Preliminary Investigation Summary of Deterioration of Fuel and Fuel Systems Storing and Dispensing ULSD Fuel at DFW Airport

In 2006, EPA limits on sulfur in petroleum products were implemented as a measure to control air pollutants. Prior to implementation, acceptable limits were 500ppm; after, less than 15ppm was acceptable by EPA standards. The consequence of a well-intentioned control on air pollution has been a host of issues affecting the supply chain from the terminal point to consumer. Since full enactment of the regulation, there have been increased fuel related issues; DFW Airport is one of many ULSD consumers experiencing ill effects[1,2]. Aviation and diesel fuel are particularly prone to microbial contamination as hydrocarbon chains in the range of C10-C18 are readily utilized as carbon sources for microorganisms.[3] Because this issue is widespread, and has the potential to lead to catastrophic failures of UST's; initial studies were conducted to determine if biodiesel fuel, ULSD in particular, is prone to cause excessive corrosion.

The suspected onset of microbial contamination in ULSD (Ultra Low Sulfur Diesel) tanks at DFW Airport has increasingly become cause for concern. Microbial contamination is known to clog fuel lines and filters, increase corrosion of metal components in fuel storage, and lead to deterioration of fuel products[4]. Consequences of corrosion related failure are not strictly limited to production loss, equipment deterioration, and increased O&M costs; an accidental release can impact human health & safety, and the environment. Recently an increased number of tank and mechanical issues have been reported and studies commissioned by API and NIST show a surge of microbial induced contamination, particularly in tanks storing ULSD (Ultra Low Sulfur Diesel) fuel.

As more studies investigating the relationship between fuel deterioration, corrosion, biodiesels, and ULSD emerged, the primary cause was ultimately linked to microbial contamination. Early studies point to higher incidences of bacterial contaminants and increased colonization present in ULSD samples, as compared to other biodiesels.[5] These studies determined, while biodiesels in general have a higher potential for corrosion, ULSD is particularly prone as the reduction of sulfur and other aromatics have removed biocide-like qualities that previously existed in fuel.[6] Results of studies like these prompted an investigation by the petroleum industry and studies were commissioned to identify bacterial and/or fungal species present in contaminated tanks and detail mechanisms by which corrosive conditions prevail. The Battelle Institute study is highly cited by professionals in the petroleum community, it provides the mechanism by which excessive corrosion occurs, and parameters highlighted in the study can be compared to current conditions of suspected contaminated tanks.

The environmental implications of excessive corrosion include soil and groundwater contamination from compromised tanks and increased waste generation. From an operational and fleet management standpoint this issue has the possibility of increasing financial cost, increasing servicing downtime of fleet and emergency response vehicles and generators. We are not compelled by regulatory compliance to address this issue, but an aggressive approach is necessary to curtail contamination and halt excessive corrosion of components of the system.

Current Conditions

In 2011, in an effort to comply with EPA regulations, DFW Airport phased in the use of ULSD fuel. Though ULSD is used and stored throughout the airport, our concerns are primarily focused on underground fiberglass tanks used for ULSD storage. Soon after implementation of the required fuel blend, dispenser related issues were reported at fueling stations DPS #2, DPS #3, and DPS #4 (Figure 1). The dispenser filtration components are generally found to be clogged and cease passing fuel. In order to restore service the dispenser filter screen is removed and solid debris similar in appearance to coffee grounds with large gelatinous matter in the matrix is removed from the dispenser filter. Maintenance logs show a consistent need to clean pump filters and in the case of DPS #2 and DPS #4, installation of replacement suction pumps was necessary (Table 1). ETAM initiated a tank cleaning at DPS#2 to remove the accumulation of solids. This process alone did not reduce the frequency of dispenser pump clogging.

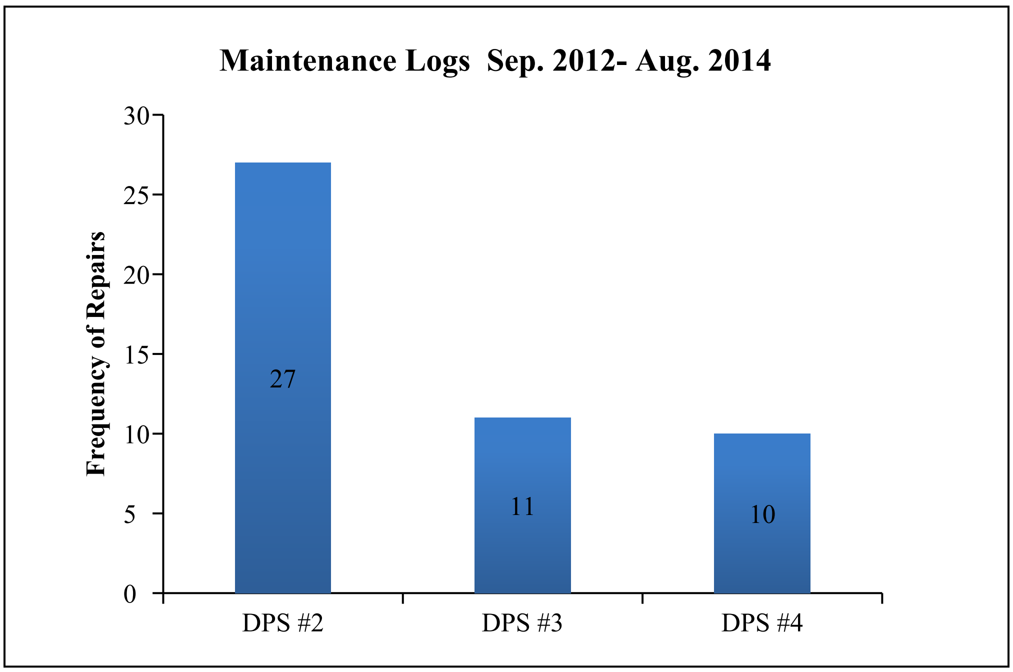

Figure 1: Frequency of repairs by station. Sept. 2012- Aug. 2014.

Location:	Date:	Type of work being performed:
DPS #2	9/20/2012	Dispenser Filter replacement
DPS #3	9/20/2012	Dispenser Filter replacement
DPS #3	12/7/2012	cleaned pump filter screen, both dispenser filters replaced
DPS #2	12/27/2012	Dispenser Filter replacement-1 of 2
DPS #3	12/28/2012	cleaned pump filter screen, both dispenser filters replaced
DPS #4	4/1/2013	Dispenser Filter replacement
DPS #4	4/1/2013	cleaned pump filter screen
DPS #2	12/3/2013	dispenser pump replaced
DPS #2	12/11/2013	cleaned pump filter screen, both dispenser filters replaced
DPS #2	12/31/2013	one dispenser filter replaced
DPS #4	1/8/2014	both dispenser filters replaced
DPS #4	1/10/2014	cleaned pump filter screen
DPS #2	1/17/2014	one dispenser filter replaced, cleaned pump filter screen
DPS #2	1/24/2014	cleaned pump filter screen
DPS #2	2/7/2014	cleaned pump filter screen
DPS #2	2/13/2014	tank cleaning project completed + cleaned pump filter screen, both dispenser filters replaced
DPS #2	2/20/2014	cleaned pump filter screen
DPS #2	3/13/2014	cleaned pump filter screen X 2, both dispenser filters replaced
DPS #2	3/17/2014	cleaned pump filter screen
DPS #2	3/31/2014	cleaned pump filter screen
DPS#2	4/14/2014	cleaned pump filter screen X 2, both dispenser filters replaced
DPS#2	4/16/2014	cleaned pump filter screen
DPS#2	5/2/2014	cleaned pump filter screen
DPS#2	5/13/2014	cleaned pump filter screen
DPS#2	5/29/2014	cleaned pump filter screen
DPS #3	6/3/2014	cleaned pump filter screen, both dispenser filters replaced
DPS #3	6/6/2014	dispenser pump replaced
DPS #4	6/9/2014	cleaned pump filter screen
DPS#2	6/12/2014	cleaned pump filter screen
DPS #2	6/19/2014	cleaned pump filter screen X 2, both dispenser filters replaced
DPS#2	6/24/2014	cleaned pump filter screen
DPS#2	6/27/2014	cleaned pump filter screen
DPS#2	7/1/2014	cleaned pump filter screen
DPS#3	7/1/2014	cleaned pump filter screen
DPS#2	7/11/2014	cleaned pump filter screen
DPS#3	7/11/2014	cleaned pump filter screen
DPS #4	7/14/2014	cleaned pump filter screen
DPS #4	7/21/2014	cleaned pump filter screen; dispenser suction pump needs to be replaced
DPS#4	7/24/2014	both dispenser filters replaced
DPS#2	7/25/2014	cleaned pump filter screen
DPS#3	7/25/2014	cleaned pump filter screen, both dispenser filters replaced
DPS#2	7/31/2014	cleaned pump filter screen
DPS#3	7/31/2014	cleaned pump filter screen
DPS#3	8/5/2014	cleaned pump filter screen
DPS#4	8/6/2014	dispenser pump replaced
DPS#2	8/11/2014	cleaned pump filter screen
DPS#4	8/12/2014	cleaned pump filter screen

Table 1: Type of maintenance performed at affected stations. Sept. 2012 – Aug. 2014

As dispensers failed to flow with increasing frequency, concerns arose that the solids generated were the byproduct of excessive corrosion of the steel and fiberglass components in affected tanks. In order to diagnose the dispenser issue, samples were collected during the initial cleaning of DPS #2. The samples were taken from gross solid filtration, centrifuge solids, centrifuge water and fill port scrapings. Analysis was performed by Xenco labs (Appendix II). The fill port scrapings were collected due to discoloration and of brass material on the fill port. The results are absent microbial analysis; however they do give us insight into chemical processes occurring in our tanks and suggest the influence of microbial induced contamination (MIC).

Field inspections of accessible equipment including the fill port and suction tube display conditions consistent with excessive corrosive conditions. When the suction tube was removed the corroded coating was estimated to be 1/8" to ¼" thick (Figure 2). The filter screen at the bottom of the suction tube could not be easily removed because screws connecting the screen to the pipe were so heavily corroded. Screens and filters are especially prone to contamination as they provide a collection area with the capacity to collect both water and matter. The presence of significant amounts of precipitated materials suggests the presence of microbes. The red- brown precipitates reflect the presence of iron or iron hydroxides, or both.[7]

Figure 2: The suction tube column. This is the upper length of the pipe. Note the blue discoloration of brass (indicative of exposure to acid). This photograph was taken after removal of 1/8" to ¼" thick precipitated material.

Examination of the top of the suction tube where it attaches to the sump at the surface revealed copious amounts of red-brown solids (Figure 3). This is the highest point in the vapor space and has relatively little or no contact with fuel. The significance of this finding suggests the vapor space of the tank is acidic. The pH of tank water sample was <4, an ideal pH for proliferation of acid resistant species. These findings are consistent with studies, including the Battelle Institute and API, which conclude acidic vapor space is a metabolic byproduct of microorganisms present in ULSD fuel storage systems[8]. Fill port photographs illustrate oxidation of brass components of the fill port (Figure 4). The blue-green discoloration indicates an acid reacting with zinc and copper. Copper and zinc are primary components of brass.

Figure 3: The suction tube attachment to the sump. This is the highest point in the system with relatively little or no contact with fuel. This piece has no exposure to outside environment.

Figure 4 : Brass fill port at DPS #2. The blue-green precipitate is the result of an oxidation-reduction reaction in the system

DPS #2 - Fill Port Scrapings (479270-003)	Result	RL
Aluminum	178	99.3
Calcium	526	199
Cobalt	20.3	19.9
Copper	**295000**	**19.9**
Iron	2230	59.6
Lead	28400	23.8
Magnesium	1360	19.9
Manganese	34.7	19.9
Potassium	2960	993
Zinc	**11900**	**19.9**

Table 2: Elevated levels of copper and zinc point to an oxidation- reduction reaction occurring in the vapor space of the fuel tank.

Laboratory analysis of generated solids has confirmed the presence of increased iron in the fuel systems of DPS #2. Elevated iron is a byproduct of excessive corrosion but it's also a physiological requirement for organisms more specifically; microorganisms consume insoluble iron and convert it to soluble iron. Studies show Fe (II) can function as an electron source for iron-oxidizing microorganisms under both oxic and anoxic conditions and Fe (III) can function as a terminal electron acceptor under anoxic conditions for iron-reducing microorganisms. This shows a complex and versatile relationship between Iron and microorganisms.[9] According to ASTM standards the presence of increased soluble iron strongly suggests bacterial activity and changes in aluminum and manganese are indicative of corrosion[10].

DPS #2 - Centrifuge - Solid (479270-002)	Result	RL
Aluminum	**1060**	**99.3**
Barium	44.7	4.72
Cadmium	51.2	2.36
Calcium	**518**	**4.72**
Chromium	49.8	2.36
Copper	219	4.72
Iron	**54100**	**14.2**
Lead	72.7	5.66
Magnesium	94.2	4.72
Manganese	**222**	**4.72**
Nickel	32.2	4.72
Sodium	**429**	**236**
Zinc	522	4.72

Table 3: Results of centrifuged solids collected from DPS #2. All results are elevated but highlighted elements are specifically indicative of biological activity/microbial contamination.

Microbes require water and nutrients in order to successfully propagate within the system. Nutritional requirements can be divided into two major groups, macro-nutrients and micro-nutrients. Macro nutrients available in the fuel include carbon, hydrogen, oxygen, nitrogen, sulfur and phosphorus; micro nutrients include calcium, sodium, potassium, iron, magnesium, manganese, copper, cobalt and nickel.[11] Analysis of both pre-centrifuged and centrifuged solids collected during the initial DPS #2 tank cleaning, confirm elevated levels of calcium, sodium, iron, magnesium, manganese, copper, and nickel; potassium and cobalt were not elevated in these samples. These results reveal the requisite nutrients needed to support growth and proliferation is abundant. Also, the presence of caustic salts such as Na and Ca are known to contribute to accelerated corrosion of metal products.

DPS #2 - Pre-Centrifuge Solid (479270-001)	Result	RL
Aluminum	**136**	**25**
Antimony	7.45	5
Barium	38.7	5
Cadmium	17.5	2.5
Calcium	**504**	**50**
Chromium	31	2.5
Copper	51.6	5
Iron	**233000**	**15**
Lead	11.7	6
Magnesium	200	5
Manganese	**1410**	**5**
Nickel	23.1	5
Thallium	36.5	5
Zinc	467	5

Table 4: Results of pre-centrifuged solids. All elevated results are reported in this tables are consistent with excessive corrosion but highlighted elements are also consistent with biological activity.

The Battelle Institute Study identifies *Acetobacter acetii* as the main constituent leading to corrosion in fuel and fuel systems; it is one of many in the family Acetobacteraceae. This genus is characterized as gram negative, aerobic, acid resistant/loving species capable of oxidizing ethyl alcohol to carbon dioxide. Rarely is a single species the cause of biodeterioration but a complex network of communities' within regions of biofilm found on tank roofs, shells, and at the fuel water interphase. The presence of aerobes and facultative aerobes within the biofilm consortium can provide nutritional requirements and protection within a mucous like polymer to species which would not normally propagate within the system, including anaerobic species known to acidify vapor space in tanks[12,13]. The dispersion of acids throughout the system is attributed to higher vapor pressure of weak acids, such as acetic acid, compared to that of ULSD[14]. The characteristics of the biofilm community largely differ from characteristics of an individual species within the system; but changes in the system are predictable and include increased acidity, the presence of accumulated solids, increased water content, the presence of low molecular weight acids, and increased levels of sodium, calcium, aluminum, manganese, and soluble iron.

The correlation between water content and contamination cannot be overstated. Less than 0.25 inch of water is sufficient for microbial growth[15]. Increased water content in systems increases the potential for biological activity within the system. The relationship between water and ULSD fuel is especially complicated by their inherent chemical properties. At room temperature ULSD will not typically hold more than 50 to 100 ppm of water in solution. Any excess water will eventually settle out of solution and create cloudiness in the fuel .Temperature and water retention have a linear relationship, warm fuel retains more moisture, conversely decreasing temperature results in decreasing solubility, increasing the content of free water in the system. An increased presence of free water, particularly in ULSD fuels, can also adversely affect fuel quality. A gel-like material can form from monoglycerides in fuel that have a stronger attraction to water than ULSD fuel. These materials exacerbate a system already plagued by copious amount of solids generated by exposure of steel components to an acidic environment. [16]

DPS #2 - Fill Port Scrapings (479270-003)	Result (mg/kg)	RL
9,12 Octadecadienoic acid, methyl ester (TIC)	262	5
Decane (TIC)	34.2	5
Docosane (TIC)	77.8	5
Dodecane (TIC)	40	5
Ethanol, 2-butoxy (TIC)	53.7	5
Heneicosane (TIC)	87.8	5
Heneicosane (TIC)	672	5
Heneicosane (TIC)	114	5
Heneicosane (TIC)	39.8	5
Hexadecanioc acid, methyl ester (TIC)	40	5
Octadecanioc acid, methyl ester (TIC)	199	5
Total TIC	2280	5
Undecane (TIC)	36.7	5

Table 5: TIC compounds not confirmed by GC/MS. Methyl esters are components of fuel. 2-Butoxyethanol has a low molecular weight and high pKa.

The source of contamination has yet to be determined and further investigation is required to establish the source, as well as verification and quantification of microbiological activity. The petroleum industry contends contamination control measures at the refinery level ensure fuel delivered to consumers is relatively free of contaminants. However experts contend multipurpose use of tanker trucks could provide a source of nutritional requirements, primarily ethanol. Future studies should focus on the presence of not only bacterial contaminants entering the system through fuel drops, but nutrients required for enhanced bacterial activity, specifically ethanol. The primary focus of our investigation was to highlight the commonalities between present conditions at DFW Airport to published scientific data documenting microbial contamination in fuel and fuel systems. Fuel degradation is suspected and further analysis of suspect particles of solid matter recovered from the filter matrix is warranted; as this unique particle is speculated to be a byproduct of fuel-water interaction. Positive identification of bacterial activity would provide more insight into specific chemical processes occurring in our tanks; but any approach should consider ULSD has a predisposition to contamination and fouling and implemented programs should plan accordingly. With current conditions reaching critical stages the implementation of a program designed according to site specific ULSD systems is recommended by the Environmental Affairs Department.

Recommendations

The literature pertaining to studies of ULSD corrosion in fuel systems and its link to microbial contamination are voluminous. Visual evidence of excessive corrosion in steel components of fuel systems has prompted interest in replacing steel and/or mild steel components of affected systems, with stainless steel parts. Initially, this step will greatly reduce the amount of filter plugging solids generated, but without proper tank cleaning and preventative maintenance measures, it is very likely conditions currently experienced, will return. There should be relatively little or no time lapse between replacement of steel components and tank cleaning. Avoiding recontamination of new components is the primary concern. Preventative maintenance programs specifically applied to combat microbial contamination in ULSD systems are highly recommended. There is no one size fits all program for this type of remediation but ASTM D-6469 Standard Guide for Microbial Contamination in Fuel Systems – provides an excellent framework for remediating microbial contamination. It includes testing utilized in preventative maintenance programs as well as a comprehensive outline for cleaning heavily contaminated systems.

Heavily contaminated systems generally require a three step process involving tank and pipe cleaning in conjunction with biocide treatment. The most effective programs include a three step process.

- Systems are first shock treated with biocide. Biocide-treated surface biofilms will slough off system walls and accumulate in tank bottoms.
- Systems should be cleaned. There are a variety of tank-cleaning strategies offered by service companies.
- Maintenance dose of biocide to treat microorganisms missed by fuel polishing and tank cleaning.

Any maintenance program implemented must address and monitor free water in fuel tanks. Microbial propagation requires nutrients and water; the absence of water greatly reduces chances for recontamination. There are many pathways, especially in underground systems, for water to enter the system, runoff from heavy rains, condensation from temperature fluctuations and tiny leaks in walls, seams, and seals. Requisite water conducive to microbial growth is only <0.25 inches. Monitoring of tank water bottoms and removal of free water when detected is imperative to prevent biological activity. Therefore, it is crucially important to implement an intensive water removal aspect of a long term preventative maintenance program. The following recommendations are a combination of ASTM standard and REG guidance for tank maintenance:

- The use of water paste or conductivity based water-detection devices (ATG systems) have been shown to give false negative results in biodiesel blends, including and especially ULSD. Strong evidence exists that neither of these methods is effective with wet biodiesel samples[17]. In ULSD, finer oil water emulsions are produced and lubricity, detergents, and additives added at the refinery lever disarm many traditional water separation filters. Floating striker plate devices don't show the last 0.75 inches of the tank bottom. (This is the ATG sensing utilized in our system.)

- Monitor storage tank bottoms by removing a sample from the bottom and comparing it to the fuel coming out of the dispenser. If the bottom sample is cloudy or has visible free water in it; tank bottoms should be cleaned and free water removed as soon as possible. Monthly bottom water sampling is recommended. Current conditions of our tanks may require more frequent sampling, tapering off as the issue is abated.
- Visual observations in conjunction with field tests would be used to determine if biological activity exists. Laboratory analysis would provide identification and quantification of positive field test results.
- At a minimum, yearly tank bottom cleaning is also recommended. Preferably not during "wet season" which is typically September through November.

Relevant Test Methods

- ATP Bioluminessence – Assessment of Adenosine Triphosphate – ASTM D7463
  HY-LiTE 2 – Merck. This is a prepared, one-shot device used to detect the presence of ATP (Adenosine Triphosphate), a substance found in all living cells and most biological material. This field test reveals the presence of residues capable of supporting microbial contamination. This system is relatively simple to operate and provides results within 60 seconds. This could be a very useful tool in the field to monitor biological activity.

- EasiCult TTC – EasiCult is another field test that can be performed in house as an aid for monitoring microbial contamination. It is highly recommended for use in jet fuel and diesel fuel. These tests are easy to perform and can determine if antimicrobial control measures are successful or determine potential future problems.

Recommended Biocides and Fuel Treatments

- Acid Glutaraldehyde (Sonacide) – This biocide is used in cold sterilization methods and has been shown to be effective on both gram-positive and **gram-negative** bacteria, **acid fast** bacteria, bacterial spores, and some fungi. It is non-ionic, non-foaming and rapid acting (1-4 hours) and is generally used as a shock treatment. It is particularly effective against SRB and biofilm and it is readily biodegradable.
- Kathon 1.5 – A non-oxidizing microbicide that is highly effective in hydrocarbon fuels including diesel and jet fuel. It can be used as a shock treatment (24-48 hours) or applied as a maintenance dose. Studies have shown it protects stored fuels for an extended time and it is effective in both fuel and water phases. It is effective over a wide range of pH and is effective

against general aerobic bacteria and spore-forming bacteria.

Table 6: Program Recommendations

Initial
• **Shock system with biocide. Allow recommended time for treatment efficacy. (Dependent on biocide type 4 – 48 hours)**
• **Remove fuel and clean tank thoroughly. These should be performed simultaneously to prevent cross contamination.**
• **Thorough cleaning includes walls and ceiling of tanks.**
• **Filter the fuel as it is put back into the system.** • **Treat with maintenance dose of biocide to dispatch residual microorganisms and prevent future propagation.**
Monthly
• **Storage tank bottom sampling. If the bottom sample is cloudy or has visible water in it, tank cleaning specifically for water removal is required.**
• **Field test for ATP Bioluminescence. If results are elevated, follow up testing with Easicult microbiological monitoring kit.**
Yearly
• **Tank cleaning. (Unless accumulation of solids or water removal is necessary in the interim.)**

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Appendix

I. Supplemental Materials

a. Microbiological and Corrosivity Characterizations of Biodiesels and Advanced Diesel Fuels

b. Effects of Microbiological Contamination in the Quality of Biodiesel Fuels

c. ULSD Corrosion: Finally an Answer

d. Preventing Problems from Biofuels and ULSD

e. A Biodiesel Blend Handling Guide

f. Renewable Energy Group Recommendations for Diesel Storage Tank Management.

g. Guidance for Underground Storage Tank Management AT ULSD Dispensing Facilities

h. The Requirement for Diesel Fuel Polishing: Fuel Stored on Site for Emergency Generator Sets

II. Analytics

III. ASTM D-6469 Standard Guide for Microbial Contamination in Fuel Systems

IV. Consumer Product Information – Test Methods and Biocides

a. EasiCult TTC – Orion Diagnostica

b. Dow Biocide Products

c. HyLite 2® – ATP Bioluminescence

d. Using Adenosine Triphosphate to Quantify Bioburdens in Various Liquid Fuels

e. Comparison Effects of Acid Glutaraldehyde and Alkaline Glutaraldehyde

f. Kathon 1.5 Product information

g. The Selection and Application of Nonoxidizing Biocides for Cooling Water Systems