) max	10 (8)	E512
Copper content	mg/kg max	0.1	D1688
Acidity as acetic acid	%m (mg/L)	0.007 (56)	D1613
pHe	—	6.5 to 9.0	D6423
Appearance	Visibly free of suspended or precipitated contaminants (e.g. clear and bright)		
NOTE The limit for inorganic chlorides was reduced in ASTM D4806 (2009) from a higher level of 40 ppm in the previous edition of this standard.			

While fuel ethanol in the United States is made in conformance with this standard, most of its properties are also defined by other ASTM designated standards (see Table 1). Additional information can be found in RFA Publ 960501. The purpose of the ASTM D4806 standard is to provide parameters so ethanol/gasoline blends will perform satisfactorily in a wide range of fuels used in consumer vehicles and not for any attributes regarding corrosion or SCC that the fuel ethanol might have in the supply chain prior to blending with gasoline. Fuel ethanol may also have properties not specified in ASTM D4806 but that may influence susceptibility to SCC. Some of these properties are discussed in 4.5.

A parameter that is commonly used in evaluation of fuel ethanol is the pHe as defined by ASTM D6423. The pHe value is a measure of the acid strength of high ethanol content fuels and their constituents and is applicable to fuels containing nominally 70 % volume ethanol or higher. This parameter is similar in some regards to the pH parameter used in aqueous solutions. An important difference is that neutrality in pHe units is near 9.5 as opposed to the familiar 7.0 pH that defines neutrality in aqueous solutions under ambient conditions. The pHe value may not directly correlate to the pH scale for aqueous solutions. Most importantly, pHe does not appear to correlate with the corrosion or SCC behavior of fuel ethanol in the supply chain prior to blending with gasoline.

Fuel ethanol is not sold with zero water content, where it would be referred to as anhydrous ethanol. Denatured fuel ethanol made to ASTM D4806 specifications typically contains up to 1 % water and other constituents as mentioned in the ASTM D4806 standard. Denatured ethanol with less than 0.5 % water is considered “anhydrous ethanol.” Ethanol with higher water content up to 4.9 %v (6.1 %m) is usually referred to as “hydrated ethanol.” Such hydrated ethanol is uncommon in the United States but has been used as a fuel in Brazil. Brazil also makes anhydrous ethanol used for blending into gasoline. Both anhydrous and hydrated grades of fuel ethanol made in Brazil are made from primarily sugarcane and the amount of each produced fluctuates with the price. Recent accounts indicate that the Brazilian market for fuel ethanol is dominated by anhydrous ethanol. There has been no documented experience with SCC in hydrated or anhydrous ethanol derived primarily from sugarcane despite over 20 years of research and their use as automotive fuel in Brazil.

4.3 Supply Chain for Fuel Ethanol

Once fuel ethanol is produced at a manufacturer's facility, it is held in storage tanks until it is released for distribution. Manufacturers generally add the denaturant prior to or during on-site storage, and an inhibitor is added during storage or just prior to release of the shipment for distribution. This may be one reason for the apparent difference in SCC experience between manufacturer and downstream facilities (i.e. no reported failures at manufacturer facilities, and SCC at some downstream facilities); see 4.5.7 for more information on the effect of inhibitors. Once the fuel ethanol

enters the distribution system, it can be transported by many methods. Transportation methods include barge, railroad tanker car, tanker truck, and pipeline.

The period that fuel ethanol is in the chain can vary considerably from days to months depending on the location of the manufacturing facility, the availability of intermediate distribution storage, the location of gasoline blending terminals, and the transportation mode used. Once fuel ethanol enters intermediate liquids storage and distribution terminal or a gasoline blending facility, the ethanol is held in storage tanks. The residence time in these tanks likewise varies depending on usage and traffic requirements. In some cases, it can be held for months during a period of inactivity. In some cases at gasoline blending facilities, however, the residence time in the storage tank is very short as incoming ethanol deliveries and outgoing shipments of blended gasoline are a near continual process. Observations of SCC have been limited to the portion of the supply chain involving the intermediate liquids storage through the gasoline blending facility and may be related to changes that occur in the fuel ethanol or conditions that develop in the distribution system.

4.4 Significance of SCC in Fuel Ethanol

4.4.1 General

A review of service experience with fuel ethanol and the literature for cited cases of SCC ethanolic environments was conducted by API and is referenced in API 939-D (see references). Highlights from this work have also been published in the open literature. This study revealed that there has been SCC of carbon steel exposed to ethanolic environments and that the range of conditions causing SCC has gained in understanding as highlighted in references found in the recent updated version of API 939-D that includes work funded by the Pipeline Research Council International (PRCI) and United States Department of Transportation (U.S. DOT). The published literature and service experience with methanol has shown for some time that methanol is a known SCC agent for steel. Water content, various impurities, temperature, and metallurgical condition of the steel determine methanol SCC susceptibility.

A follow-up research program was conducted by API in cooperation with the RFA to identify the variables that control SCC of steel in fuel ethanol. This in-depth study involved:

- a) gathering of further service experience;
- b) field corrosion and SCC monitoring; and
- c) laboratory research on environmental and metallurgical variables pertaining to steels commonly used in tanks, pipes, and pipelines handling fuel ethanol.

The work product of the API and API/RFA studies provided the basis for this standard. The detailed results of these studies and related information are also found in API 939-D.

Several documented cases of SCC in steel equipment exposed to fuel ethanol obtained from the aforementioned study are presented in Annex A. These findings indicate that documented failures of ethanol process equipment have dated back to at least the early 1990s. Additionally, this work indicated that SCC failures continue to be observed in various parts of the distribution system for ethanol, albeit being reported less frequently in recent surveys since the guidelines contained herein have been published. A form for reporting additional failure incidents resulting from SCC in fuel ethanol to API is available in Annex B. Companies experiencing what they think are cases of SCC in fuel ethanol (or ethanol fuel blends) are encouraged to confirm these cases through review and documentation of service conditions, as well as metallurgical analysis of the cracked or failed components that includes metallographic or fractographic examination of the cracks to confirm similarity to known cases of SCC given herein and to provide a summary of this information to API using the form provided in Annex B. For more information on the identification of SCC in fuel ethanol, see 4.4.2, and the examples in Annex C. Information on ethanol SCC failures or leaks provided in this form will be utilized to further this document with the name of the submitting company not associated with the information. Such information is of benefit in confirming the extent of the ethanol SCC problem and in future updates to this document.

4.4.2 Identification of SCC in Fuel Ethanol

SCC cracks of steel in fuel ethanol have a similar appearance to the cracks caused by other cracking environments. Examples of SCC in steel equipment exposed to fuel ethanol are shown in Annex C. The cracks are typically branched and may be intergranular, transgranular, or mixed mode. In laboratory testing, both intergranular and transgranular cracking may occur depending on the composition of the ethanol and testing methods used. Most cracks documented from field failures show predominately intergranular cracking. When analyzing a field failure in fuel ethanol service, internal surface originating intergranular cracking suggests ethanol SCC, but transgranular or mixed-mode cracking may also exist. Cracks generally are tight and often filled in corrosion product, making visual detection on the surface difficult in some cases and thus requiring alternative methods as discussed herein.

4.4.3 Cited Example Cases of SCC

Cases of SCC of steel components in fuel ethanol have been reported in the following types of equipment in fuel ethanol distribution and gasoline blending facilities:

- a) welds and adjacent metal in tank bottoms, floating roofs, and associated seal components (e.g. seal springs);
- b) facility rack piping, fittings, and associated equipment (e.g. air eliminators);
- c) vertical seam and nozzle welds in lower tank shells located off bottom; and
- d) pipeline used to transport fuel ethanol from terminal to EU facility.

A total of 34 cases of SCC have been reported in the API survey efforts conducted from 2003 through 2011 and include cases of SCC that go back through the early 1990s. Many of these involved multiple occurrences of cracking at the same location over time or instances of SCC in multiple equipment types at the same facility at the same time. All but one case of ethanol SCC was in equipment exposed to fuel grade ethanol (FGE). The one case not in FGE occurred in a small shop-built tank used to hold an E85 ethanol fuel blend. It was complicated by the presence of pitting that occurred while in prior waste oil service and a high degree of plastic deformation and residual stress occurring during manufacturing. Time to failure by ethanol SCC has been observed to range from 3 months to over 10 years from the date when the equipment was placed in FGE or ethanol fuel blend service.

A listing of the documented instances of SCC from the API surveys and the percentages they have occurred in each type of application are given below:

- tank floor plates seam welds—seven cases (20.6 %),
- tank floor/sidewall fillet welds—three cases (8.8 %),
- tank sidewall first course butt weld—three cases (8.8 %),
- tank floating-roof seam welds—three cases (8.8 %),
- tank nozzle—one case (2.9 %),
- tank roof springs—two cases (5.9 %),
- shop-built tank in E85—one case (2.9 %),
- facilities piping/fittings—eight cases (23.5 %),
- facilities piping/supports—two cases (5.9 %),

- pipeline—two cases (5.9 %),
- ancillary equipment—two cases (5.9 %).

Examples of many of these types of failures are provided in Annex C.

It is significant to note that thus far, all but one failure was in fuel ethanol and that no SCC failures have been documented in the following cases:

- a) ethanol manufacturer facilities (storage tanks, piping, or associated equipment);
- b) intermediate supply chain equipment, barges, tanker cars, or tank trucks; and
- c) blending or transportation facilities handling products containing fuel ethanol after it has been fully blended with conventional grade gasoline (E10).

The reasons for this latter behavior may be related to reduced susceptibility to SCC in these portions of the supply chain, but it could also be related to a lack of reporting. A form for reporting failure incidents resulting from SCC in fuel ethanol to API is available in Annex B.

4.4.4 Experience with SCC in Fuel Ethanol

A survey of five major EUs in North America determined that approximately 100 tanks were in ethanol service (of which 75 were currently coated). It was reported that the average age of the tanks was approximately three years and represented 120 tank years in fuel ethanol service. The number of reported SCC events was 19, with some including multiple occurrences per event that increases the number to approximately 24. More information on reported failure occurrences through February 2005 is available in Annex A. Additional information is available in the recent update to API 939-D.

In the 2011 API survey, a canvass of the NACE International pipeline and refining committees indicated the following new information on recent experience with ethanol SCC from the following sectors.

- Ethanol SCC incidents:
 - 13 % failure, 37 % leak, 25 % cracked, 50 % nonfailure (crack found before leakage), 0 % severe corrosion or polymeric material incompatibility (some reported more than one category);
 - 50 % reported conducting an investigation to document ethanol SCC cracks, leaks, or failures (balance had no investigation).
- Equipment:
 - 0 % tank (bottom, side, roof), 17 % facilities piping, 33 % pipe fittings, 50 % other components and equipment.
- Regions:
 - United States and Europe.
- Time to SCC incident once in ethanol service:
 - 40 % less than 1 year, 20 % 1 to 3 years, 20 % 3 to 10 years.
- Time equipment in prior (nonethanol) service:
 - 60 % none, 40 % 10 to 12 years.