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Investigation Of Corrosion-Influencing Factors In Underground Storage Tanks With Diesel Service

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Abbreviations And Acronyms

ALS	ALS Environmental
ANOVA	analysis of variance
API	American Petroleum Institute
ASTM	ASTM International
ATG	automatic tank gauge
ATR	attenuated total reflectance
CDFA	Clean Diesel Fuel Alliance
CRC	Coordinating Research Council
DNA	deoxyribonucleic acid
EGT	emergency generator tank
EPA	Environmental Protection Agency
FAME	fatty acid methyl esters
FID	flame ionization detector
FTIR	Fourier transform infrared
GC-MS	gas chromatography-mass spectrometry
IC	ion chromatography
ID	identification
L	liter
LOQ	limit of quantitation
LSD	low sulfur diesel
mg/kg	milligrams per kilogram
mg KOH/g	milligrams potassium hydroxide per gram
mg/L	milligrams per liter
MIC	microbiologically-influenced corrosion
mm Hg	millimeter of mercury
MRL	method reporting limit
mS/cm	millisiemens per centimeter
NACE	National Association of Corrosion Engineers International
NAVFAC	Naval Facilities Engineering Command
NIOSH	National Institute for Occupational Safety and Health
OOB	out of bag
ORD	Office of Research and Development
OUST	Office of Underground Storage Tanks
PEI	Petroleum Equipment Institute

PLS	partial least square
POM	polyoxymethylene
ppbv	parts per billion by volume
ppm	part per million
pS/m	picosiemens per meter
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
RFS	Renewable Fuel Standard
STP	submersible turbine pump
STREAMS	Scientific, Technical, Research, Engineering and Modeling Support
SW	EPA Test Methods for Evaluating Solid Waste
TAN	total acid number
ULSD	ultra-low sulfur diesel
US	United States
UST	underground storage tank

Executive Summary
Investigation Of Corrosion-Influencing Factors In Underground Storage Tanks With Diesel Service

This research focused on better understanding a type of rapid and severe corrosion of metal components in underground storage tanks (USTs) storing diesel fuel. UST owners first began reporting this corrosion to UST industry servicing companies in 2007. Several changes to the national fuel supply and fuel storage practices have occurred since the mid-2000s. To address the potential for corrosion problems, the U.S. Environmental Protection Agency's (EPA) Office of Underground Storage Tanks began working on this research in 2014 to understand how serious and widespread the metal corrosion problem could be. In addition, to help identify the cause or solutions, we wanted to identify predictive factors between UST systems with corrosion issues and UST systems relatively free of the problem. EPA's objective for the research was to develop a better understanding of potential risks to human health and the environment caused by the evolving corrosion problem in USTs storing diesel fuel.

In 2014, EPA held discussions with UST industry experts and worked collaboratively to develop field-based research that would further the understanding of corrosion inside USTs storing diesel. EPA designed our research to examine many factors on a diverse population of 42 UST systems in order to find potential predictive factors among them. We thought any predictive factors identified in our research would help focus the search of potential causes for the next phase of follow-on research.

In January and February 2015, EPA conducted on-site inspections of 42 diverse, operating UST systems at 40 sites across the country. Of these UST systems, 24 had fiberglass tanks, and 18 had steel tanks. Field teams documented the conditions of the UST systems with in-tank video cameras and photos so they could later assign a category of corrosion coverage to each system. The field teams also collected samples of vapor, fuel, and aqueous phase (also known as water bottom), if present, from each of the tanks. Field teams used a detailed questionnaire to gather information from each owner about the storage history, operation, and maintenance practices of each UST system.

EPA chemically analyzed the vapor, fuel, and aqueous phase samples. Three assessors reviewed the videos of each UST and categorized the USTs by the extent of the corrosion judged to be present: minimal, moderate, or severe. In an attempt to identify corrosion predicting factors among UST systems experiencing either minimal corrosion or severe corrosion, we then statistically evaluated the analytical results and responses from the questionnaires against the corrosion categories.

The major finding from our research is that moderate or severe corrosion on metal components in UST systems storing diesel fuel in the United States could be a very common occurrence. Observations suggest that corrosion may be commonly severe on metal surfaces in the upper vapor space of UST systems, an area that before 2007 was not known to be prone to corrosion. Furthermore, it appears many owners may not be aware of the corrosion nor are they aware that corrosion, which could affect the operability of their UST systems, could already be at

an advanced stage. We observed 83 percent of the inspected tanks had moderate or severe metal corrosion. Prior to our research inspections, less than 25 percent of owners reported knowledge of corrosion in their UST systems.

It appears from our research that corrosion inside of UST systems could result in an increased chance of releases of fuel to the environment and subsequent groundwater contamination. Across the sample population, EPA observed corrosion occurring on all types of UST system metal components, including submersible turbine pump shafts, automatic tank gauge probe shafts, risers, overfill equipment like flapper valves and ball valves, bungs around tank penetrations, inner walls of tanks, and fuel suction tubes. Many of these UST system components are designed to prevent overfilling the tank or to identify leaks, and the components must be able to move and function as designed. Corrosion of some metal components could hinder their proper operation and possibly allow a release of fuel to occur or continue unnoticed. Anecdotal reports since EPA began our research suggest that other metal components in UST systems, such as tank walls, could also eventually fail by corroding completely through the metal if corrosion is not stopped. This would most likely occur in the bottom of an UST where aqueous phase and tank sludge collect. Corrosion through the bottom or wall of a tank could potentially allow fuel to leak into secondary containment areas or release to the environment.

EPA has heard anecdotes of functionality failures of release prevention equipment and leak detectors, as well as failures of metal walls resulting in leaks into secondary containment areas. Outside of anecdotes, however, very little verifiable data exists about how equipment functionality and integrity are being affected by corrosion in USTs storing diesel fuel. However, that information should become more available as owners become more aware of the findings of our research and corrosion in USTs storing diesel becomes more visible.

Even absent a release of fuel to the environment, severe corrosion poses concerns for owners. Corrosion increases servicing and equipment maintenance costs for UST system owners. Anecdotes suggests that dispenser filters may become clogged with corrosion debris that resembles coffee grounds, resulting in filters needing to be changed more frequently. Other equipment may need to be repaired more often and sometimes may need to be prematurely replaced.

The data and analyses could not pinpoint a cause of corrosion that UST owners began reporting in 2007. It appears multiple underlying factors and corrosion mechanisms could be contributing to the corrosion; one such mechanism is microbiologically-influenced corrosion (MIC). Previous research on the recent corrosion phenomenon is limited, but suggests that the reduced sulfur in diesel could be allowing microbial life to proliferate in ultra-low sulfur diesel tanks and, through MIC, cause corrosive conditions that were less possible in USTs storing low sulfur diesel. Several independent organizations have produced publicly available resources that suggest following certain enhanced maintenance practices when storing diesel fuel in USTs. If followed, these practices can likely minimize MIC risks by reducing bacterial populations or preventing an environment where microbial life can thrive.

EPA's research builds on industry's first study about rapid and severe corrosion in USTs storing diesel, which the Clean Diesel Fuel Alliance (CDFA) completed in 2012.[1] The objective of CDFA's study was to produce an initial hypothesis about the mechanism of corrosion from data collected on six UST systems. EPA's Office of Research and Development (ORD) completed research shortly after CDFA.[2] Both the CDFA and ORD research hypothesized that biofuel components in diesel, such as ethanol and biodiesel, could be providing the energy source for microbial populations of bacteria like *Acetobacter* in USTs. This genus of bacteria was the most abundant in samples that underwent DNA sequencing in CDFA's study. EPA's research plan to identify any predictive factors by default included checking the plausibility of the hypotheses previously suggested. However, there are numerous other types of bacteria that could also be consuming chemical components of the fuel or fuel contaminants found in USTs. In addition to bacteria, there are also a number of other microorganisms that could cause or contribute to the corrosion attacks, including fungi, archaea, and eukaryotic organisms. A combination of one or more of these factors could also be responsible, but we did not test for those factors in our research.

Because only limited scientific research was available, EPA assumed from the beginning of our research that, within our research scope, it was not feasible to definitively pinpoint a cause of the corrosion. Further, most familiarity with the extent and geographic spread of the problem was anecdotal. Therefore, an exploratory approach was most helpful in expanding the knowledge around the issue. Our research suggests that MIC is likely involved in the moderate or severe internal corrosion in USTs storing diesel. However, further identification of specific bacteria was not possible within the scope of our research. Therefore, while previous research hypotheses about the role of specific species of a genus oxidizing biofuel components were not disproven by the results of our research, validation would be speculative.

EPA's research population of 42 USTs was geographically, materially, and operationally diverse and was the largest field research of this issue to date. However, the population is a small percentage of diesel USTs across the United States, and the types of USTs and maintenance practices by owners in the research population may differ from those in the national population of USTs storing diesel. Therefore, EPA cannot predict if the presence of moderate or severe corrosion in diesel USTs across the United States will be higher or lower than identified in our research.

EPA recommends owners check their diesel UST systems for corrosion and take steps to ensure the proper operability of their UST systems. EPA is recommending this because 83 percent of USTs in the study affected by moderate or severe corrosion is very high, most of the owners were not aware of the extent of the corrosion in their USTs, and it appears that corrosion could potentially affect equipment functionality and potentially lead to a release of fuel to the environment.

Our research provided us with key takeaways that, by increasing the knowledge around corrosion, may help prevent releases of diesel fuel from UST systems. Below we list our key takeaways; see Chapter 6 for additional discussion about them.

- Corrosion of metal components in UST systems storing diesel appears to be common.
- Many owners are likely not aware of corrosion in their diesel UST systems.

- The corrosion is geographically widespread, affects UST systems with steel tanks and with fiberglass tanks, and poses a risk to most internal metal components.
- Ethanol was present in 90 percent of 42 samples, suggesting that cross-contamination of diesel fuel with ethanol is likely the norm, not the exception.
- The quality of diesel fuel stored in USTs was mixed.
- Particulates and water content in the fuel were closest to being statistically significant predictive factors for metal corrosion, but causation cannot be discerned.
- MIC could be involved as hypothesized by previous research.
- EPA recommends owners visually inspect USTs storing diesel as part of routine monitoring.

Chapter 1
Introduction And Background

The U.S. Environmental Protection Agency (EPA) Office of Underground Storage Tanks is responsible for ensuring that, in order to prevent releases and avoid contaminating vital underground water supplies, fuels and hazardous substances are safely stored in underground storage tank systems (USTs). This report describes the process and results of EPA's research to understand the extent, severity, and potential impacts of severe and rapid corrosion occurring in USTs storing ultra-low sulfur diesel (ULSD), hereafter referred to as diesel, that has been reported across the country since soon after the fuel was introduced in 2006. Our research also attempted to identify predictive variables that could narrow the search of possible causes of and solutions for the corrosion of metal components in UST systems storing diesel fuel.

EPA is working proactively with industry to help solve the problem because some anecdotes about the severe or rapid corrosion reported compromised functionality and failure of some metal components of UST systems. If corrosion prevents UST system equipment from functioning properly, then UST systems may be at higher risk of operational failures, which could result in the release of fuel into the environment. EPA heard anecdotes about new submersible turbine pumps that were replaced due to corrosion in as few as six months after being installed. Reports of UST system components corroding with this combination of speed, severity, and location within the vapor spaces of USTs were not reported prior to 2007.

EPA worked with industry experts to design our field research, which built on the Clean Diesel Fuel Alliance's (CDFA) 2012 research findings. EPA contracted with Battelle to execute the work. We designed our research to be exploratory in nature, examining data from 42 operational USTs storing diesel. The goals of the research were twofold: to understand the extent, severity, and potential risks of the corrosion; and to identify any predictive factors between UST systems with severe corrosion issues and UST systems relatively free of the problem. We wanted to identify predictive factors so they could help narrow the scope of investigation for future research efforts. EPA appreciates the efforts of our contractor, industry partners and stakeholders, as well as volunteer UST owners, who helped make our research possible.

New And Historical Trends In Corrosion In USTs Storing Diesel

The first known report of severe corrosion on internal metal components in the vapor space of an UST system storing diesel was posted on the Petroleum Equipment Institute's (PEI) website in 2007.[3] This location of corrosion in USTs storing diesel fuels has since been commonly reported, with anecdotes often describing the corrosion as severe and sometimes occurring rapidly on new equipment.

Prior to 2007, corrosion risks identified in tanks storing diesel fuels were generally thought to be limited to the bottoms of tanks and were not observed in upper portions of tanks. The risk of corrosion in the bottoms was usually considered to be easily addressed with good UST system maintenance practices. The corrosion observed since 2007, however, may affect owners who have followed the same maintenance practices for years and previously had no corrosion issues. Reports of the corrosion of metal components in the upper portions of USTs indicate the functionality of metal components of UST systems can be compromised. EPA and the fueling industry do not yet understand the exact cause of the corrosion nor its full effects on equipment.

Unchecked corrosion in UST systems could result in a potentially higher risk of an environmental release and subsequent groundwater contamination by fuel. A release could potentially occur either from malfunctioning overfill prevention, fuel level monitoring, or leak detection equipment that cannot operate properly due to restricted movement or from a direct failure of integrity of metal components.

The shaft of the submersible turbine pump (STP) is the component most often observed as being severely corroded, but corrosion anecdotes often discuss corrosion on other metal components in UST systems as well. Reports range from severe corrosion in only a few small areas or on certain equipment to uniform coverage of metal components in the vapor space. Sometimes corrosion is reported or observed on metal surfaces in wetted portions of the UST system in areas normally submerged in fuel. The corrosion has also been sometimes described as layers of tubercles that coat the metal surfaces of equipment.

Even in the absence of a release, the metal corrosion observed in UST systems can be harmful to industry because corrosion creates increased expenses for UST system owners through more frequent servicing and filter changes, as well as likely shorter lifespans for equipment.

Early Efforts To Understand The Issue: 2010 Industry Survey

In 2010, PEI gathered more information around the corrosion anecdotes by conducting a five-question survey for owners, operators, field technicians, and equipment manufacturers. PEI's survey resulted in a list of UST system equipment that was reportedly affected by operational problems, accelerated corrosion, rust, gasket or seal problems, or premature failure since 2007.[4] Results from PEI's survey suggested that all metals in contact with the fuel or exposed to vapors inside the UST could potentially be negatively impacted. Table 1 lists the UST equipment or components most frequently reported in additional comments submitted to the survey as problematic, and how they were affected. Figure 1 shows a simplified diagram of a typical diesel UST system and identifies the equipment.

Proper functionality of all UST equipment is crucial to ensuring fuel can be stored safely in the UST system and preventing releases of fuel to the environment or quickly detecting releases should they occur. Components most commonly affected by the corrosion are inside the UST system, so operators may not easily or readily notice the problem.

Changes In Fuel And The Fueling Industry

As industry became more aware of the corrosion, momentum to search for the cause and a solution grew. The onset of corrosion reports in USTs storing diesel began around the time of several notable changes to fuel supplies in the United States that affect diesel fuel storage. A direct change to diesel fuel was introducing cleaner burning diesel to help reduce atmospheric pollution from diesel powered engines. Prior to 2006, diesel fuel was characterized as low sulfur diesel (LSD) and requirements limited fuel to a maximum of 500 parts per million (ppm) sulfur. Beginning in 2006 and phasing in over the next several years, EPA's standards required diesel fuel contain no more than 15 ppm sulfur; this was referred to as ultra-low sulfur diesel (ULSD). Lowering the sulfur in diesel necessitated additional modifications to the fuel, which sometimes included adding to diesel formulations lubricity additives, cold flow improvers, corrosion inhibitors, and conductivity additives.

Table 1. UST Equipment Reported Most Frequently Experiencing Operational Problems In Additional Comments Of 2010 Survey

Affected Equipment	Operational Malfunction
Dispenser fuel filters	Clogging and requiring more frequent replacement
Seals, gaskets, O-ring	Deterioration
STP and pump components	Replacement/column pipe wear/motor problems
Tanks	Rusting and leaking (includes tanks on vehicles)
Meters	Premature failure
Line leak detectors	Damaged or broken
Automatic nozzle shutoff	Failure/shorter lifespan
Tank probes	Malfunctioning
Check valves	Not seating
Shear valves	Not sealing/failing tests
Hose swivels	Failing prematurely
Dispenser	Leaks/failure/premature replacement
Solenoid valves	Clogged/failing
Riser pipe	Corrosion
Pipes	Failure

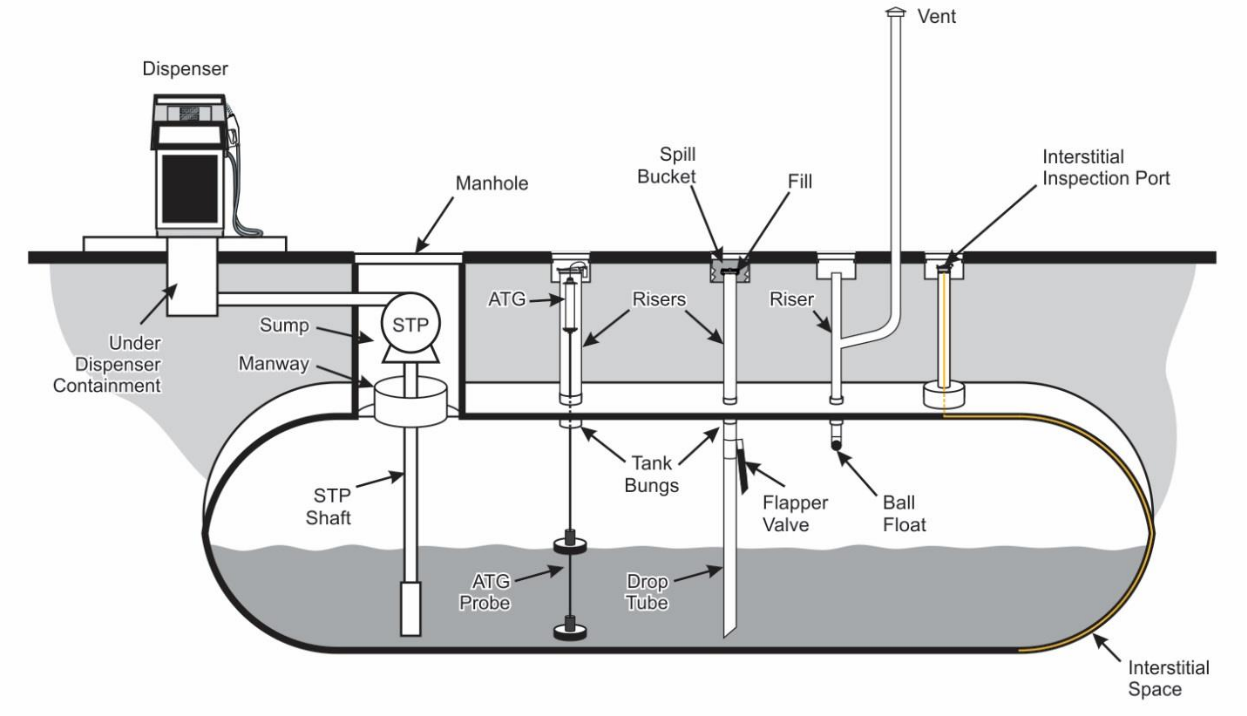


Figure 1. Simplified Diagram Of Diesel UST System Equipment

Around the same timeframe, Congress, which enacted the Renewable Fuel Standard (RFS) established by the Energy Policy Act of 2005 and amended by the Energy Independence and Security Act of 2007, directed EPA to require the volume of ethanol and biodiesel available for blending into petroleum-based fuels to increase significantly. Ethanol is blended into gasoline, and biodiesel is now regularly blended into diesel fuel, partly because biodiesel adds lubricity lost from the reduction of sulfur. Some microorganisms may preferentially degrade biofuel components in petroleum-biofuel mixtures.

A third change in the mid-2000s was a trend of more retailers storing and selling diesel. Retailers transitioned tanks that formerly stored mid-grade gasoline to diesel when blender pumps reduced the need for storing all three grades of gasoline. Diesel fuel infrastructure and blending and distribution practices vary across companies and over time as market conditions and fuels change. It is possible that any combination of these factors affecting the diesel fuel supply could be involved in the first-identified-in-2007 corrosion in USTs.

Initial Research Efforts

Because of the relative newness of this situation, little is known about the cause of the manifestation of vapor space corrosion in USTs storing diesel. Although extensive literature on corrosion in industrial applications is available, EPA thinks research specific to vapor space corrosion in USTs storing diesel is limited. Two pieces of research of particular interest are a 2012 hypotheses investigation by the Clean Diesel Fuel Alliance (CDFA) and an EPA Office of Research and Development (ORD) poster about research investigating corrosion in USTs.[5, 6]

In 2012, CDFA completed the first major field study on the issue. CDFA physically and analytically examined six operating UST systems storing diesel and concluded that the corrosion of UST metallic equipment was caused or enhanced by low molecular weight acids, such as acetic acid and formic acid, distributed throughout the system. CDFA believed the acids were the metabolic byproducts of microorganisms proliferating within diesel USTs and were contributing to corrosion through a mechanism called microbiologically-influenced corrosion (MIC). MIC is defined as the deterioration of metals due to the metabolic activities of microorganisms and is well-established in corrosion literature, although the mechanisms are not fully understood.[7] CDFA hypothesized that MIC is occurring when microbial populations degrade ethanol present in the fuel to low molecular weight acids. This was based partially on CDFA's study findings of ethanol presence in some of the fuel and aqueous phase samples collected from the population of tanks.

Ethanol was not expected to be present in diesel fuel. CDFA hypothesized the source of the ethanol was contamination of the fuel supply through cross or switch loading of fuel transportation trucks with no or inadequate cleaning procedures between deliveries or through other methods. CDFA further hypothesized that in the presence of oxygen and water, *Acetobacter* bacteria living in the aqueous phase of the USTs could use, or oxidize, the ethanol. Because a byproduct of that species' microbial metabolism is acetic acid, CDFA's hypothesis suggested that acid could then volatilize into the vapor space of the USTs, coating the metal equipment and causing corrosion.

Further examination of CFDA's results suggested that some of the organic acids indicated another possibility of MIC occurring as a result of microbial populations feeding on constituents of the diesel fuel itself and not only ethanol contamination. This could include the diesel fuel, fatty acid methyl esters (FAME) components of biodiesel in the fuel, and trace amounts of glycerol remaining from biodiesel production.[8] USTs storing diesel may contain up to 5 percent biodiesel blended, according to ASTM D975 standard for diesel fuel.[9] This biodiesel in turn may contain glycerol, a byproduct of the production process, also referred to as glycerin in the analytical methods used in our research. Most glycerol is removed after biodiesel production, but a low concentration is allowed to remain according to ASTM fuel standard D6751.[10] Specifically, free glycerin may not exceed 0.02 percent mass and total glycerin may not exceed 0.24 percent mass in the 100 percent biodiesel blend stock.[11]

Metabolic consumption of glycerol by microbial populations could produce volatile organic acids such as propionic, lactic, or glyceric acids. In CDFA's study, acetic acid prompted the hypothesis of ethanol presence being the cause of the corrosion; however, other organic acids were found. In 2013, EPA's ORD conducted a research study on ethanol and glycerol's potential role in microbial corrosion in USTs.[12] Comparison of ORD's findings on corrosion in water bottoms to CDFA's hypothesis suggested that CDFA's hypothesis of ethanol's role in MIC could also apply to glycerol in diesel fuel, although it was not listed as a hypothesis in CDFA's report conclusions. The role of biofuels in diesel corrosion has been suggested but not proven by these previous research efforts. The exploratory nature of our research was designed with the intent of being able to examine for any predictive factors in the corrosion and not just the hypothesized role of biofuels. By checking for predictive factors, the feasibility of either of these hypotheses should be determined in our research along with possible identification of other common patterns in the operation or maintenance, fuel distribution, environmental conditions, or UST equipment demographic areas.

While research publications specific to understanding the root cause of post-2007 corrosion in diesel USTs is limited, independent groups and private companies have developed several documents targeted towards minimizing the corrosion associated with storing diesel fuel. Five examples are the Coordinating Research Council's (CRC) *Preventive Maintenance Guide for Diesel Storage and Dispensing Systems* and *Diesel Fuel Storage and Handling Guide*; Clean Diesel Fuel Alliance's *Guidance for Underground Storage Tank Management at ULSD Dispensing Facilities*; Steel Tank Institute's (STI) *Recommended Practice for Storage Tank Maintenance R111 Revision*; and ASTM D6469, *Standard Guide for Microbial Contamination in Fuels and Fuel Systems*.[13, 14, 15, 16, 17]

Some owners indicated that following treatments presented in these documents has been effective in slowing corrosion in their affected USTs. These documents generally focus on minimizing water in order to limit microbial growth in USTs, among other enhanced maintenance practices. These practices may suggest more frequent water or fuel monitoring, using different filters, or using biocides or corrosion inhibitors. Other fuel treatment products intended to dissolve and remove microbial colonies and protect submerged metal surfaces anecdotally suggest success in limiting corrosion. Preliminary anecdotal results of limiting oxygen in USTs through the use of nitrogen blanketing equipment in order to prevent the growth of microorganisms that require oxygen for survival show potential to limit corrosion in the vapor space of USTs, as do filtration systems designed to remove particulates in fuel and water in which microbial colonies could live. These results are circumstantial, but together suggest probable supporting evidence from field experiences for laboratory research, which has hypothesized that MIC is likely involved in corrosion in USTs storing diesel and suggested limiting microbial populations is key to minimizing corrosion.

Changes to diesel fuel formulation, distribution, or storage practices over the last ten years could also be allowing natural processes other than bacterial oxidation of fuel or fuel contaminants to occur. These natural processes may not have been possible or may not have been as prevalent before diesel fuel began to change around 2006. Fungi, archaea, and eukaryotic organisms are other mechanisms that could be causing or contributing to corrosion attacks in UST systems storing diesel. A combination of these and other yet to be identified factors could also be responsible. Because the base of scientific research specific to corrosion in underground storage tanks since 2007 was limited, and because of the multitude of variables potentially involved, our research needed to have a broad focus that could best help all involved in the diesel fuel industry understand the issue more completely.

EPA Research Plans

EPA worked with industry to design research that would maximize the understanding of the issue within budget constraints. EPA collaborated extensively with CRC during the development of our research; CRC was already planning to complete an additional research study after EPA completed our work. EPA's collaboration with industry was important to avoid duplicating or overlapping efforts. EPA, by beginning research first, had the opportunity to focus on understanding the risks and best narrowing down, without needing to pinpoint, the possible causes. CRC's research plans are moving forward and taking into account preliminary findings from EPA's report.

After significant consultation with CRC, EPA decided that the most useful approach was to undertake exploratory field research of a large population of UST systems and look for predictive factors with UST systems experiencing severe corrosion. We present our findings in this report. EPA attempted to identify any corrosion predicting factors across 42 UST systems, a much larger group than examined before. This approach allowed EPA to look for anything that could be common to UST systems with corrosion, including the opportunity to examine if the hypotheses suggested by previous research would hold true across a larger and more diverse sample population. EPA weighed the opportunity to examine dozens of systems nationwide with the significant cost involved in laboratory work for the most extensive analysis options. Therefore, our research design required careful balance in selecting laboratory analyses that would reveal the most information about corrosion development without adding such significant cost that we would have to reduce the research population.

After we finalized our plan, we selected 42 operational USTs storing diesel. We inspected all UST systems in the diverse sample population for corrosion; gathered samples of fuel, vapor, and aqueous phase where possible; recorded site observations; and gathered available historical information about the UST systems from owners. We then categorized the UST systems according to the observed corrosion coverage as minimal, moderate, or severe. Next, we combined analytical results of the collected samples with historical information about the USTs, the site observations, and corrosion coverage categorizations and analyzed for predictive factors. The research results will add information to the body of knowledge about the extent of corrosion in USTs storing diesel fuel. We thought any predictive factors identified in our research would help focus the search of potential causes for the next phase of follow-on research.

Chapter 2
Methodology

EPA's research attempted to identify predictive factors for severe or minimal corrosion in USTs storing diesel by (1) inspecting and sampling UST systems and categorizing the USTs by corrosion extent, (2) analyzing the collected samples and building a large data set, and (3) statistically evaluating those data for predictive factors.

EPA developed and implemented our research according to a quality assurance project plan (QAPP); see Supplement A. The QAPP presents procedures for inspecting, categorizing, and sampling the 42 USTs storing diesel; methods for analyzing the samples; and methods for evaluating the data. The QAPP ensured uniform inspections of equipment. The QAPP also ensured that we accurately conducted a comprehensive analysis of samples and data. Our procedures and methods are described in detail in the QAPP and summarized briefly below.

I. Information Gathering

We followed the procedures and methods presented in the QAPP and conducted on-site inspections of 42 UST systems. The inspections included visually examining the UST system equipment and collecting samples of vapor, fuel, and aqueous phase, when present. This research also attempted to gather significant additional information from owners via questionnaire about each UST system's operation, maintenance, fuel supply, and storage history.

The on-site inspections were conducted between January 26 and February 13, 2015 at 40 different sites (at two sites, two USTs were inspected). EPA chose a diverse population of UST systems from available sites voluntarily offered by owners; some of the UST systems had known corrosion issues prior to the inspections. It is important to note that when attempting to organize inspection sites, EPA specifically asked potential volunteer owners for sites with a history of corrosion. Because EPA had no data on the percentage of systems actually affected by corrosion, we wanted to examine a population where 50 percent of sites had previous corrosion and 50 percent had no corrosion. Ultimately, EPA selected a research population that included less than 25 percent reporting knowledge of past or present corrosion. Regardless, the population of inspected systems in our research is not a random or representative sample of the diversity of USTs storing diesel in the United States. We discuss this and other differences between the sample and national population in more detail later in Chapter 2.

The USTs inspected during this research included both UST systems with steel tanks and UST systems with fiberglass tanks. CDFA's 2012 study gathered information through deeper analysis, but only focused on UST systems with fiberglass tanks. When planning EPA's research, we wanted to include an equal number of steel and fiberglass tanks in the sample population; however, due to logistical challenges of arranging available volunteered sites in clustered locations so as to minimize the amount of our research budget spent on travel, we inspected more fiberglass tanks than steel tanks, 24 and 18, respectively. The storage capacity of the tanks ranged from 5,000 to 20,000 gallons, with many of them 12,000 gallons, which is a typical size for the industry. Figure 2 presents the tank population by storage capacity and tank material. The inspection locations represented various geographic areas across the United States and were arranged in 10 general clusters to minimize inspection expenses and maximize the number of USTs sampled. Figure 3 presents a map depicting the site cluster locations. Site inspections were conducted on UST systems that had been operational for time periods ranging from 1 to 29 years. Eight different owners volunteered their systems. The owners included a federal agency, a local government, several commercial fuel retailers, and a non-fueling servicing company that maintains an UST storing diesel for its fleet. Some of the commercial retailers own one UST storing diesel and some own multiple USTs storing diesel. Details about the 42 USTs inspected in this study are presented in Supplement B.

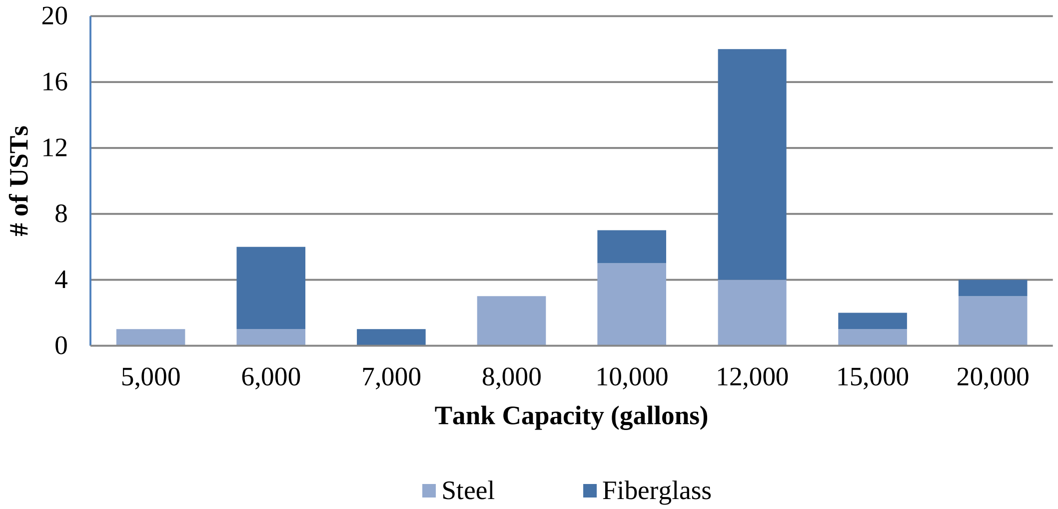


Figure 2. Summary Of 42 UST Systems By Capacity And Material

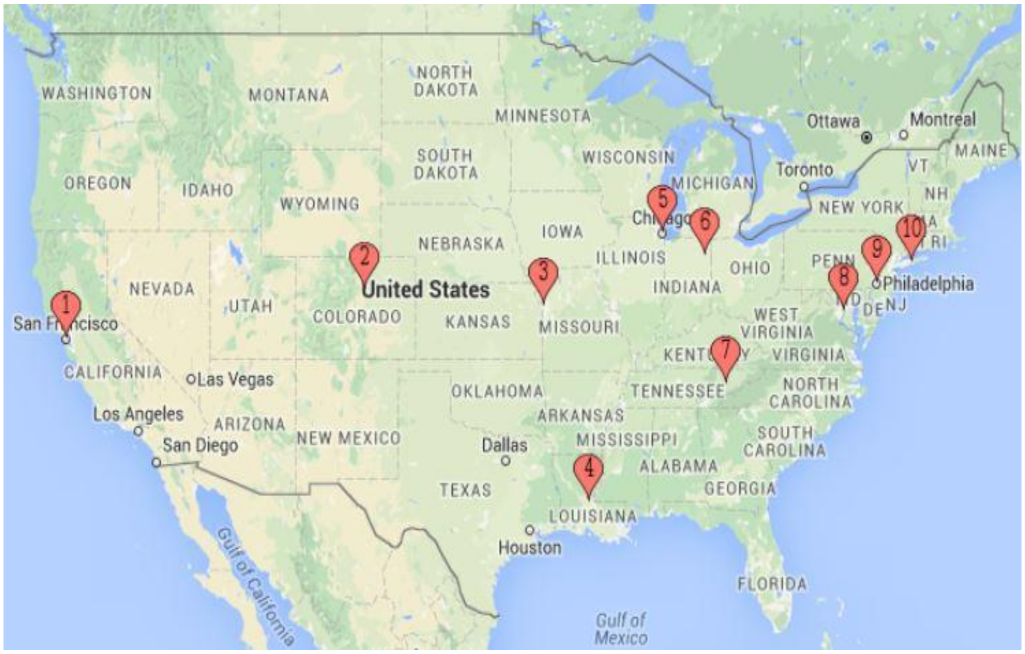


Figure 3. Locations Of Clusters Of USTs In Study Population

Difference In The Sample Population And National UST Population

The sample population of USTs storing diesel EPA examined is diverse, but most likely does not accurately represent the national demographic of the owners of federally regulated USTs storing diesel for three reasons. First, EPA estimates that more than 50 percent of federally regulated retail UST locations are the responsibility of owners with only one retail station, but because of limitations associated with coordinating volunteered sites, our research included only one individual site owner out of the eight participating owners. This is significant because owners of multiple UST systems are generally likely to be more aware of UST system issues and maintenance needs simply because of the requirements associated with operating multiple fueling systems. Large company owners of USTs at multiple locations often have employees specifically dedicated to managing these systems.

The second reason the diesel USTs selected for our research are not an accurate representation is because the sample was not random. During research design, EPA originally attempted to use a population with 50 percent of the UST systems with current or prior corrosion problems and 50 percent of the population without corrosion issues, according to the owners self-reporting. EPA specifically looked for UST systems whose owners believed or knew whether their UST had previous or current corrosion issues in order to use an equally split corrosion population to begin our research. However, EPA was not able to obtain a 50-50 split of reported corroded and non-corroded sites from the viable site options before the research began.

EPA and our contractor asked about corrosion histories through email and phone calls when scheduling potential participant USTs. The vast majority of potential volunteer owners said that although they would participate and allow inspection of their sites as part of the research, they were not aware of their UST systems having current or past corrosion issues. To verify the corrosion history of each UST system in the final research population, we asked all owners of USTs in the population if they knew of current or past corrosion in the UST. This was the first question on the questionnaire in Supplement A, Appendix B, which is discussed in more detail later in this chapter.

Of the 27 USTs for which this questionnaire was mostly answered, only five affirmatively reported current or past corrosion. The other answers were mixed: 15 owners replied to the question with unknown; two replied no; four replied none known about; and one did not answer the question. Of the remaining 15 USTs in the research population from whom responses were not collected, EPA is aware through conversations prior to beginning the research that at least three had reported prior corrosion issues. EPA believes for the other 12 of 42 sites the owners were not aware of past corrosion issues. In total EPA thinks eight of the 42 sites, or 19 percent of the total population, were aware of current or past corrosion issues prior to EPA beginning its research, but acknowledge that we cannot be certain.

We did not factor the number of sites with current or past corrosion into the statistical analysis of potential predictive factors, so it did not affect the research results. However, the number of owners who were aware of corrosion prior to research beginning is relevant to interpreting the findings. Therefore, we wanted to err on the side of more conservative results and use less than 25 percent to describe the number of UST systems reporting current or prior corrosion to account for the fact that it could actually be higher than the 19 percent we believe actually knew of corrosion. Therefore, despite an intentionally biased sample for the research population, overall less than 25 percent of the 42 chosen UST system owners reported a prior knowledge of corrosion in their system before EPA inspections.

The third reason the results are not an accurate representation of federally regulated USTs storing diesel fuel is that no emergency generator tanks (EGTs) were included in this study. EGTs number in the thousands nationwide; they store fuel for long periods of time with fuel storage turnover rates much lower than USTs for refueling at retail stations and non-retail locations. Therefore, many conditions in EGTs will be different and corrosion may be more or less of an issue than in USTs used for fueling vehicles or equipment other than emergency generators.

A. UST System Background Information

After EPA identified the UST systems to be inspected, we gathered additional background information by asking owners, via a detailed questionnaire, about their UST system service and maintenance histories. The goal of collecting this information was to include UST system storage history, fuel supply, maintenance, and oversight practices in the search for predictive factors for USTs having severe corrosion. We gathered some of this information on site during inspections, but much of it was collected via emails with owners or managers before and after the on-site inspections. Some owners of multiple USTs across several locations did not allow on-site employees to provide information, while other owners allowed direct communications only with local site managers. For some of the UST systems, maintenance records were housed on site, while other records were maintained in centralized databases.

Overall, much of this background information was incomplete or unavailable from the owners, regardless of the UST maintenance documentation method. This was the case for several reasons; for example, owners and operators may not have received the records when they purchased the UST from previous owners, or owners may not have retained records unessential to compliance reporting. Other reasons included staffing or procedural changes that made continuity of recordkeeping difficult. Sometimes the reason for incomplete answers was unknown.

Of the 42 UST systems inspected in our research, we obtained answers to all or the majority of the background questions for 27 of the systems; see information in Supplement B. Of the answers obtained, the level of detail and certainty varied greatly among the various owners. For example, some owners reported an estimated monthly throughput rounded to the nearest one thousand with no indication of the delivery volumes or number of deliveries. Others sent delivery records of the past 12 months containing the dates and accurate volumes of the deliveries. Owners were approached no more than three times for gaps or clarifications about the information.

B. On-Site Inspections And Corrosion Classification

On-site inspections included visual documentation of the UST system conditions with an internal tank video and photos and sample collection. We conducted one site inspection for each UST system, capturing the conditions of the UST system on the day of inspection. We then collected samples of fuel, vapor, and the aqueous phase, or water bottom, from each UST system following the detailed inspection procedure in the QAPP, which included completion of a checklist and sample collection log sheet. Inspection teams packed and shipped samples to laboratories for analysis of composition and various chemical properties according to the QAPP. The QAPP in Supplement A details our inspection procedures and contains the inspection checklist and sample collection log sheet used for the inspections. We used the recordings of the internal video inspections to categorize the level of corrosion in each of the USTs in the sample population.

The CRC Diesel Performance Group, comprised of industry stakeholders, developed a standard protocol to be used as an on-site corrosion coverage classification assessment procedure.[18] The CRC protocol was finalized in 2014 and was intended to be used by researchers in selecting UST systems for researching corrosion. The protocol instructs an assessor to inspect the dispenser filter and then estimate the amount of corrosion coverage on the STP shaft as an indication of the extent of corrosion throughout the system. The metal shaft is commonly reported as being affected by corrosion and is an integral piece of equipment for system operation. The STP shaft is a good indicator to assess corrosion because it is oriented vertically within the tank and has portions of the shaft that are almost always wetted with fuel, sometimes wetted with fuel, and almost always in the vapor space.

EPA used much of CRC’s protocol as the basis for determining the level of corrosion in each UST system. However, our procedure diverged from CRC’s protocol in that CRC’s protocol was designed for selecting specific sites to investigate, where EPA’s sites were predetermined. Our process also included more metal components than just the STP shaft in the assessments if the STP shaft did not appear to accurately represent the overall amount of corrosion observed on other metal surfaces in the UST. EPA’s process also diverged from CRC’s protocol because we based the final assessment of corrosion coverage on three independent assessments, and for some systems, finalized the categorization only after follow-up discussion by assessors. The field team camera operator made an initial assessment during the inspection, then EPA and our contractor each independently assessed the corrosion coverage present in an UST system by reviewing the internal tank videos. Similar to CRC’s protocol, the assessments in our research initially estimated the percent of corrosion coverage on the STP shaft. Then if necessary, we reviewed and discussed the overall corrosion coverage. The end result was a determination of the extent of corrosion coverage inside the UST systems according to three categories.

- Minimal: less than 5 percent coverage
- Moderate: 5 percent to 49 percent coverage
- Severe: 50 percent or greater corrosion coverage

Often, the three independent assessments were accurately made based only on the STP shaft, and often agreed unanimously on which corrosion category best represented the condition of the UST systems. However, the assessors did not agree unanimously on 22 initial assessments. In the situations where the three initial assessments were not in agreement, EPA, our contractor, and the field team manager discussed the UST system classification. For those observations where we did not reach unanimous consent after this additional discussion, the QAPP called for EPA to make the final determination. In each of those few situations where unanimous consensus was not reached through discussion, EPA made the final determination to classify the UST system in the category where there were already at least two of the same assessments out of the three, regardless of what EPA's individual assessment was.

II. Sample Collection

EPA collected vapor, fuel, and aqueous phase samples, if present, from each UST system per the QAPP procedures; the samples were then shipped to various laboratories for multiple analyses. See Table 2 for analytical methods by sample type. The first step of the inspection was to open the UST system and immediately collect vapor samples, as well as in-tank temperature and relative humidity readings. The vapor samples were collected from the vapor space above the fuel inside each UST. This space is also called the ullage space. In order to minimize mixing of fresh air in the tank, only one cap was opened during the vapor sampling, and the vapor collection was completed before opening any other parts of the UST. The vapor was collected at a rate of 1 liter (L) per minute for 100 minutes through a single tank opening, either the fill opening after removing the drop tube or the ATG opening after removing the ATG probe.

Once vapor sampling was complete, we collected a fuel sample from the tank through either the fill opening or the ATG opening. We did not collect fuel samples from the fuel dispenser because tank collection more accurately captures the condition of the bulk, unfiltered fuel stored.

Field teams then attempted to collect aqueous phase samples. Water in USTs can be entrained in fuel, separated into an aqueous phase at the bottom of the tank, or both. For this reason, the aqueous phase is more commonly referred to as a water bottom in the fueling industry. Approximately 0.5 L of aqueous phase water was needed to fill the sample jars per the QAPP; we collected this volume from 11 of the 42 UST systems. The low number of aqueous phase samples collected could be attributed to the sampling techniques and tank openings sampled in relation to the sloping of the tanks. USTs storing diesel are generally prone to have water accumulation that require active management to regularly remove it, so we acknowledge that a sufficient water sample collection from only 26 percent of the UST sample population could have been a result of the limitations of the sampling approach.

The field teams had two sampling methods available to them to attempt to collect the water. One was a closed-core type grab sampler, commonly known as the Bacon Bomb. This device can be swung slightly and drug along the bottom of the tank to collect water directly under the opening and in relatively close proximity to the area under the opening. The second sampling device was the Tanknology Vacuum Sampler (TVS), which was designed for the specific purpose of sampling water at the bottom of USTs. Tubing for this device is weighted and can be maneuvered to either side, reaching areas along the bottom of the tank further away from the area directly under the opening. All access points were available for entry to the USTs except for the point where the STP was installed. Depending on the sloping of the tank, the lowest point of the tank where water would collect may be under the STP. We did not remove and disassemble the STPs for our research. STPs are quite heavy and cumbersome to remove, and if severe corrosion were found, the corrosion could have hindered reinstallation of the pump or required replacement. This risk to an operational system, in addition to the possible interruption of diesel service to customers, was undesirable to research participants and EPA. EPA decided in the research planning stages not to remove STPs because of these concerns, and that may have affected our ability to gather more than 11 water samples for analyses.

For 10 of the 11 aqueous phase samples collected, an aliquot (~0.25 L) was filtered through a cellulose filter to capture biological material until the filter clogged. We did not conduct microbial community analysis on these filters as part of this investigation; rather, the filters remain archived at less than -60°C for potential deoxyribonucleic acid (DNA) identification analysis by EPA or any other interested entity researching the diesel corrosion issue.

EPA intended for all samples to be handled according to the QAPP; however, upon analysis of the samples, we identified isopropyl alcohol in 18 of the fuel samples. Isopropyl alcohol was specified in the QAPP as the cleaning agent for the sampling equipment because it should quickly volatilize into air, leaving the equipment clean and dry. We could not determine with certainty the source of the isopropyl alcohol in the sample results, but it is likely that the equipment may have been used before the isopropyl alcohol completely evaporated. Isopropyl alcohol was chosen as the cleaning agent because it is not expected to be present in diesel or in the UST environment through microbial metabolic processes; therefore, the identified isopropyl alcohol is most likely contamination from the cleaning procedure. Because the key findings of this study relate to general trends of corrosion observations and the search for predictive factors, the contamination does not change the results reported or impact the final conclusions of the report.

III. Sample Analyses

The samples were shipped from inspection sites directly to various analytical laboratories. Table 2 summarizes the chemical analyses conducted. Each laboratory was responsible for performing one or more of the analysis methods uniformly for all of the relevant samples collected. Table 2 is organized by sample type (fuel, aqueous phase, and vapor) and includes the method title and standard method number, if applicable, for each analysis performed on the samples. Each method listed in Table 2 has quality control (QC) procedures and samples that were required for analysis, along with the field samples, to ensure the quality of the measurements. A duplicate liquid fuel and aqueous phase sample was collected for at least 10 percent of the total samples collected. For the vapor samples, a field blank and a duplicate sample were collected and submitted for analysis for 10 percent of the samples. In addition, analytical QC samples were included by all the laboratories to verify that there was no cross-contamination or carry-over between samples during analysis. The QC requirements and acceptance criteria for fuel, aqueous phase, and vapor samples are presented in the QAPP.

IV. Statistical Analysis Approach

EPA's research was primarily exploratory in nature and sought the most likely association and pathway between corrosion of metal components inside diesel USTs and the potential predictor variables collected. For the main analysis, in terms of classification and prediction accuracy, the machine learning technique known as random forest classification was used. This technique is considered one of the best among all available methods as shown recently by Fernandez-Delgado, Cernadas, and Barro.[19] It is also particularly well suited to situations with more variables than observations. This approach helps to define the problem and to understand what characteristics are potential predictors of an UST system experiencing corrosion.

In addition, since previous research hypothesized that acids in the vapor space resulting from MIC were the cause of corrosion, an analysis was conducted to determine if the amount of acids correlated with the presence of corrosion. A calculation was performed to sum the concentrations of the low molecular weight acids to a single value of acid concentration, represented as total acidic hydrogen value, in order to compare this value across USTs in the population. An analysis of variance (ANOVA) was performed for this outcome, and simultaneous 95 percent confidence intervals were obtained for all three pairwise contrasts of the corrosion categories. This total acidic hydrogen quantity was also analyzed as a potential predictor in a random forest classification along with the other variables to see how it measures in importance compared to the rest of the variables.

Table 2. Analytical Methods By Sample Type

Determination Of	Fuel Analysis Methods	Method Identifier
Water content	Water in petroleum products, lubricating oils, and additives by coulometric Karl Fischer titration (Procedure B)	ASTM D6304
Density	Determination of density, relative density, and American Petroleum Institute (API) gravity of liquids by digital density meter	ASTM D4052
Total acid number (TAN)	Acid number of petroleum products by potentiometric titration	ASTM D664
Corrosion rating	Determining corrosive properties of cargoes in petroleum product pipelines	NACE TM-0172
Particulates	Particulate contamination in middle distillate fuels by laboratory filtration	ASTM D6217
Biodiesel content	Determination of biodiesel (FAME) content in diesel fuel oil using mid-infrared spectroscopy (FTIR-ATR-PLS method)	ASTM D7371
Flashpoint	Flash point by Pensky-Martens closed cup tester	ASTM D93
Free and total glycerin	Determination of free and total glycerin in biodiesel blends by anion exchange chromatography	ASTM D7591
Unknowns of interest	Gas chromatography mass spectrometry (GC-MS) full scan	Lab in-house method
Sulfur content	Determination of total sulfur in light hydrocarbons, spark ignition engine fuel, diesel engine fuel, and engine oil by ultraviolet fluorescence	ASTM D5453
Conductivity	Electrical conductivity of aviation and distillate fuels	ASTM D2624
Acetic, formic, propionic, butyric, and lactic acids	Determination of short-chain fatty acids by GC-MS	Lab in-house method
Determination Of	**Aqueous Phase Analysis Methods**	**Method Identifier**
Acetic, formic, propionic, lactic acids	Ion chromatography (IC) for short-chain fatty acids	Modified EPA 300
Glycerin	IC test for free glycerin	Lab in-house method
Cations (sodium, calcium, magnesium, potassium, ammonium) and anions (chloride, sulfate, nitrate and fluoride)	Determination of dissolved alkali and alkaline earth cations and ammonium in water and wastewater by ion chromatography	ASTM D6919[21]
pH	pH (electric)	EPA 150.1
Conductivity	Conductance (specific conductance, µmhos at 25°C)	EPA 120.1
Ethanol and methanol	Nonhalogenated organics using GC-flame ionization detector (FID)	SW846 8015B
Determination Of	**Vapor Analysis Methods**	**Method Identifier**
Percent relative humidity	Vapor space percent relative humidity	Hygrometer
Acetic, formic, propionic, and butyric acids	Carboxylic acids in ambient air using GC-MS	ALS Method 102
Lactic acid	Determination of lactic acid in ambient air	Modified NIOSH 7903

Chapter 3
Observations And Results

I. Corrosion Observations In USTs Storing Diesel

Data collected on site by the field teams were combined with the UST system information provided by owners through the questionnaire. Table 3 lists the UST systems that were inspected, along with some basic characteristics of each of the systems: location, installation year, tank material, and tank capacity. The UST systems are presented by corrosion category and then by geographical cluster. Table 3 also shows, with shaded rows, where aqueous phases were found and successfully sampled from 11 UST systems. We were unable to collect aqueous phases of sufficient volume from the other UST systems. An asterisk in the corrosion coverage column indicates where the three independent assessments were not in initial unanimous agreement and EPA, our contractor, and subcontractor discussed the classification before a category was assigned; we discuss this in Chapter 2. See Supplement B for additional UST system characteristics and inspection data.

Corrosion Was More Prevalent Than Expected

Approximately 83 percent – 35 of 42 – of the USTs evaluated in the 10 clusters around the United States were classified with moderate or severe corrosion; 17 of the 42 USTs were classified as having moderate corrosion and 18 of the 42 were classified as having severe corrosion. The remaining seven USTs were classified as having minimal corrosion. Figure 4 groups the UST systems by corrosion category and tank material – fiberglass and steel. The data showed that minimal, moderate, and severe corrosion of metal components occurred in both types of tank construction. Within the research population, even some of the few USTs classified as having minimal corrosion had corrosion. The inspections were a snapshot in time, and therefore predicting future corrosion is not possible. However, it is possible that even those USTs exhibiting minimal corrosion were at the beginning stages of more advanced metal corrosion. The corroded bung in Figure 6, an UST with overall minimal corrosion, is an example.

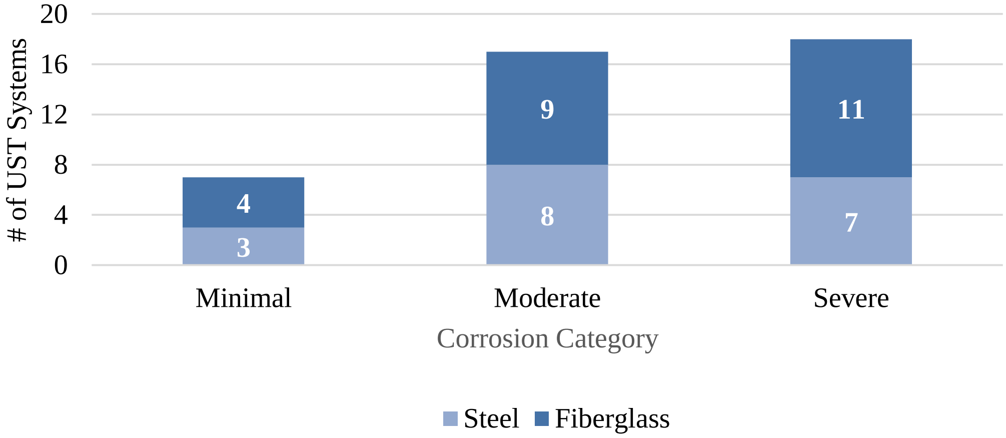


Figure 4. Forty-Two USTs By Corrosion Category And Material

For the USTs categorized as moderate or severe, visual corrosion observations from this research were consistent with observations of corrosion reported from PEI’s 2010 industry corrosion survey and CDFA’s 2012 study as well as general anecdotes from the UST industry.[20, 21]

Table 3. Characteristics And Corrosion Coverage Of USTs Inspected For This Research

UST System ID	Corrosion Coverage	Geographic Cluster	State	Installation Year	Tank Material	Capacity
22-CO-FG	Minimal	Denver	CO	2012	Fiberglass	12,000
48-CO-FG	Minimal	Denver	CO	2012	Fiberglass	12,000
14-LA-ST	Minimal*	Louisiana	LA	2012	Steel	8,000
29-CA-FG	Minimal	San Francisco	CA	2003	Fiberglass	20,000
31-CA-ST	Minimal	San Francisco	CA	1987	Steel (coated)	12,000
42-PA-ST	Minimal	Southeast PA	PA	1994	Steel	10,000
45-VA-FG	Minimal	Washington, DC	VA	Unknown	Fiberglass	10,000
2-IL-FG	Moderate	Chicago/Northern Indiana	IL	1995	Fiberglass	6,000
1-IL-FG	Moderate*	Chicago/Northern Indiana	IL	2006	Fiberglass	12,000
5-IL-FG	Moderate*	Chicago/Northern Indiana	IL	2005	Fiberglass	12,000
46-IL-ST	Moderate*	Chicago/Northern Indiana	IL	1992	Steel	20,000
23-CO-FG	Moderate*	Denver	CO	2014	Fiberglass	12,000
8-IN-FG	Moderate	Ft. Wayne	IN	2006	Fiberglass	12,000
12-MO-FG	Moderate	Kansas City	MO	2006	Fiberglass	12,000
10-MO-FG	Moderate*	Kansas City	MO	2005	Fiberglass	12,000
17-TN-FG	Moderate	Knoxville	TN	2011	Fiberglass	12,000
18-TN-ST	Moderate*	Knoxville	TN	1986	Steel	12,000
34-NY-ST	Moderate*	Long Island	NY	1992	Steel	12,000
37-NY-ST	Moderate*	Long Island	NY	1991	Steel	5,000
49-NY-ST	Moderate*	Long Island	NY	2010	Steel	10,000
27-CA-ST	Moderate*	San Francisco	CA	1998	Steel	12,000
39-NJ-ST	Moderate*	Southeast PA	NJ	1990	Steel	10,000
40-PA-ST	Moderate*	Southeast PA	PA	1994	Steel	10,000
44-VA-FG	Moderate*	Washington, DC	VA	Unknown	Fiberglass	10,000
3-IL-ST	Severe	Chicago/Northern Indiana	IL	1992	Steel (coated)	20,000
6-IN-ST	Severe*	Chicago/Northern Indiana	IN	1992	Steel	8,000
9-IN-FG	Severe*	Chicago/Northern Indiana	IN	2006	Fiberglass	12,000
26-CO-FG	Severe	Denver	CO	1996	Fiberglass	6,000
24-CO-ST	Severe*	Denver	CO	Unknown	Steel	8,000
7-IN-ST	Severe*	Ft. Wayne	IN	1992	Steel	6,000
11-KS-FG	Severe	Kansas City	KS	Unknown	Fiberglass	15,000
16-TN-FG	Severe	Knoxville	TN	2004	Fiberglass	12,000
32-NY-FG	Severe	Long Island	NY	2007	Fiberglass	12,000
33-NY-FG	Severe	Long Island	NY	2010	Fiberglass	7,000
35-NY-FG	Severe*	Long Island	NY	1986	Fiberglass	6,000
36-NY-ST	Severe*	Long Island	NY	1991	Steel	15,000
13-LA-FG	Severe	Louisiana	LA	2008	Fiberglass	12,000
15-LA-FG	Severe	Louisiana	LA	2000	Fiberglass	6,000
47-LA-FG	Severe*	Louisiana	LA	2002	Fiberglass	12,000
30-CA-FG	Severe	San Francisco	CA	2003	Fiberglass	6,000
28-CA-ST	Severe*	San Francisco	CA	1999	Steel	10,000
43-MD-ST	Severe	Washington, DC	MD	1992	Steel	20,000

Shading indicates aqueous phase was present and sampled.
*Indicates where initial classifications were not in unanimous agreement and EPA, our contractor, and subcontractor discussed the classifications as described in Chapter 2.

Observations sometimes included tubercle formations covering various metal surfaces of components within these UST systems. In many of the 42 USTs, we observed corrosion or tubercle coverage of the STP shaft only on the portion of the shaft in the vapor space. In some severe cases, we observed corrosion or tubercle coverage on the entire length of the STP shaft. Corrosion coverage patterns are discussed in more detail later in this report.

Most Owners Likely Not Aware This Could Be Affecting Their UST Systems

Approximately 83 percent of the inspected tanks showed moderate or severe metal corrosion. Owners reported having known corrosion in less than 25 percent of the USTs. As discussed earlier, the systems inspected may not be a representative sample of the United States; however, considering the many anecdotal reports from owners and service companies, previous research efforts, and results of the classification effort, the true prevalence of severe corrosion of metal components in UST systems storing diesel appears to be significant. Many owners are likely unaware that such corrosion may be affecting their UST systems.

Corrosion Of Metals Affects Both USTs With Steel Tanks And USTs With Fiberglass Tanks

In our research, we attempted to identify and include an equal number of USTs with steel tanks and fiberglass tanks in the sample population. However, our research used 24 fiberglass tanks and 18 steel tanks in the sample population. Our research shows that corrosion of metals is not dependent on the type of tank. Corrosion on metal components of diesel USTs was present in both UST systems with fiberglass tanks and UST systems with steel tanks. The corrosion was not limited to particular geographic regions. Our observations of the corrosion suggest that metal components in the vapor portions of tanks are most susceptible to corrosion, but several UST systems showed corrosion of metal equipment in lower portions, which generally remain wetted by fuel.

We observed corrosion on metals in the vapor space, including STP shafts, ATG shafts, drop tubes, and overfill prevention devices such as flapper valves and ball floats. Anecdotes from industry suggest that flapper valves and ball floats may be particularly prone to malfunction due to corrosion. These anecdotes also suggest that equipment not within the tank area, including shear valves, which are also known as fire valves or impact valves, and line leak detectors may be prone to functional failure from corrosion in diesel UST systems. In our research, we observed corrosion on metal bungs in the top of the tank and on the walls of some steel tanks.

Most Severe Corrosion Was Observed In The Highest Parts Of The Tank

One important observation was that the steel surfaces exposed to the vapor phase above the fuel generally appeared to show more corrosion than the surfaces that were generally fully immersed in the fuel. In multiple USTs, we observed corrosion appearing to be more advanced on portions of the STP shaft, which are generally in the vapor space than on the section generally in the fuel level. See an example from UST system 9-IN-FG in Figure 5.

From the inspections and general observations, the corrosion is more commonly severe in vapor spaces versus wetted portion of the tanks. Please note that because diesel fuel is often opaque, our assessment of the bottoms of the tanks was generally limited unless fuel levels were very low. However, in some USTs we evaluated, we observed severe corrosion nearly to the bottom of the UST.

Figure 5. UST System 9-IN-FG STP Shaft Corrosion
(Left Photo: Middle Of Shaft [Generally Submersed], Right Photo: Top Of Shaft [Generally Exposed To Air])

The STP shaft is situated vertically in the tank, and much of the shaft is exposed to both fuel and vapor space as fuel is dispensed and resupplied and the fuel level line rises and lowers in the tank. In some moderate corrosion cases observed in our research, the STP shaft was coated with corrosion in the vapor space, but was not yet affected in the wetted or sometimes wetted portions of the shaft. In some severe cases, the STP was entirely covered with corrosion. With the corrosion generally appearing heaviest in the highest points of the vapor space, the ATG riser and undersides of the manways, also referred to as tank bungs, are where the initial signs of corrosion should be expected, making these areas ideal for observing the beginning of corrosion development. In addition, we observed heavier corrosion in some areas of the ATG riser where vapor cannot circulate as easily around the rest of the tank.

Examples Of The Range Of Corrosion Effects

In general, there are limitations in drawing one corrosion assessment conclusion from many pieces of equipment within USTs. Different equipment reacts to the UST environment in different ways; components may be of different ages and constructed of different materials. However, inspecting other metal components in the UST when our first assessments using the STP shaft were not in unanimous agreement allowed us to confidently determine whether the corrosion problem existed in an UST system. Figures 6 through 9 show four examples of UST systems observed in our research: minimal corrosion in an UST system with a fiberglass tank, minimal corrosion in an UST system with a steel tank, severe corrosion in an UST system with a fiberglass tank, and severe corrosion in an UST system with a steel tank. These cases provide examples of the range of corrosion observed during our research.

Figure 6 presents an example of minimal corrosion coverage of metal components in an UST with a fiberglass tank that is approximately 12 years old (29-CA-FG). The riser and STP shaft are completely free of corrosion, and the filter is so new it gives no indication of the state of the UST. The bottom right photo of the bung at the top of the tank shows that some tubercles are forming around the inside of the bung. The formation of corrosion on the metal bung indicates that metals in this tank could be at the beginning of more advanced corrosion development.

Figure 7 shows photos of an UST with a steel tank that was also classified as having minimal corrosion (42-PA-ST). This tank has been in operation for approximately 21 years. The steel shell itself is in excellent condition for the age, as are the risers and the drop tube. This system uses a safe suction system rather than an STP to dispense fuel. The suction tube is also devoid of corrosion. It is unknown if any of this equipment has been replaced.

Figure 8 shows UST 35-NY-FG; with 29 years in service, this is the oldest tank in our research. The fiberglass tank in this UST system is expected to show signs of aging; however, corrosion development on the bungs is particularly severe, as is the corrosion coverage along the entire length of the STP shaft. It is interesting to note that this UST had the highest Total Acid Number (TAN) result; at 0.36 mg KOH/g it was significantly higher than all but one other UST. 48-CO-FG, at .28 mg KOH/g, was the only other UST to have a TAN result over .06 mg KOH/g. One additional general observation from some particularly severe cases is that the aluminum pieces, such as the ATG shaft and the drop tube, were sometimes coated with white deposits. Generally, aluminum will not corrode in environments with a pH above 4, so the white deposits in these severe cases suggest that the UST environment has been below pH 4 at least at some point in their service histories.

Figure 9 shows UST 43-MD-ST, a 23-year-old steel tank. The fill riser that was used for the video camera access lacks almost any evidence of corrosion. However, because it is generally protected from vapor by the drop tube, the riser is not a good indicator of the overall extent of corrosion in this UST system. The STP shaft is completely covered by corrosion and the ATG has some deposits along the length of the shaft and on top of the float.

II. Corrosion Coverage Classification Process

As discussed earlier in this report, we classified three levels of corrosion coverage by using a modified approach to the CRC protocol. For all but one UST classified as having minimal corrosion, our three assessments were in unanimous agreement. Classification consensus was most difficult to reach between moderate and severe coverage categories. The asterisks in Table 3 indicate when we discussed differing initial assessments of the videos before making final decisions.

Using the STP shaft as a surrogate piece of equipment for determining corrosion severity throughout the systems worked well for 20 of the 42 USTs, but the other 22 required more discussion to adequately assess the overall condition of each UST. This was particularly true with suction systems that do not use STPs for dispensing fuel. It appeared that some STP shafts or suction pipes were brand new, made of a different metal, coated or painted, or somehow non-corrodible because they appeared pristine; but other metal components in those USTs showed varying levels of corrosion, sometimes severe. It is possible that some shafts or pipes had been recently replaced due to corrosion, resulting in the appearance of less corrosion relative to other metals. Such information on maintenance histories was not always available from owners. We recognized that sometimes the entire system needed to be taken into account because of unique conditions observed in each UST inspection video (for example, differing visible portions of the STP and other equipment due to the fuel height, tank entry point, equipment orientation, and the technique of the camera operator).

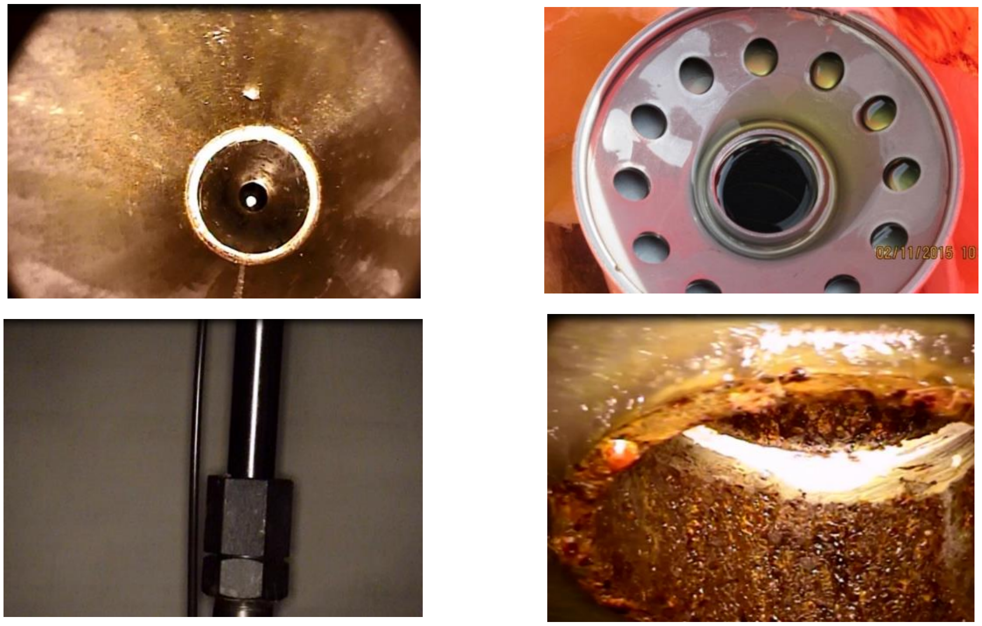


Figure 6. UST System With Fiberglass Tank With Minimal Metal Corrosion (29-CA-FG: Installed 2003, Age Of Filter < 1 Month). Top Left: Fill Pipe Opening; Top Right: Fuel Filter; Bottom Left: STP Shaft; Bottom Right: Tank Top Opening

Figure 7. UST System With Steel Tank With Minimal Metal Corrosion (42-PA-ST: Installed 1994; Age Of Filter Unknown). Top Left: Drop Tube And Tank Overview; Top Right: ATG Opening; Bottom Left: Ball Float; Bottom Right: Tank Top Showing Manway

Figure 8. UST System With Fiberglass Tank With Severe Metal Corrosion (35-NY-FG: Installed 1986; Age Of Filter Unknown). Top Left: ATG Opening; Top Right: Tank Top Openings; Bottom Left: Drop Tube; Bottom Right: STP Shaft And Tank Top Openings

Figure 9. UST System With Steel Tank With Severe Metal Corrosion (43-MD-ST: Installed 1992; Age Of Filter Unknown). Top Left: Fill Pipe Opening; Top Right: STP Shaft; Bottom Left: Tank Top Showing Manway; Bottom Right: ATG Probe And STP Shaft

In the video inspections showing the condition of each STP shaft or suction pipe, it was not clear if the black steel pipes were seamless or welded; however, according to the Steel Tank Institute, these fittings are generally made from seam-welded steel. Black steel pipe has a black oxide coating that forms during the forging process. The black coating is magnetite (Fe_3O_4) and provides some protection to atmospheric corrosion but can form red rust in the presence of water. The degree of corrosion depends on several factors, including, but not limited to, pH of the aqueous phase, availability of oxygen, duration of exposure, temperature, and presence of other contaminants such as chlorides.

In cases where the assessments disagreed, we launched the discussion with the state of the STP, but made the classification decision based on the discussion of the overall extent of corrosion coverage visible in the UST system video. In an ideal corrosion assessment, we would inspect each metal component of the UST system to determine the severity of corrosion. Although some pieces are easier to inspect than others, the most useful pieces of equipment to observe are the fuel filter, STP shaft, ATG riser, and undersides of the manways and bungs. Dispenser filters, while generally not reported as corroded equipment, may collect corrosion product and particulates over time and can be accessed without entering the tank. Anecdotes suggest that in some severe corrosion cases, what has been described as looking like coffee ground deposits clog the filter, causing the need for more frequent filter replacements. Clogged dispenser filters have sometimes prompted further investigation of the UST system for corrosion. However, if the service life of the filter is unknown or is known to be only a short period, it might not be a good indicator of the presence of corrosion inside the UST.

III. Analytical Results

Analytical results derived by performing the methods listed in Table 2 on the available vapor, aqueous phase, and fuel samples obtained from each UST system are summarized in table format in the following sections and are presented in full in Supplement B. The associated quality assurance (QA)/QC data summary tables are provided in Supplement C.

A. Quality Assessment And Oversight

The QAPP presents the QA assessment and oversight activities established to conduct our research. Supplement E includes a summary of the QA assessments performed throughout our inspections, data collection, and reporting process, as well as the audit reports and deviation reports. During our research, we identified six deviations from the QAPP, and we assessed the impact to the data quality in deviation reports. These deviation reports concluded there was no negative impact to the data quality or the conclusions of our report.

B. Vapor Analytical Testing And Results

In our research, the vapor analysis focused on identifying several acids that were hypothesized in previous research by CDFA and ORD as likely contributors to MIC. Additional acids were added to our research in order to test the feasibility of other contributors to corrosion. Table 4 summarizes a subset of the results. Vapor measurements from the 42 tanks showed both acidic and high moisture content conditions, which are conducive to corrosion development. Either acetic acid or formic acid, or both acids, were present in the vapor space of every UST inspected. These acids are the most abundant and volatile of the microbial byproducts hypothesized by previous research to be produced in the aqueous phases through MIC. Concentrations of acetic and formic acid were as high as 6,200 parts per billion by volume (ppbv) and 2,100 ppbv, respectively. Another acid suggested by previous research to be related to microbial consumption of biofuel components in diesel, propionic acid, was found in 12 of the samples at much lower concentrations (up to 8 ppbv). The vapor pressure of propionic acid, at 2.4 millimeters of mercury (mm Hg) at 20°C is significantly lower than acetic and formic acids, at 11.4 and 31.5 mm HG at 20°C, respectively.[22, 23, 24] Therefore, propionic acid would be expected to be at lower concentrations in the vapor even if liquid-phase concentrations of these acids were similar. Lactic acid was tested but not detected in any of the samples, possibly due to its low volatility (0.0813 mm Hg at 25°C).[25] The method reporting limit (MRL) for the vapor analysis is approximately 0.5 ppbv for these acids.

Hygrometer readings were also taken during the vapor sampling. The average relative humidity inside the USTs was 68 percent with a standard deviation of 15 percent; readings ranged from 33 percent to 98 percent. The sampling took place in January and February in different climates where the average annual ambient relative humidity ranged from 52 percent to 79 percent. We observed condensation produced by high humidity along the top of many of the tanks. In general, corrosion is more severe in humid environments than in dry environments. On some metals, corrosion can be slowed when humidity is reduced to below 50 percent.[26] High humidity and subsequent condensation allows the acids in the vapor space to sustain contact with the equipment as an aggressive electrolyte and liquid condensate. This facilitates corrosion development on the metal components. Furthermore, when the liquid condensate evaporates, the acids may concentrate on the surface, although the corrosive action may vary on the metal components.

USTs storing diesel are vented to the atmosphere, allowing ambient air to continually enter the tank. Therefore, USTs storing diesel are not expected to be free from climate influence on the UST environment, and relative humidity inside USTs storing diesel may be especially influential on corrosion development. Although the effect may be varied due to climate, USTs in all regions of the United States are susceptible to ambient air affecting conditions inside of the USTs. In humid climates, the UST is expected to have nearly continual high humidity, which promotes condensation in an UST, possibly making the UST more susceptible to corrosion development. Where the ambient humidity varies due to season, weather events, or geographic location, the relative humidity in the tank will also vary, allowing for the condensation and drying cycles that could affect the susceptibility and severity of corrosion development. Even in areas where humidity is low, daily temperature swings can produce periods of high humidity in USTs when warmer and cooler air mix. Again, this allows for condensation of liquids on surfaces inside the UST that may increase the severity of the corrosion on UST system components.

Table 4. Vapor Results In Order Of Corrosion Classification And Increasing Acetic Acid Concentration

UST System ID	Corrosion Class	Acetic Acid	Formic Acid	Propionic Acid	Average Relative Humidity in Tank (%)	Cluster Location Designation*	Cluster Location Annual Average Ambient Relative Humidity (%)**
		(ppbv)					
14-LA-ST	Minimal	15	ND	ND	73	Louisiana	76
45-VA-FG	Minimal	29	650	ND	82	Washington, D.C.	64
31-CA-ST	Minimal	80	490	ND	82	San Francisco	74
22-CO-FG	Minimal	100	ND	3.5	36	Denver	52
42-PA-ST	Minimal	150	76	4.5	69	Southeast PA	67
48-CO-FG	Minimal	550	470	ND	58	Denver	52
29-CA-FG	Minimal	1500	86	ND	62	San Francisco	74
8-IN-FG	Moderate	ND	210	ND	65	Ft. Wayne	79***
17-TN-FG	Moderate	ND	120	ND	71	Knoxville	76***
18-TN-ST	Moderate	ND	71	ND	70	Knoxville	76***
46-IL-ST	Moderate	ND	2100	ND	55	Chicago/ Northern Indiana	70
49-NY-ST	Moderate	14	840	ND	77	Long Island	63
10-MO-FG	Moderate	28	76	ND	88	Kansas City	68
40-PA-ST	Moderate	31	100	1.1	57	Southeast PA	67
37-NY-ST	Moderate	39	160	0.96	71	Long Island	63
27-CA-ST	Moderate	87	74	2.1	57	San Francisco	74
12-MO-FG	Moderate	88	85	3.1	59	Kansas City	68
1-IL-FG	Moderate	200	320	ND	41	Chicago/ Northern Indiana	70
23-CO-FG	Moderate	230	64	ND	52	Denver	52
34-NY-ST	Moderate	230	250	1	98	Long Island	63
5-IL-FG	Moderate	340	570	ND	33	Chicago/ Northern Indiana	70
44-VA-FG	Moderate	630	ND	ND	83	Washington, D.C.	64
39-NJ-ST	Moderate	1200	83	ND	76	Southeast PA	67
2-IL-FG	Moderate	2700	140	ND	83	Chicago/ Northern Indiana	70
3-IL-ST	Severe	ND	960	ND	59	Chicago/ Northern Indiana	70
6-IN-ST	Severe	ND	1500	ND	80	Chicago/ Northern Indiana	70

UST System ID	Corrosion Class	Acetic Acid	Formic Acid	Propionic Acid	Average Relative Humidity in Tank (%)	Cluster Location Designation*	Cluster Location Annual Average Ambient Relative Humidity (%)**
		(ppbv)					
9-IN-FG	Severe	15	270	ND	74	Chicago/ Northern Indiana	70
24-CO-ST	Severe	21	300	ND	72	Denver	52
26-CO-FG	Severe	28	ND	ND	35	Denver	52
30-CA-FG	Severe	62	96	1.4	61	San Francisco	74
33-NY-FG	Severe	81	330	8	83	Long Island	63
7-IN-ST	Severe	150	1700	ND	86	Ft. Wayne	79***
15-LA-FG	Severe	220	70	ND	95	Louisiana	76
36-NY-ST	Severe	230	620	1.2	77	Long Island	63
32-NY-FG	Severe	400	150	5.5	68	Long Island	63
11-KS-FG	Severe	990	300	ND	79	Kansas City	68
43-MD-ST	Severe	1000	140	3.8	66	Washington, D.C.	64
16-TN-FG	Severe	1100	65	ND	67	Knoxville	76***
28-CA-ST	Severe	1900	69	ND	56	San Francisco	74
47-LA-FG	Severe	2500	ND	ND	61	Louisiana	76
35-NY-FG	Severe	2600	110	ND	72	San Francisco	74
13-LA-FG	Severe	6200	ND	ND	84	Louisiana	76

ND = not detected at approximate MRL of 0.5 ppbv

* Weather data for cluster location based on the following cities (Cluster name - city used for weather data): Louisiana - New Orleans, LA; Washington, D.C. - Washington, D.C.; San Francisco - San Francisco, CA; Denver - Denver, CO; Southeast PA - Philadelphia, PA; Ft. Wayne - Ft. Wayne, IN; Knoxville - Knoxville, TN; Chicago/Northern Indiana - Chicago, IL; Long Island - New York, NY; Kansas City - Kansas City, MO.

**Data obtained from http://www.currentresults.com/Weather/US/humidity-city-annual.php unless otherwise noted.

*** Data obtained from http://www.usa.com/.

C. Aqueous Phase Analytical Testing And Results

EPA sampled aqueous phase per the QAPP at 11 of the 42 USTs inspected. Samples were analyzed by the methods presented in Table 2 and a subset of results is presented in Table 5. The aqueous phase analyses focused on identification of acids that were hypothesized in previous research to result from MIC and other water quality factors that may influence corrosion development. These methods included analysis for low molecular weight acids, glycerin, cations and anions, pH, conductivity, and nonhalogenated organics, in particular ethanol and methanol.

Acetic, formic, and propionic acids were identified in the majority of water samples analyzed. Acetic acid was measured in all 11 samples, ranging from 7 ppm to about 27,000 ppm. Formic acid was found in 10 of the 11 samples, ranging from 1.3 to 269 ppm. Lactic and propionic acids, both less likely to be measured in vapor due to their heavier vapor pressures, were detectable in most of the water samples. Lactic acid was found in nine samples at concentrations up to 1,900 ppm, and propionic acid was measured in seven samples up to 480 ppm. The water samples were also analyzed for ethanol and methanol content. All of the samples had measurable ethanol concentrations that ranged from 210 to 71,000 ppm. Methanol was measured in eight of the 11 samples from 33 to 3,200 ppm. Glycerol was measured in five of the samples as high as 18,322 ppm. pH measurements were acidic, ranging from 3.4 to 6.2.

Unfortunately, we did not have information available about the tilt of the tank or location of the lowest point in the tank. As a result, we may not have been sampling at the lowest point where water would collect. This is of note because UST management best practices suggest water levels should always be maintained as low as possible, so a well-maintained tank may not have water in one end of the tank because of the tilt. Despite not knowing where the water was located in the tanks, we were still successful at gathering the required volume of water for sample collection from 11 of the USTs.

D. Fuel Analytical Testing And Results

EPA analyzed fuel samples according to the methods presented in Table 2; Table 6 summarizes some fuel variables of note for our research. The fuel analyses focused on characterizing diesel fuel properties that may be affected by the presence of corrosion or contribute to or signal the presence of corrosion. In addition, we used a broad screening for identification of unknown chemicals.

The density, flashpoint, and sulfur concentrations were all within the expected ranges for all fuel samples. All conductivity results were above the minimum level of 25 picosiemens per meter (pS/m).[27] A qualitative full-scan analysis did not identify short-chain fatty acids or methanol. Water content of the fuel was above the benchmark of 200 milligrams per kilogram (mg/kg) in 10 percent of the samples.[28]

Biodiesel was identified in 70 percent – or 29 out of 42 – of the samples, with one sample (5-IL-FG) appreciably higher than the 5 percent allowable limit according to the ASTM D975 standard, measuring at 11 percent biodiesel. Free glycerin was measured in five samples, and all five results were at levels equal to or greater than the maximum amount of 0.001 percent when using 5 percent of the B100 D6751 standard of 0.02 percent. Even though 70 percent of fuel contained biodiesel, less than half – or 20 out of 42 – had a measurable total glycerin. Only two of those were over the maximum limit of 0.012 percent, when compared to 5 percent of the D6751 standard at 0.24 percent. Similarly without a standard, the TAN allowable for the B100 is 0.50 mg KOH/g, meaning an expected level in diesel samples should be no more than 0.025 mg KOH/g; however, 16 samples exceeded this value. These comparisons to D6751 provide a bench mark for what could be expected when assuming that any glycerin or acid measured was due to the biodiesel blending. Knowing there are acids in the vapor and aqueous phases, these glycerin and TAN levels may be elevated from another source, like microbial byproducts. In addition, there were three instances where the free glycerin result was higher than the total glycerin result. These results may have been affected by water that was entrained in the fuel during sampling but separated into an aqueous phase containing some glycerin before analysis. The results could have also been affected by systemic variation of the analysis method, since the results were close to each other.

Ninety percent, or 37 out of 41, of the National Association of Corrosion Engineers (NACE) International corrosivity tests of the fuel reported 0 percent corrosion observed on the testing material surface, which equals an A rating.[29] One NACE result was not available from the 42 USTs because the sample was lost in transport. The NACE test results for the other four available samples received the worst rating of E, meaning greater than 75 percent corrosion coverage on the testing material surface; having no ratings in between the A and E results is an interesting result. Possibly confounding the NACE result was that one of the four fuel samples receiving an E rating was collected from an UST categorized as having minimal corrosion. This is very possible because the NACE test is performed on a sample in a lab as a measure of the corrosivity only of the fuel that was gathered at the time of sampling, and is not a measurement of all conditions inside of an UST that could lead to corrosion of internal metal components. Even if this instance of highly corrosive fuel coming from a minimally corroded UST is accurate, the uniformity of the NACE fuel corrosivity test results on both extremes appears suspect, suggesting it may not have been performed correctly. However, this does not affect our research results because the key findings relate to general trends of corrosion observations and the search for predictive factors, none of which were found with any statistical significance.

Contaminants Identified In Fuel Samples

The most notable observation from the fuel analyses is contamination in the form of particulates, ethanol, and possibly gasoline, which is identified as gas contamination C4-C8 in Table 6 in the report and Table B-5 in Supplement B. We observed contamination from ethanol at high occurrences. The results of the qualitative gas chromatography-mass spectrometry (GC-MS) full scan for unknown chemicals of interest found ethanol was present in 90 percent – or 38 out of 42 – of the samples. The test also identified C4-C8 carbon chains in all of the fuel samples. We performed this test with the intention of identifying any gasoline contamination, but now understand that C-8 carbon chains could possibly be present from allowable sources in the fuel, such as biodiesel, and not only gasoline. We cannot therefore definitively say if gasoline was the source of the C4-C8 findings in 100 percent of the samples. Fuel particulate results are generally accepted by the industry under 10 milligrams per liter (mg/L); however, 40 percent – or 17 out of 42 – of the samples were above this threshold, with five of the results well over 100 mg/L.[30, 31]

Table 5. Aqueous Phase Results In Order Of Corrosion Classification And Increasing Acetic Acid Concentration

UST system ID	Corrosion Class	pH	Conductivity (µS/cm)	Ethanol (ppm)	Methanol (ppm)	Acetic Acid (ppm)	Formic Acid (ppm)	Propionic Acid (ppm)	Lactic Acid (ppm)	Glycerin (ppm)
45-VA-FG	Minimal	3.37	4,450	320	33	5,421	19.1	<0.26	80.0	2,183
48-CO-FG	Minimal	4.55	6,470	4,800	3,200	25,157	196	<0.26	1,915	2,590
37-NY-ST	Moderate	4.14	3,700	210	140	6.98	<0.14	<0.14	0.70	<0.006
18-TN-ST	Moderate	6.24	5,280	71,000	ND	1,517	54.3	481	38.2	<0.50
2-IL-FG	Moderate	4.07	2,670	4,100	100	13,003	43.4	12.5	28.1	<0.10
44-VA-FG	Moderate	3.86	6,180	5,100	440	17,684	106	2.40	599	18,322
6-IN-ST	Severe	4.86	2,540	230	ND	3,745	269	32.1	<2.56	<0.25
13-LA-FG	Severe	4.14	1,010	8,400	110	8,790	1.29	11.0	17.4	<0.025
7-IN-ST	Severe	5.04	7,530	4,100	ND	19,078	123	22.6	<2.53	<0.10
26-CO-FG	Severe	4.49	8,610	2,000	2,400	19,919	67.0	<0.26	1,464	2,399
35-NY-FG*	Severe	5.43	6,390	5,700	650	26,971	73.9	4.08	345	496

µS/cm – microsiemens per centimeter
ND – not detected
*Not enough sample volume to collect filtered water sample.

Table 6. Fuel Results In Order Of Corrosion Classification And Increasing Particulate Contamination

UST System ID	Corrosion Class	Particulates (mg/L)	TAN (mg KOH/g)	NACE* (Rating)	Water Content (ppm)	Aqueous phase Presence	Biodiesel Content (%)	Free Glycerin (wt. %)	Total Glycerin (wt. %)	Ethanol Presence	Gas Contamination (C4-C8)
Standard		**< 10[32, 33]**	**0.025[34]**	**A[35]**	**< 200[36]**		**5[37]**	**0.001[38]**	**0.012[39]**	**Not Expected**	**Not Expected**
42-PA-ST	Minimal	3.2	0.03	A	48		2.7	<LOQ	0.002	x	x
29-CA-FG	Minimal	3.6	0.03	A	62		3.5	<LOQ	0.003	x	x
31-CA-ST	Minimal	4.0	0.00	A	46		0.6	<LOQ	<LOQ	x	x
14-LA-ST	Minimal	4.8	0.01	A	63		0.0	<LOQ	<LOQ	x	x
45-VA-FG	Minimal	5.2	0.01	A	76	x	0.1	<LOQ	<LOQ	x	x
22-CO-FG	Minimal	6.8	0.01	A	67		2.0	<LOQ	0.001	x	x
48-CO-FG	Minimal	103	0.28	E	322	x	2.0	0.068	0.109	x	x
5-IL-FG	Moderate	3.6	0.02	A	83		11	<LOQ	0.004	x	x
39-NJ-ST	Moderate	4.4	0.00	A	41		0.98	<LOQ	<LOQ	x	x
23-CO-FG	Moderate	4.8	0.01	A	50		2.3	<LOQ	0.001	x	x
1-IL-FG	Moderate	5.2	0.01	A	76		4.9	<LOQ	<LOQ	x	x
49-NY-ST	Moderate	5.2	0.04	A	73		5.1	<LOQ	0.002	x	x
17-TN-FG	Moderate	5.6	0.01	A	16		0.0	<LOQ	<LOQ	x	x
34-NY-ST	Moderate	5.6	0.03	A	74		5.1	<LOQ	0.006	x	x
18-TN-ST	Moderate	6.0	0.00	A	26	x	0.0	<LOQ	<LOQ	x	x
12-MO-FG	Moderate	6.8	0.01	A	58		4.5	<LOQ	0.005	x	x
27-CA-ST	Moderate	8.0	0.04	A	58		2.1	<LOQ	0.001	x	x
8-IN-FG	Moderate	9.6	0.02	A	40		0.0	<LOQ	<LOQ	x	x
10-MO-FG	Moderate	13	0.01	A	172		4.4	0.028	0.021	x	x
40-PA-ST	Moderate	13	0.02	A	218		2.4	<LOQ	0.002		x
2-IL-FG	Moderate	17	0.01	A	78	x	0.0	<LOQ	<LOQ	x	x
44-VA-FG	Moderate	52	0.03	E	72	x	0.3	0.004	0.001	x	x
37-NY-ST	Moderate	63	0.01	A	154	x	1.2	<LOQ	<LOQ		x
46-IL-ST	Moderate	85	0.00	A	32		0.7	<LOQ	<LOQ	x	x
36-NY-ST	Severe	2.0	0.01	A	41		0.1	<LOQ	<LOQ	x	x
15-LA-FG	Severe	5.6	0.01	A	66		0.0	<LOQ	<LOQ	x	x

Table 6. Fuel Results In Order Of Corrosion Classification And Increasing Particulate Contamination (Continued)

UST System ID	Corrosion Class	Particulates (mg/L)	TAN (mg KOH/g)	NACE* (Rating)	Water Content (ppm)	Aqueous phase Presence	Biodiesel Content (%)	Free Glycerin (wt. %)	Total Glycerin (wt. %)	Ethanol Presence	Gas Contamination (C4-C8)
Standard		**< 10[40,41]**	**0.025[42]**	**A[43]**	**< 200[44]**		**5[45]**	**0.001[46]**	**0.012[47]**	**Not Expected**	**Not Expected**
43-MD-ST	Severe	5.6	0.00	NA	41		0.0	<LOQ	<LOQ	x	x
13-LA-FG	Severe	6.4	0.02	A	81	x	0.0	<LOQ	<LOQ	x	x
47-LA-FG	Severe	8.4	0.03	A	58		1.7	<LOQ	0.003	x	x
33-NY-FG	Severe	9.2	0.04	A	84		5.3	<LOQ	0.003	x	x
7-IN-ST	Severe	9.6	0.01	A	68	x	0.0	<LOQ	<LOQ	x	x
16-TN-FG	Severe	10	0.02	A	50		0.0	<LOQ	<LOQ	x	x
3-IL-ST	Severe	11	0.03	A	38		0.0	<LOQ	<LOQ	x	x
32-NY-FG	Severe	11	0.04	A	96		5.2	<LOQ	0.003	x	x
30-CA-FG	Severe	12	0.02	A	94		3.3	<LOQ	0.002		x
6-IN-ST	Severe	19	0.04	A	105	x	0.0	<LOQ	<LOQ	x	x
35-NY-FG	Severe	41	0.36	A	186	x	3.4	<LOQ	0.003	x	x
26-CO-FG	Severe	78	0.03	A	134	x	0.9	<LOQ	<LOQ	x	x
28-CA-ST	Severe	112	0.03	E	107		0.8	<LOQ	<LOQ	x	x
11-KS-FG	Severe	158	0.06	A	127		4.3	0.001	0.006		x
9-IN-FG	Severe	286	0.04	A	202		0.0	<LOQ	<LOQ	x	x
24-CO-ST	Severe	294	0.02	E	264		2.1	0.008	0.006	x	x

*The NACE rating corresponds to the percent of the test surface corroded in the laboratory (A = 0%, B++ = less than 0.1%, B+ = less than 5%, B = 5-25%, C = 25 – 50%, D = 50-75%, E = 75 – 100%)

mg/L – milligrams per liter

NA – not available; samples lost in transport.

Blank space – not present

LOQ – limit of quantitation

Since ethanol is routinely blended at 10 percent in gasoline, identifying in diesel tanks high occurrences of contamination of both ethanol and possible gasoline, represented by C4-C8 carbon chains, suggests these contaminants could be present because the diesel was delivered in trucks that previously shipped gasoline. Shipping multiple fuels in the same truck is referred to as transport switch loading. Ethanol generally is not transported in pipelines and is almost exclusively transported by trains or transport trucks to the blending terminals and blended with gasoline in trucks for distribution. The contamination attributed to switch loading occurs when gasoline is not completely removed between loads due to incomplete emptying or residual gasoline on the tanker walls due to surface tension. Switch loading may be the main source of the ethanol contamination measured in the samples; however, the presence of ethanol could also be attributed to or partially attributed to other unknown sources, such as shared venting from neighboring tanks, proprietary additives, or other unidentified sources.

EPA compared the fuel sample results to a variety of specifications and guidelines, including ASTM D975, ASTM D6751, NACE corrosion ratings, and CRC fuel selection guidelines.[48, 49, 50, 51] ASTM D975 and ASTM 6751 standard specifications were developed to help ensure these fuels are fit for use at the point of production, but these specifications do not guarantee that contamination will not occur downstream of the production facility during transport or storage. These guidelines and specifications cannot individually guarantee that the fuel dispensed is fit for use when it leaves the UST. By comparing each of the fuel samples collected to all of these fuel criteria, EPA tried to determine a better overall picture of the quality of the fuel mixtures being stored in the sample population USTs. When we assessed the fuel samples by looking at all the criteria together, the results were mixed; 90 percent or more of the fuel samples met the ASTM D975 criteria examined, while only 62 percent met the proportional TAN limit if using 5 percent of the maximum limit per D6751. Only 62 percent passed the CRC protocol for particulate. Fuel quality is usually verified against standards at various points in the production and distribution chain; however, by the time fuel reaches the final UST destination, fuel quality may be degraded or may become degraded by poor conditions in the UST. Our research results indicate that the fuel quality, including water or contaminants present in the UST, is moderate at best in the sample population.

Overall, the diesel fuel stored in the USTs in our research appears to meet the requirements for D975. Fuel filters are designed to remove most of the particulate contaminants found in fuel. However, water collecting at the bottom of the tank and the condition of the tank and equipment could also influence the quality of fuel stored. Additionally, water may not be caught in the filter. Excessive water in an UST system could be pumped up and dispensed with the fuel. Water-stopping filters are available to owners of USTs, but cost more than filters that do not stop water. The presence of contaminants and acids in all aqueous phase samples indicates that the fuel and water mixture stored in USTs may not meet all standard specifications required for fuel further upstream.

If a fueling mixture including water, contaminants, or acids is dispensed into vehicles, it could cause performance problems for end users of the fuel. This assumption is supported by reports of severely corroded vehicle fuel system parts. Automakers have found formic and acetic acids in fuel obtained from the dispensers at filling stations, in residual fuel from vehicle fuel tanks, and absorbed by vehicles' organic tank inner coating.[52]

Chapter 4
Statistical Analysis Procedure And Results

EPA's research was primarily exploratory in nature and sought to determine the most likely connections between predictor variables and UST corrosion severity, as well as if those connections would be strong enough to be predictive or show causation. Overall, none of the variables is considered statistically significant to predict with certainty when severe corrosion will be present. However, variables that were the closest to being significant predictors were particulates in the fuel and entrained water content in the fuel, which is different from aqueous phase presence on the bottom of the UST. These do not necessarily suggest causation, but may be areas for further investigation.

Supplement D presents full descriptions of all variables collected, the assessed corrosion coverage category, which is considered as the main outcome of interest, and all others as potential explanatory variables. Continuous-scale variables such as acid concentrations, descriptive statistics such as the mean, standard deviation, minimum, maximum and the 10th, 25th, 75th, and 90th percentiles, as well as counts of missing and non-missing measurements, are also provided in Supplement D.

For variable classification and prediction accuracy, the machine-learning technique known as random forest classification is considered one of the best available methods, as shown recently by Fernandez-Delgado et al.[53] It also is particularly well suited to situations with more variables than data points. With a random forest classification for corrosion category, two associated measures of variable importance were calculated for prediction for all variables: the mean decrease in accuracy and the mean decrease in Gini index, which is a measure of homogeneity with respect to corrosion category. Each of the 10,000 trees generated for the forest is fit on a bootstrap sample of approximately two-thirds of the UST systems. The predictive accuracy is then estimated by using the tree to classify the corrosion category for the UST systems not used in the current fit, referred to as out-of-bag (OOB) estimates, and comparing them with their true corrosion category. The mean decrease in accuracy measure is computed by randomly rearranging the values of the OOB UST systems and comparing the number of UST systems correctly classified with the true corrosion category. Thus, those variables with a higher mean decrease in accuracy over all the trees are more important for prediction. Alternatively, the mean decrease in Gini index gives an estimate of how homogeneous the partitions of the data become when using that variable to make decision rules that define the tree structure. While this does not provide an estimate of the variable's direction of effect, both measures quantify how important a particular variable is for classification and prediction accuracy. We provide the results of these analyses for corrosion classified as minimal, moderate, or severe, as well as combined categories of only minimal versus moderate plus severe.

In another supporting analysis, univariate analyses are presented by examining how well corrosion is predicted by each variable on its own. Corrosion is assessed by a three-category ordered scale, so the natural, parametric model to use is proportional odds logistic regression. Due to the limited number of tanks sampled in comparison to the number of explanatory variables that can be used for analysis, the quantitative results of statistical significance presented as p-values serve as a general guide to the most likely indicators of corrosion examined in our research. These analyses provide intuition for expected marginal effects. Similarly, we provide the ordered p-values from the logistic regressions with corrosion classification further combined to minimal versus moderate plus severe.

The results of the two measures of variable importance, the mean decrease in accuracy and the mean decrease in Gini index, are presented in Figures 10 and 11. Variables with scores farther right on the horizontal axes are more important for predictive accuracy in terms of the given criteria. Overall, when evaluating all three corrosion categories, the model does reasonably well at prediction with an OOB estimate of the error rate of 52.38 percent. The sulfur and particulate measures from the fuel are deemed more important across the two importance measures. Variable names include a description and a letter at the end. These letters refer to the variable type, identifying explanatory variables without a letter and continuous-scale variables by sample type of fuel (f) or vapor (v).

When evaluating minimal corrosion against moderate and severe together, the OOB estimate of error rate associated with Figure 11 is 19.05 percent. For the dichotomous corrosion outcome, particulate measurement from the fuel is the one covariate singled out across both importance scores. The random forest does better at the OOB error rate, but does so by classifying all but one UST system as corroded. But, because the random forest model for the combined corrosion classification does not have good discriminatory ability on its own, its value is to again find that the particulate measurement from the fuel is the one most associated with a corrosion classification. The marginal p-value ranking from the ordered logistic regression is presented in Table 7, with smaller p-values providing a higher ranking of variable significance in predicting corrosion category. In the proportional odds logistic model, the conditional odds of going from the lower to higher corrosion category on the three-category scale are the same from minimal to moderate or severe and from minimal or moderate to severe. A positive log odds estimate means that a higher value for the relevant variable is associated with increased odds of having a more severe corrosion classification.

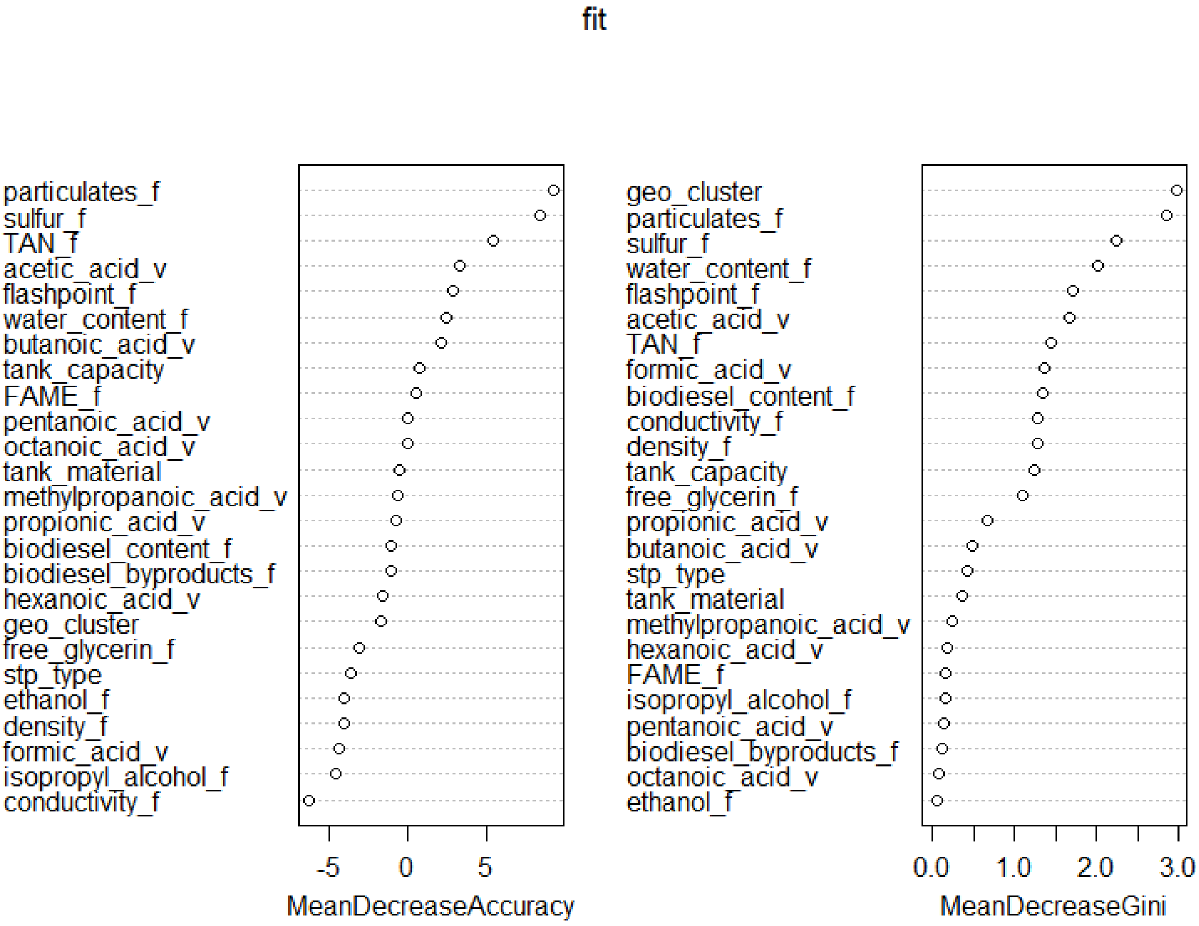
Statistical Analysis Results

Overall, due to the complexity of the processes at play, none of the findings is statistically significant to identify a definitive predictor of factors leading to the corrosion. We did not find a statistical significance with respect to tank age, size, material, geographic location, or owner. Many variables are interrelated and represent various portions of an ongoing process. No statistical significance was found in having or not having an aqueous phase, although as discussed earlier, we acknowledge that we expected water to be present in a larger percentage of the tanks than in the 26 percent where samples were collected; we do not know if a larger sample size may have yielded a different result.

None of the variables is considered statistically significant to predict with certainty when severe corrosion will be present. However, these variables were the closest to being significant: water content in the fuel, which is different from aqueous phase presence, and fuel particulates, with the particulates measurement being one of the more significant variables. A high measurement on the random forest analyses combined with a positive log-odds coefficient is associated with higher odds of having a more severe corrosion assessment.

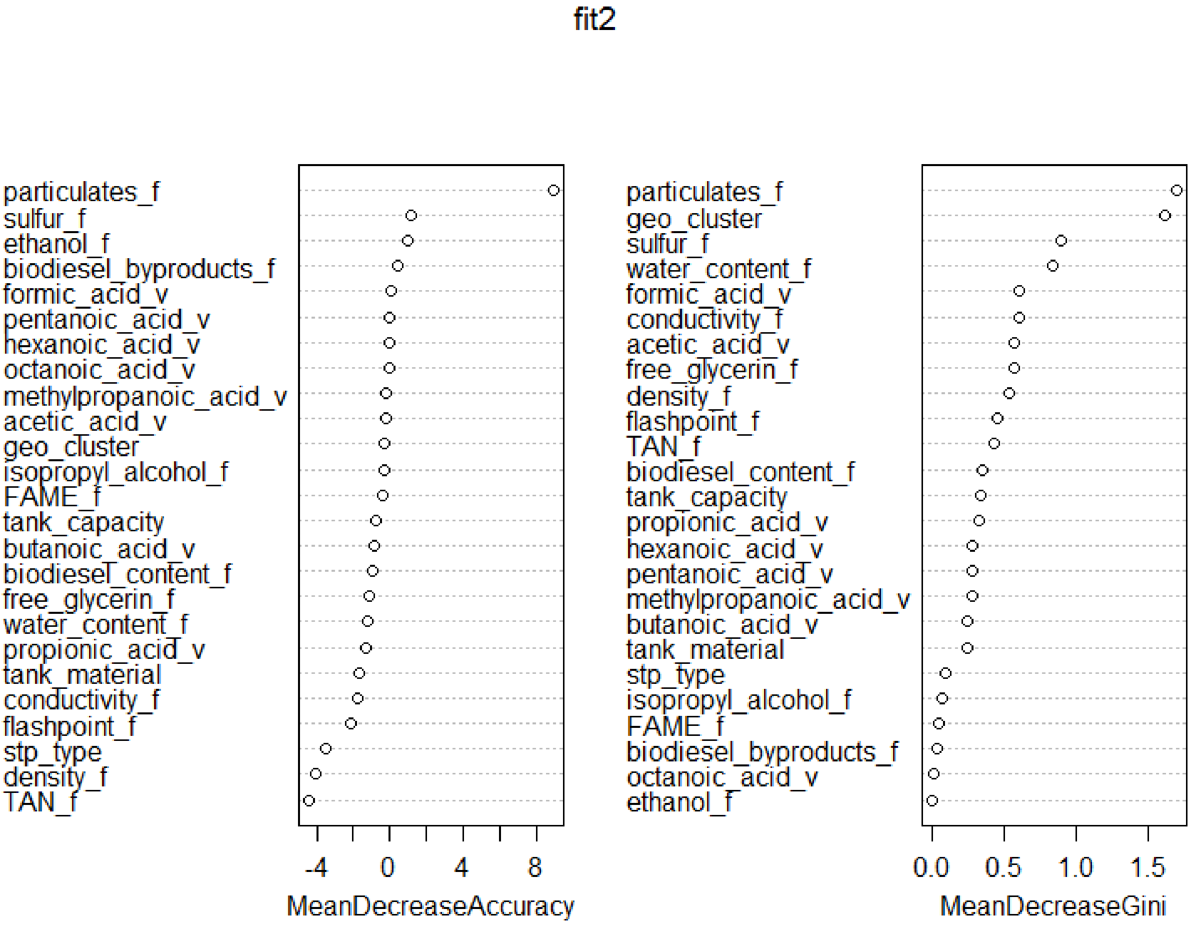
While tank capacity is not ranked highly in terms of variable importance scores from the random forest model analyses, when entered as a categorical covariate with eight levels representing different sizes of tanks, tank capacity is close to significant at 0.059, which is just outside the 0.05 significance level. However, the p-values for each individual level are all greater than 0.16 with large standard errors due to the small cell sizes in each. As a continuous covariate, it is also not significant with a p-value of 0.445 from the ordered logistic regression model.

We completed the same analytical approach when the data set included a throughput variable but was restricted to the 26 UST systems with throughput data available. The OOB estimate of error rate is 57.69 percent. This does a reasonably better job than chance at predictive accuracy and again finds particulates and water content near the top of the variable importance scores, with acetic acid measurements from vapor the third most important by both criteria. The results of the variable importance results and the univariate, ordered logistic regression are presented in Supplement D.



Corrosion Class	Predicted Class			Error In Predicting Class
	Minimal	Moderate	Severe	
Minimal	0	3	4	1
Moderate	2	9	6	0.471
Severe	0	7	11	0.389

Figure 10. Random Forest Analysis And OOB Accuracy Assessment Results Of Each Variable Evaluated Individually According To Three Corrosion Categories



Corrosion Class	**Predicted Class**		**Error In Predicting Class**
	Minimal	**Moderate + Severe**	
Minimal	0	7	1
Moderate + Severe	1	34	0.03

Figure 11. Random Forest Analysis And OOB Accuracy Assessment Results Of Each Variable Evaluated Individually According To Minimal And Moderate + Severe Categories

Table 7. P-Value Rankings From The Ordered Logistic Regression For The Variables For All 42 USTs

Variable	P-Value	Log Odds Estimate
tank capacity	0.059	NA
particulates_f	0.103	0.0125
free_glycerin_f	0.132	-0.0051
acetic_acid_v	0.133	0.0006
hexanoic_acid_v	0.134	-2.436
total_glycerin_f	0.242	-0.0045
owner	0.279	NA
biodiesel_byproducts_f	0.328	-0.5739
methylpropanoic_acid_v	0.353	-0.2700
stp_type	0.464	NA
water_content_f	0.516	0.0033
biodiesel_content_f	0.532	-0.0727
ethanol_f	0.562	-0.5635
formic_acid_v	0.564	0.0004
sulfur_f	0.571	-0.0759
butanoic_acid_v	0.643	0.1226
propionic_acid_v	0.653	0.0808
octanoic_acid_v	0.670	-0.8899
TAN_f	0.676	2.234
isopropyl_alcohol_f	0.728	-0.2033
geo_cluster	0.751	NA
conductivity_f	0.760	0.0004
density_f	0.792	-8.672
tank_material	0.882	NA
FAME_f	0.889	-0.0819
NACE_f	0.919	NA
flashpoint_f	0.990	-0.0008
pentanoic_acid_v	0.991	-16.42

NA - Not Applicable

Total Acidic Hydrogen Analysis

In an alternate statistical analytical approach, we used the total acidic hydrogen in micromoles per liter, which is designated as AcidH_mml in the analysis, as an outcome measure with corrosion category as the predictor in an ANOVA. Since this quantity is a concentration, it was first log-transformed to better satisfy the normality and constant variance assumptions of the ANOVA model. Indeed, after examining the normal probability, quantile plots, residuals, and Bartlett's test for equal variances from the natural scale analysis, these assumptions were not satisfied. The probability and quantile plots showed deviations from normality near the center and tail of the distribution, and the residual plots and Bartlett's test (p=0.01) showed evidence of a lack of constant variance across corrosion categories. These discrepancies were all alleviated after transforming to the log scale. The ANOVA results in Table 8 give an overall F-test p-value of 0.13, and the resulting contrasts and confidence intervals are also presented, along with the natural scale ratio of geometric means and associated 95 percent confidence intervals.

The variable importance plots in Figure 12 show that when included with the other variables considered, total acidic hydrogen ranks near the top in terms of value for classification. However, we did not perform a test to identify which, if any, of the acids is most significant of the total acidic hydrogen.

Table 8. Statistical Results Acidic Hydrogen Using ANOVA

Source	SS	df	MS	F	Probability > F
Between groups	6.753	2	3.377	2.14	0.1314
Within groups	61.57	39	1.579		
Total	68.33	41	1.666		

Log Scale Contrast And Confidence Interval (CI)					Natural Scale Contrast And CI For Ratio		
Corrosion Class	**Contrast**	**Std. Error**	**Scheffe 95% CI**		**Contrast Ratio**	**Scheffe 95% CI**	
Moderate vs minimal	0.1968	0.5643	-1.239	1.633	1.218	0.2896	5.118
Severe vs minimal	0.9381	0.5597	-0.4862	2.362	2.555	0.6150	10.62
Severe vs moderate	0.7413	0.4249	-0.3402	1.823	2.099	0.7117	6.188

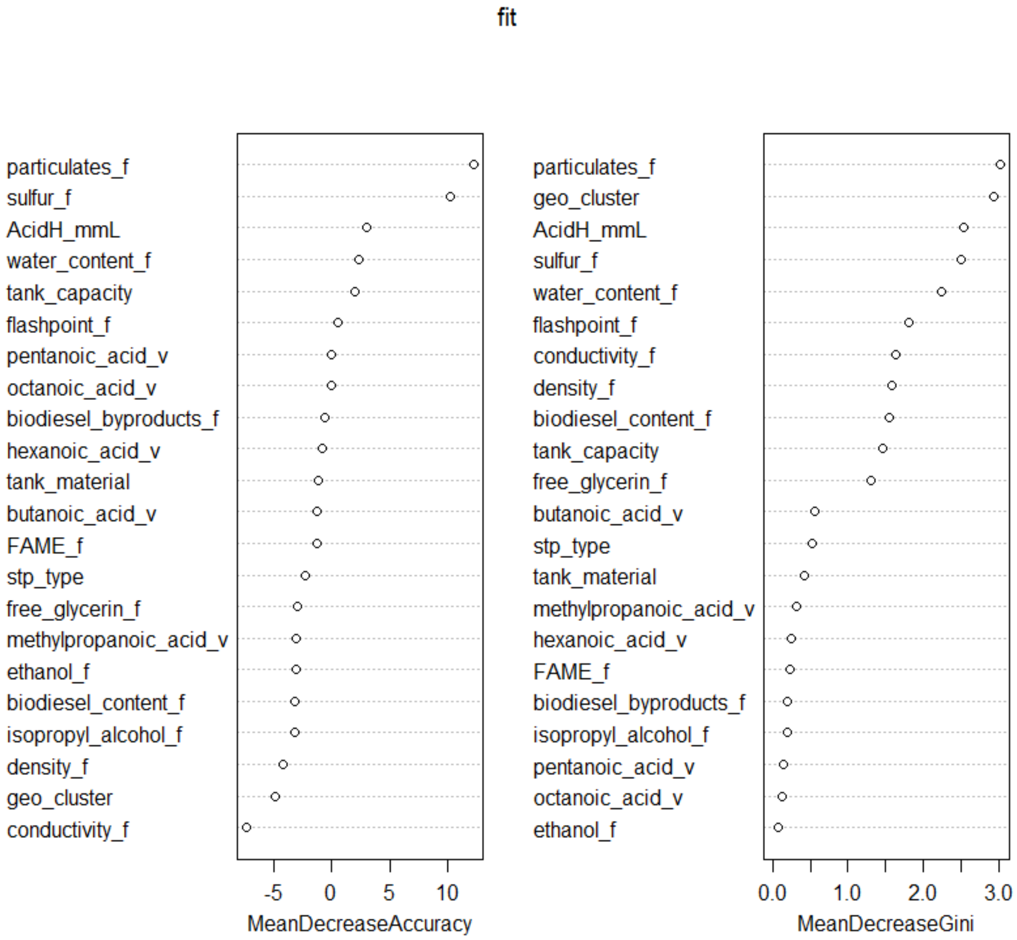


Figure 12. Random Forest Analysis Results Of Each Variable Evaluated Individually According To Three Corrosion Categories And Including The Acidic Hydrogen Variable

Chapter 5
Discussion Of Results

I. Corrosion Process

Metal corrosion is almost always caused by an electrochemical reaction. An electrochemical reaction is defined as a chemical reaction involving the transfer of electrons. During that transfer, there is a simultaneous oxidation reaction, which is also called an anodic reaction, and reduction reaction, which is also called a cathodic reaction. Electrons are produced in an anodic reaction and are consumed in a cathodic reaction. The number of electrons produced and consumed in any reaction must always be equal to maintain charge balance.

Corrosion is categorized as MIC when metals deteriorate due to the metabolic activities of microorganisms enhancing or accelerating the corrosion reaction. In the MIC process, any bacteria present do not directly participate in the electrochemical reaction—hence the term microbiologically influenced corrosion. In our research and the underlying assumptions, the role of MIC was assumed based on prevailing theories and previous research. The results of our research could not rule out MIC under the previously suggested hypotheses as a factor in the corrosion. However, in our research we did not perform a microbiological analysis that could prove bacterial contributions to corrosion.

In order to understand why corrosion is occurring in a specific environment, such as an UST system storing diesel fuel, we need to understand the relationships of the factors involved in the corrosion processes. One factor needed for corrosion in liquids and vapor phases is the presence of an electrolyte, which is defined as a substance in solution that conducts ions. It is the transfer of ions that participates in aqueous corrosion reactions. The higher the concentration of ions, the higher the conductivity of the solution, which can determine the limits of the corrosion rate. Specifically, corrosion-inducing factors for UST equipment include the following:

- a surface that is susceptible to corrosion, such as UST equipment,
- an electrolyte from chemical or biological processes,
- a distribution mechanism for the electrolyte to be transferred to the surface, and
- the pH of the aqueous component of the electrolyte.

Susceptible Surface

Equipment parts used in the UST are made from a variety of materials such as polymers and other metals. One of the key observations used in our research was the relative corrosion seen on the STP shaft within the UST. There are a couple of different vendors for these pumps, but they use several common metals and polymers in their materials of construction, including cast aluminum, wrought aluminum, brass, steel, grey cast iron, black steel pipe, and stainless steel such as Type 304. The polymers include Buna-N, Viton, and polyoxymethylene (POM).

Each of the metals comprising UST system tanks and equipment has its own distinct electrochemistry and corrosion susceptibility, depending on the specifics of the environment.

Additionally, some components may receive nickel coatings for corrosion resistance, while others may be coated with various epoxy, enamels, varnish rust inhibitors, or lacquer topcoats.

Electrolyte

The ingredients for an aggressive electrolyte from either a chemical or biological process exist within the UST systems inspected for our research. Specifically, available water, oxygen, and acids can create an environment that we expect would have the potential to attack most of the metals used in USTs. Water can be present in the vapor or condensed as a liquid along with other fuel or contaminant components on the inner walls of the UST, entrained in the diesel, or collected at the bottom of USTs. Minimizing water presence is and has always been an important part of UST maintenance. However, diesel blended with biodiesel can hold in solution more water than diesel without a biodiesel component. This means more water is likely arriving in USTs entrained in fuel today since biodiesel is more common in diesel than prior to 2007.

Oxygen is readily available from the atmosphere since diesel tanks are vented to the atmosphere. When fuel is delivered, the entire contents of the tank, both liquid and vapor, are disturbed and the contents of the vapor space are pushed into the environment. When fuel is dispensed, fresh air is pulled into the tank. In an UST, intake of ambient air can result in the vapor space above the product level being very humid, with condensation often occurring.

The previous research efforts discussed earlier in this report suggest that moisture and oxygen rich environmental characteristics of an UST system can support the growth of microorganisms. In this process, microorganisms can influence corrosion in USTs by producing acids; several industry sources recommend limiting water and performing other actions to prevent or limit active microbial growth in order to minimize corrosion in USTs storing diesel. Whether present due to a biological or chemical process, acids were present in the vapor spaces of all of the 42 USTs systems inspected. Therefore, all three of the ingredients for an aggressive electrolyte were present in all of the 42 UST systems inspected. Other processes that cause corrosion could also be more impactful under these environmental conditions.

Distribution Of The Electrolyte

The distribution of the electrolyte and the mode of contact between a metal and its environment have direct bearing on corrosion development. For example, three distinct regions within the USTs are represented along the STP shaft: always in liquid, always in the vapor, and the intermediate region that is sometimes in the liquid and sometimes in the vapor space. Metals can be constantly exposed to water as a bulk liquid electrolyte or as thin electrolyte layers with wetting and drying cycles in the vapor space, depending on where they are located in the tank. The vapor space at the top of the tank theoretically does not see liquid fuel but exists at relatively high humidity and with condensation. The tank bottom is constantly submerged and may contain water that drops out of the diesel or water that enters the tank from ambient air intake and condenses on the walls of the UST. Finally, an intermediate region, which depends on the product level in the UST, can be either submerged or in the vapor space.

Each region experiences a different set of conditions that directly influence the type and extent of corrosion development. In the vapor space, corrosion could occur under thin electrolyte layers from a high relative humidity or from the condensate, readily available oxygen, and presence of acids in the vapor space. When the fuel level drops as fuel is dispensed, an intermediate region that was previously submerged in fuel effectively becomes exposed to conditions that simulate the vapor space (and to residue and contaminants in the fuel that may be left behind). In these regions that experience wetting and drying cycles, the variation in thickness of the electrolyte results in a change in the corrosion rate.[54] When we included in the analysis fuel throughput data available from 26 of the UST systems, no statistical significance was indicated for fuel throughput; however, the distribution of the low molecular weight acids and the mode of contact are mechanically dependent on how often the UST environment is disturbed by changes in fuel level through delivery or dispensing of fuel. These disturbances also allow for additional water, oxygen, and carbon sources to enter the UST in the new fuel or ambient air that is drawn in when fuel is dispensed. This continual resupply of ingredients necessary to some microbial life could enhance the rate of MIC or other corrosion mechanisms. While metals in those areas most often exposed to the vapor space of USTs appeared to be most susceptible to heavy corrosion, anecdotes suggest all of the metal equipment in all three regions of USTs are susceptible to corrosion when the right conditions are present. Such corrosion of any metal component in USTs could potentially lead to a failure of functionality of the equipment and an increased risk of release of fuel to the environment.

pH Of The Electrolyte

The pH of either the bulk liquid electrolyte or the thin electrolyte layers in the vapor space will also impact the degree of corrosion on various exposed UST equipment alloys. For example, for carbon steel such as the tank, the corrosion rate is relatively low in aqueous solution, around a couple of thousandths of an inch per year, when the pH is between 3 and 9; however, below 3 it dramatically increases, and above 9 the corrosion rate decreases. See Figure 13.[55] In the case of aluminum such as the drop tube, however, the corrosion rate in aqueous solution will increase below 4 and above 8.5, as illustrated by the Pourbaix diagram of electrochemical potential-pH equilibrium for corrosion susceptibility of aluminum; see Figure 14.[56]

The water pH may have contributed to corrosion reactions. In all tanks where we detected measurable water, the pH values were between 3.4 and 6.2, which are favorable values for the growth of some acid-generating microorganisms but not always indicative of corrosion as seen in Figures 13 and 14. The bacteria identified and hypothesized as the leading species in MIC in CDFA’s 2012 study, *Acetobacter*, exhibit optimal growth under moderately acidic pH of 5.2 to 6.5 conditions and are capable of growth at pH 4.5.[57] The water in four of the five severely corroded tanks in our research had pH at or above 4.5; the fifth had a pH of 4.1. In contrast, water from five of the six minimally or moderately corroded tanks had pH at or below 4.5. The pH is an important indicator of the corrosivity of the electrolyte adjacent to a metal surface, but the presence of other contaminants that can allow corrosion to proceed at pH values normally not associated with susceptibility to corrosion is important.

The actual corrosion rate on a metal will also depend on other factors, mainly the presence or absence of oxygen and contaminants such as chlorides, sulfides, other chemicals, and particulates, which may be found in UST environments. These factors can disrupt protective films on the metal surface, thereby allowing corrosion reactions to proceed at intermediate pH values.

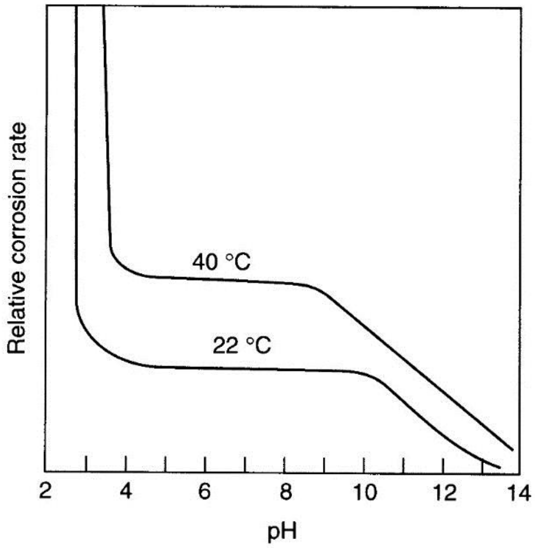


Figure 13. Relative Corrosion Rate Versus pH And Temperature Of Carbon Steel In Water[58]

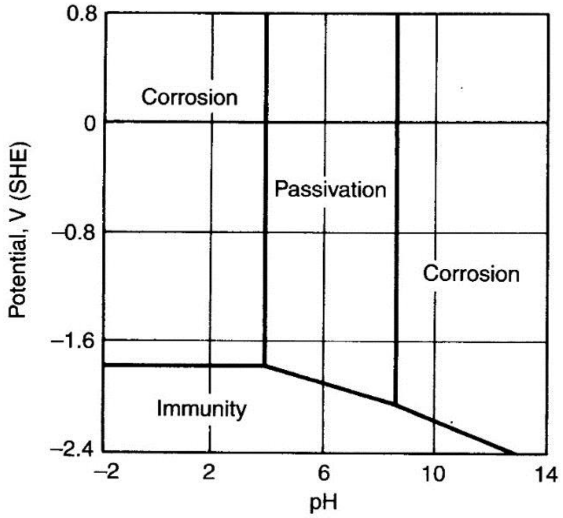


Figure 14. Potential Versus pH Equilibrium Diagram For Aluminum (vertical axis, standard hydrogen electrode (SHE); horizontal axis, pH)[59]

II. Feasibility Of Previous Hypotheses About Biofuels And MIC

Although no microbial analyses were performed in our research, the inspections and chemical examinations conducted as part of a search for predictive factors tested the feasibility of previously suggested hypotheses about MIC and biofuels. We evaluated observations and data from the UST sample population to identify if a suitable environment was available for microbial growth and if potential evidence of microbial metabolism pathways suggested by previous research could be identified or could be correlated with corrosion presence. The results cannot prove or disprove those theories of MIC in which microorganisms oxidize biofuel components found in diesel fuel.

For MIC to occur, the presence of water is required for microbial growth. An aqueous phase with sufficient water present to allow sampling for chemical analysis was collected from 11 tanks. We rated five out of 11 tanks from which water was collected as severely corroded. However, the statistical analysis did not determine that the presence of free water in the USTs corresponds with any particular corrosion rating. We observed dissolved water in fuel over the allowable standard specification limit in 10 percent of samples, although the water would likely need to separate from the fuel into an aqueous phase for most microorganisms to use it.

MIC typically occurs in two environments: aerobic and anaerobic. In aerobic environments, the corrosion reactions are electrochemical with multiple reactions that are mediated by many organisms. Localized ion concentration cells may form with aerobic biofilms, as well as concentrations of acid-producing bacteria. In aerobic environments, the biotic and abiotic corrosion processes enhance one another and are difficult to distinguish.[60] Another process in aerobic environments is tubercle formation, in which cathodic oxygen reduction occurs at the tubercle base and anodic iron dissolution occurs underneath the tubercle. Once initiated, tubercle formation results in sustained reducing conditions within the tubercle despite the overall aerobic environment.[61]

In anaerobic environments, MIC tends to be dominated by sulfate-reducing bacteria. Differential aeration cells form by oxygen depletion under microbial surface films.[62, 63] In adjacent aerobic environments, sulfur oxidizers such as *Thiobacilli* can convert hydrogen sulfide if present to corrosive sulfuric acid.[64]

An indication of MIC suggested by previous research was the presence in the aqueous phases of alcohols or certain acids, which would be consistent with bacterial oxidization of those alcohols. In our research, we found such acids in many tanks. All the aqueous phase samples had an acidic pH and all contained ethanol, as well as volatile low molecular weight organic acids. Previous research suggested these acids are likely contributing to a corrosive environment in the USTs.[65] For example, in UST system 18-TN-ST, ethanol was present in the water phase at a concentration of 7 percent. Ethanol is likely present from gasoline ethanol-blend contamination entering with the diesel fuel delivery or from diesel and gasoline USTs improperly manifolded together. Acetic acid can be generated directly from ethanol by acetic acid bacteria such as *Acetobacter*.[66] *Acetobacter* was the genus identified as most prevalent in the microbiological analyses in CDFA's 2012 study. In addition to ethanol, we identified glycerin in many of the tanks; glycerin can be fermented into propionic acid, glyceric acid, and other organic acids by acid-producing bacteria.[67, 68] Similarly, acetic acid, lactic acid, and formic acid could be products of glycerol utilization for bacteria from the *Enterobacteriaceae* family.[69] Many other bacteria may also may thrive in UST environments and may also contribute to MIC.

Biodiesel in fuel is indicated by the presence of fatty acids of typically 16- and 18-carbon lengths and FAME characteristic of commonly used vegetable oil and animal feed stocks. As such, shorter-chain-length fatty acids such as those in our research are likely not an initial component of the biofuel. Free fatty acids can also represent the decomposition of FAME into its primary components by hydrolysis of the ester chemical bond. Hydrolysis of biodiesel occurs relatively slowly at neutral pH; however, it is catalyzed in the presence of acids, and the formation of acids further accelerates the decomposition reaction. Other residual constituents in biodiesel include mono-, di-, and triglycerides (more commonly referred to as animal or vegetable oils and fats), which also represent a source of glycerol in the form of glycerol esters as differentiated from free, unbound glycerol. Although there is an allowance within the specification for mono-, di-, and triglycerides, data indicate that the acidic environment in USTs may contribute to the glycerol concentration approaching and exceeding the specification.

As noted previously, the presence of glycerol in diesel can suggest a source of residual glycerin from the biodiesel component. Since ASTM 6584 allows for maximum concentrations of 0.02 percent free glycerol, and glycerol would selectively partition to the water phase of a biodiesel-water mixture, relatively high glycerol concentrations (for example, >2,000 ppm) in the water phase are theoretically achievable when using an octanol-water partition coefficient for glycerol of 0.017 and assuming partitioning would be similar in a water-biodiesel system. It can also be insightful when an anticipated component is not present, or is not present at the level commensurate with other related components (for example, biodiesel FAME, free fatty acids, and glycerol) based on historic levels or fixed chemical relationships. This may indicate that a particular component has undergone degradation or been converted by a chemical or biological process to another compound. For example, free fatty acids, sources of energy for microorganisms, can be metabolized, producing fatty acids of successively shorter chain lengths. Thus, if the glycerol measurement is low and the three organic acid measurements are high, the acids could have been produced by the degradation of the glycerol. However, other processes or combinations of processes could also have a similar result.

Vapor measurements from the 42 tanks showed both acidic and high moisture content conditions, which are conducive to corrosion development. The presence of acetic, formic, and propionic acids could indicate that MIC, where bacterial populations feed on ethanol and glycerol as suggested by previous research, could be occurring as part of the many microbial processes occurring in the tanks. The most volatile among the detected organic acids, such as formic acid or acetic acid, were found in the vapor phase of each tank. We observed the highest concentrations of organic acids in the vapor phase of severely corroded tanks.

The data collected could not disprove the MIC hypothesis suggested by previous research, but microbiological analysis would be required to confirm that these processes were taking place. However, numerous processes are occurring at the microscopic level. Attributing microbial activity to only a handful of bacteria is not supportable by the evidence collected in our research. If MIC in which certain bacteria feed on biofuels is occurring as suggested by previous research, it could be a contributor to the corrosion in USTs, but may be occurring alongside other species actively involved. The role of other corrosion influencing organisms such as fungi, archaea, or eukaryotic organisms cannot be determined by these results. In general, it appears that MIC is likely happening in USTs storing diesel, and taking action to limit the environmental conditions necessary for microbial growth is recommended by multiple industry groups and anecdotally appears to be successful in minimizing the chances of severe corrosion in USTs. Future research by CRC or others may help to further understand the feasibility of the hypotheses of biofuels' role in MIC.

Chapter 6
Key Takeaways

EPA's research determined that corrosion is very common; it appears all USTs storing diesel could be susceptible to developing corrosion. We could not identify predictive factors that pinpointed a cause, but our results suggest that previous research by EPA and others hypothesizing MIC remains a likely possible contributor. However, MIC is likely not the only contributor. We noted several key observations, discussed below, that may help prevent releases of diesel from UST systems.

Corrosion Of Metal Components In UST Systems Storing Diesel Appears To Be Common

Based on the inspections of the 42 UST systems evaluated in 10 clusters around the United States, severe corrosion of metal components in the vapor space of USTs storing diesel appears to be a common occurrence. Approximately 83 percent of the UST systems – or 35 of 42 – were experiencing moderate or severe corrosion of metal components; 18 UST systems had severe corrosion and 17 had moderate corrosion. This sample is biased because EPA specifically looked for UST systems experiencing corrosion; however, less than 25 percent of the USTs in the study reported knowledge of corrosion prior to the site investigation. Only seven of 42, or 17 percent, of inspected tanks were classified with minimal corrosion. However, even some of the USTs classified with minimal corrosion showed some corrosion, suggesting that they may be at the beginning stage of increased corrosion. The true prevalence of severe corrosion of metal components across the entire nationwide population of UST systems storing diesel, including UST systems storing fuel for emergency generator systems, could be higher or lower than observed in our research. The number of tanks storing diesel fuels potentially affected by severe corrosion and potentially at an increased risk of releasing fuel to the environment – including ASTs, home-heating oil, and smaller, unregulated USTs – is many times larger than the estimated 100,000 federally-regulated USTs storing diesel fuel.

Many Owners Are Likely Not Aware Of Corrosion In Their Diesel UST Systems

Less than 25 percent of our UST sample population owners reported that they were aware of any corrosion in their UST systems. But we found moderate or severe corrosion in 83 percent of the USTs. It appears most owners were not aware of the corrosion. This may be the case across the country for many owners of UST systems storing diesel fuel. An owner may be able to identify severe corrosion problems easily if it affects fuel flow, but without proactive best management practices and visual inspection, an owner may be unable to identify the onset of the problem before equipment becomes corroded and potentially compromised.

The Corrosion Is Geographically Widespread, Affects UST Systems With Steel Tanks And With Fiberglass Tanks, And Poses A Risk To Most Internal Metal Components

Corrosion coverage on metal components of USTs was present in both UST systems with fiberglass tanks and UST systems with steel tanks; the corrosion was not limited to specific geographic regions. Our observations suggest that metal components in the vapor space of tanks are most susceptible to this corrosion, and severe cases showed corrosion of metal equipment in lower wetted portions of UST systems as well. Corrosion in aqueous phase contact areas of steel tanks has been a known risk for decades, but has generally been prevented with good maintenance practices that focus on limiting water in USTs.

EPA's research initially focused on investigating only corrosion reported in vapor spaces of tanks. However, we heard during the course of our research from several companies that they now routinely remove significant quantities of sludge from the bottoms of USTs storing diesel; we also heard several anecdotes of pinhole corrosion through tank bottoms. Corrosion in the bottoms of steel tanks where water or sludge could collect has been a known risk for decades, but generally has been prevented with good maintenance practices. Historically, internal corrosion failure in UST bottoms has been very rare or undocumented.

At least three of the aqueous phase corrosion anecdotes came directly from UST regulators in three different states. All three discussed cases of metal tank bottoms, with one regulated tank an AST, which apparently failed from corrosion from the inside of the primary wall of double walled tanks storing diesel fuel. Other anecdotes of probably corrosion-induced failure in double walled tanks have been reported. In many cases, an investigation was prompted when owners learned via an interstitial alarm that there was fluid in the interstitial space between the walls. In some cases, it appears other leak detection equipment did not identify any release of fuel, but the leak detection equipment appeared to be working properly. Therefore, it appears that pinhole leaks from tank bottoms could slowly develop and leak for a period of time, with the leak below the threshold for some leak detection equipment. In these anecdotes, no fuel released to the environment because the fuel was contained by the secondary wall of double walled tanks. However, the anecdotes suggest that if similar corrosion occurred under sludge or in an aqueous phase in the bottoms of single wall tanks, the tanks could potentially slowly leak undetected under the threshold for leak detection, if no interstitial space exists to identify the leak.

The type of corrosion observed in aqueous phases may or may not appear the same as the visual corrosion seen in the vapor spaces during internal tank inspections; corrosion in vapor spaces is often described as having tubercles and extensive surface coverage. Instead, aqueous phase corrosion under water or sludge may be of the pinhole or burrowing corrosion presentation that generally does not appear with tubercles or cover large amounts of surfaces. However, corrosion of this kind could also occur in the vapor space and remain undetected without a detailed examination or test. Overall, it appears from our research and anecdotes from industry and regulators that corrosion of metal components either in the vapor or fuel or aqueous phase may be more prevalent than prior to 2007, and this presents an elevated risk to the integrity and functionality of all internal metal components in UST systems. Shear valves, overfill valves, ball floats, risers, ATG probe shafts, tank bungs, STP shafts, and other metal components in the vapor space of fiberglass and steel tanks may be particularly susceptible to functional failure due to corrosion.

Ethanol Was Present In 90 Percent Of 42 Samples, Suggesting Cross Contamination Of Diesel Fuel With Ethanol Is Likely The Norm, Not The Exception

EPA identified ethanol in 90 percent of the fuel samples. We also identified C4-C8 carbon chains in all tanks; these carbon chains could represent gasoline contamination. It is reasonable to assume that ethanol's presence in diesel is at least partially due to diesel being delivered in the same truck as ethanol or gasoline-ethanol blended fuels.

The Quality Of Diesel Fuel Stored In USTs Was Mixed

We tested the diesel in each UST against several fuel specifications and guidelines in order to characterize the fuel quality at the end user point of the distribution process. Fuel may or may not be tested between the time it leaves the terminal and before it is delivered to the end user, but numerous opportunities exist for fuel to become off specification during the distribution or storage process. We compared the results of the samples tested in this population to a variety of specifications and guidelines to determine if the fuels are acceptable; our results were mixed. The presence of water, particulates and contaminants in the fuel, and acids in all of the water samples suggest that the fuel stored in many of our sample population USTs did not generally meet all the quality standards applied at different points in the production and distribution chain. If the same conditions observed in the collected fuel and water samples are representative of UST systems storing diesel across the country, we can assume that many UST systems may be storing fuel that is less clean and dry than many standards intend.

Particulates And Water Content In The Fuel Were Closest To Being Statistically Significant Predictive Factors For Metal Corrosion, But Causation Cannot Be Discerned

Although EPA did not identify any statistically significant predictive factors, it appears that USTs with cleaner and drier fuel could be less likely to be associated with severe corrosion issues. All corrosion assessments and data collected were analyzed statistically for predictive accuracy in identifying UST corrosion severity categorized as minimal, moderate, or severe or alternatively as with corrosion (moderate + severe) or without corrosion (minimal).

According to the statistical analysis, factors that scored closer to being predictors of metal corrosion severity, albeit with low accuracy, are fuel particulates and water content in the fuel. It is important to remember that even if significant, the findings of these would not necessarily signal causation. Correlation could be possible. Many variables are interrelated and represent various portions of an ongoing process. However, industry generally agrees that one factor that would significantly reduce corrosion seen in the field is minimizing the presence of water in USTs.

MIC Could Be Involved As Hypothesized By Previous Research

The data do not contradict the hypotheses suggested by CDFA's 2012 study and EPA ORD's research that MIC is occurring in USTs when bacteria oxidize biofuels or residuals from biodiesel production found in diesel fuel. Previous research suggested that acid-producing bacteria, including those of the genus *Acetobacter*, and other acid-producing bacteria, can metabolize ethanol from switch-loading fuel delivery or biodiesel components of diesel, such as glycerol and FAME breakdown products. Our research could not verify or disprove the role of this specific bacteria, but there are numerous other types of bacteria that could also be consuming chemical components of the fuel or fuel contaminants found in USTs. In addition to bacteria, there are also a number of other microorganisms that could cause or contribute to the corrosion attacks, including fungi, archaea, and eukaryotic organisms. Although other mechanisms of corrosion are possible, MIC is likely playing a major role in the corrosive environments in USTs storing diesel. Preventing and treating MIC is recognized throughout industry as a standard recommended approach to maintaining UST systems.

Impacts, Recommendations, And Next Steps

The results of our research suggest that USTs storing diesel are susceptible to developing rapid and severe corrosion. It is possible that increased corrosion could lead to the potential for an increased risk of release due to failure of release prevention equipment, release detection equipment, or the UST itself. Aboveground storage tanks (ASTs) storing diesel are outside of the regulatory purview of EPA's Office of Underground Storage Tanks, but during the course of this research it has come to EPA's attention that ASTs and USTs may both be at risk of corrosion. Small, non-federally regulated USTs storing diesel fuels both aboveground and belowground are likely also at risk of corrosion.

Multiple approaches to addressing the corrosion in UST systems nationwide may be called for. Initially, awareness that the problem exists is imperative for owners, industry, and state and federal agencies so that owners know they should inspect their UST systems. Via our state and industry partners, EPA will notify UST owners about the findings of our research. The notice will share the findings with owners and recommend steps for identifying and minimizing corrosion risks in their UST systems.

Recommendation To Visually Inspect USTs Storing Diesel As Part Of Routine Monitoring

Our research on 42 UST systems demonstrates the wide variation of corrosion that can be seen in USTs storing diesel. The high percentage of the sample population experiencing moderate or severe corrosion suggests that a large percentage of USTs nationwide could be affected by this corrosion, so EPA recommends that owners inspect their USTs for corrosion. It is important to check visually from the surface when possible, but a more in-depth examination by internal photo, video, or manned access could also be helpful since corrosion may not be visible from visual observations when opening access points on the surface. Figure 9, UST 43-MD-ST, is an example. In that UST, noting the lack of corrosion in the fill riser is important, but using it as the sole indicator of the extent of corrosion in the UST system would be misleading because some of the equipment inside the tank had severe corrosion. Owners should be aware that visual inspections from the surface of only risers may lead owners to believe corrosion is not an issue in their systems, when in fact corrosion could be very advanced on other components.

Going forward, research should continue to foster further understanding of influencing factors and to find solutions. EPA provided our early research results to CRC to assist them as they considered a new phase of research that could attempt to pinpoint the cause or causes of corrosion development. Industry also is continually working to improve equipment and ensure functionality with various fuels on the market, including designing and offering equipment in corrosion resistant versions like stainless steel.

Enhanced UST system maintenance and routine inspections are immediate steps owners of USTs storing diesel can take to combat this issue. Vigilant aqueous phase monitoring and removal is a critical maintenance step; most owners of diesel tanks are already aware and may already be practicing this maintenance. In our research, the presence of water appears possibly correlated, albeit not statistically significantly, with an increased prevalence of corrosion. Industry documents suggest the removal of any water when found, possibly more often than owners have traditionally done in the past, should be considered to limit the potential for microbial growth in USTs storing diesel fuel. Owners should understand where water collects in their particular systems due to tank tilt and ensure water sensor equipment is monitoring at the most appropriate place. EPA suggests owners consult industry documents as sources of helpful information about limiting the potential impacts of corrosion.

Knowing the condition of equipment through routine inspections is key to identifying corrosion development over time. This could include looking at filters and filter housings for signs of debris or deterioration and removing the drop tube and ATG probes to assess whether automatic shutoff overfill devices and monitoring floats can function properly. In-tank video inspections, if available to the owner, are a proactive and useful way to visualize and document the conditions inside the tank, because visual observations from the surface may not always show the true extent of corrosion inside an UST. Addressing corrosion issues at early stages could reduce repair and replacement costs later, as well as reduce the risk of extremely expensive environmental release clean-up costs.

Below are several approaches, but not an exhaustive list, that may be helpful with limiting corrosion. These are available now or in development in the market.

- Regularly monitor and remove any water present in the UST
- Filter fuel for water and particulates before it is delivered into the UST or recirculate and filter water and particulates while it is stored
- Add nitrogen generating equipment to limit an oxygen rich atmosphere inside tanks
- Use liquid corrosion inhibitor additives or other corrosion inhibitors, including filming amines
- Use biocides to kill or other fuel treatments to disrupt existing microbial colonies or prevent future MIC

Research is ongoing in an attempt to pinpoint the cause of corrosion in USTs storing diesel and identify a solution. Until the cause and solution are identified, a combination of actions may be helpful. These actions include: education and outreach, additional investigations, preventive maintenance, adoption of corrosion mitigating products in the market, and potential modifications to fuel distribution and storage practices throughout the industry. These may help reduce corrosion and the risk of potential releases of fuel to the environment from USTs and other tanks storing diesel fuel.

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