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DIESEL FUEL LONG TERM STORAGE AND TREATMENT RECOMMENDED TESTS AND PRACTICES

I. INTRODUCTION

The Clean Air Act (1970) is the comprehensive federal law that regulates air emissions from stationary and mobile sources. Among other things, this law authorized the Environmental Protection Agency (EPA) to establish National Ambient Air Quality Standards to protect public health and public welfare and to regulate emissions of hazardous air pollutants. In recent years, EPA regulations have forced oil refineries into producing a very low sulfur diesel fuel and incentives for adding up to 5% bio-diesel. These changes to the fuel oil formulation are beneficial to air quality and to energy conservation, but adversely impact heat content, long term storage stability, engine power, and injection system reliability.

Diesel engines typically have a high incidence of injector failure resulting from poor diesel fuel quality. Since standby diesel engines do not run continuously it is necessary to implement periodic surveillance's to ensure the quality of diesel fuel is acceptable for reliable operation when a loss of power occurs. The information contained in this document is a compilation of best practices to be used as a guide for maintenance of a reliable diesel fuel system.

III. SUMMARY

The diesel engine fuel system is sensitive to fuel properties, to contamination, and to poor fuel quality that may cause the engine to fail to perform its design function. Excessive water, dirt, debris, oxidation products, and microbiological growth can cause plugged filters and early fuel injector failure. Water and dirt are especially destructive to the tight clearances and valve seat arrangements in modern fuel injectors. Erosion and corrosion of valve seats and lapped metal-to-metal interfaces cause poor combustion, smoke, and high or low combustion temperatures in the cylinder. A poorly adjusted or performing injector can wash the cylinder walls of its lubricating oil film causing premature wear and failure of the piston rings, pistons and cylinder liners.

It is important to realize that over an extended period of time diesel fuel oil will become degraded through oxidation and thermal instability. Since the rate at which diesel fuel degrades is unpredictable, it is important to take the necessary precautions to increase fuel system reliability. Good design, preferred fuel properties, sampling, testing and maintenance all contribute to fuel quality that can maintain your diesel generator as a reliable backup power source.

The Colonial pipeline currently delivers ultra low sulfur diesel (ULSD) and in the near future a 5% bio-diesel blend **(B5)** along the East Coast from Houston to New York. The formulation is predicted to be viable for about 4 months without stability additives. *Long term stability additives should be used* to extend the shelf life of all diesel fuel on site.

Stability additives typically contain metal de-activators to prevent reactive metals like copper and zinc from serving as an oxidation catalyst, dispersants to prevent filter plugging by reducing the size of suspended particulate, and corrosion inhibitors to protect the diesel engine fuel system components.

Acid number is considered a leading indicator of bio-diesel blend stability, so if B5 is stored for any amount of time it should be tested for changes in acid number. Particulates will begin to form as the acid number increases and the oxidation reaction becomes self sustainable. New additives are being developed to specifically treat bio-diesel blends. Sufficient lubricity and conductivity are concerns with ULSD; the ASTM standard gives minimum required values. Additives are used to ensure the product complies with specification.

Table 1 tabulates the recommended tests and test frequencies to be used by diesel engine owners to monitor fuel quality.

II. PURPOSE

The American Standards for Testing and Materials ASTM D975-08a specification for diesel fuel oils defines long term storage of #2 diesel fuel oil as fuel not replaced, filtered, or used within 6 months. The purpose of this addendum is to discuss the present concerns for the diesel fuel quality while in long term storage and compensatory actions to avoid or prevent consequences of poor diesel fuel quality.

IV. CONCERNS

Fuel Specification

Fuel oil normally recommended by engine vendors and used most commonly in backup power diesel engines is diesel fuel oil grade No. 2-D or DF-2. NFPA 30 designates grade 1-D and 2-D diesel fuel oils as Class II combustible liquids having flash points of 100 to 140 degrees Fahrenheit.

TABLE 1 - PRODUCT SPECIFICATIONS FOR STORED DIESEL FUEL OIL (GRADE 2-D)

Property Number	Property	ASTM Method	LIMIT	Frequency(5)
1	Kinematic Viscosity @ 100°F Centistokes	D445	1.9 min. 4.1 max.	**R**
2	API Gravity, °API	D1298	30 min. 38 max.	**R**
3	Water and Sediment, % by vol.	D2709	0.05 max	**R** **M OR Q**
4	Flash Point, °F	D93	125 min.	**R**
5	Cloud Point, °F	D2500	23 max(1)[1]	**R**
6	Grade 2-D S500 Sulfur, % by weight Grade 2-D S15 Sulfur, ppm	D4294 D5453	0.05% max. 15 ppm max.	**R**
7	Carbon Residue, %	D524	0.35 max.	**R**
8	Ash, % weight	D482	0.01 max	**R**
9	Corrosion rating, copper strip	D130	Number 3	**R**
10	Cetane Index Cetane Number	D976 D613	40 min. 47 min (4)	**A** **A**
11	Particulate content, mg/L	D6217	20(2) max.	**R** **M OR Q**
12	Distillation Range 90% point °F, (°C)	D86	540 min, (282) 640 max, (338)	**R**
13	Visual Appearance	D4176	Clear/Bright	**R** **M OR Q** **Bottom sample**
14	Microbiological Testing	Per Vendor's Literature	Use D6469 for guidance	**P**
15	Lubricity, HFRR@60 deg C	D6079	520 micron max	**A**
16	Storage and Oxidation Stability	EN 14112 D6468	European (3.1) 80% reflectance, min (3.2)	**A**
17	Presence of Bio-diesel	D7371	5%	**A**
18	Acid Number	D664	0.50 mg/KOH/g	**A**
19	Conductivity	D2624	25 pSm	**R**
20	Aromaticity	D1319	35% volume max	**NR**
(1)	As specified in ASTM D975. "Max" means a cloud point higher than that is not acceptable			
(2)	Particulate Content Limit is 5 mg/L for new fuel and 20 mg/L for stored fuel.			
(3.1)	As of 2008, US to use the European standard method rather than to develop a new ASTM for Rancimat			
(3.2)	Reference Polaris Laboratories, LLC when D6468 is used			
(4)	Reference ASTM D6751 X1.7 cetane number test is used for bio-diesel blends			
(5)	**A**nnual, **Q**uarterly, **M**onthly, **R**eceipt, when **P**articulates or Sediment are found, **NR** not required (yet) *Recommended for SS and SC equipment*			

Receipt Sampling

The purpose of receipt sampling is to verify the delivered product has the correct properties for diesel operation and to screen for gross contamination. It is important to know the quality of fuel arriving at each facility; determination of aging over time is made much easier knowing the initial conditions.

Fuel Additives and Periodic Sampling

The two most common failure modes in diesel fuel quality are from particulate contamination and bio-fouling. Particulate contamination occurs when chemically unstable fuel oil oxidizes to form a particulate. In severe cases, particulate contamination and biological fouling of a fuel tank can lead to failure of the diesel engine. For this reason the recommendations in this document focus primarily on minimizing the conditions that can lead to failures of this nature. Most sources of diesel fuel oil available to the site are refined through a catalytic hydro-cracking or hydro-treating process which produces a lower quality fuel oil than "straight run" fuel oil. Straight run was the term used for fuel oil refinery distilled at low temperatures and one atmosphere pressure. Straight run fuels are more stable because the molecules are not fractured during the refining process, do not interact with each other, and are much less influenced by moisture, heat and contamination. Catalytically cracked distillates have a projected shelf life of as little as 3-6 months. Even with stability treatments, after one year the fuel oil is highly susceptible to breakdown through oxidation. Shelf life of the fuel oil is a significant problem because the amount of time fuel is stored at the supply depot and on-site cannot be controlled. Biocides, stabilizers and dispersants should be added to prevent bacteria and fungi growth, prevent fuel from aging rapidly, and to prevent entrained water or smaller oxidized particles from collecting into larger particles that damage injection equipment and plug filters. Currently, 200 ppm Biobor JF or Kathon FP 1.5 are recommended; long term storage additive LTSA-35A has been used historically at SRS for stability.

Particulate and Biological Growth

Since the chemical composition of diesel fuel oil is complex and variable it is extremely difficult to predict how long treated fuel will remain stable. For this reason particulate analysis should be performed quarterly to verify that particulate levels are below 20 mg/L per ANSI N195 (3.1). This limit is typically recognized as the maximum level of particulate contamination that will not interfere with diesel operation. For procurement purposes the limit is set at 5 mg/L. Once stored fuel oil reaches particular concentrations of 8-10 mg/L, predicting the acceleration of oxidation is extremely difficult. For this reason planning to correct fuel stability problems at 10 mg/L is recommended to avoid an inoperability condition.

Biological fouling of diesel fuel can lead to diesel inoperability. Certain living organisms grow at the water / oil interface even in a completely dark tank. The monthly bottom sampling and a biocide addition to new fuel oil are precautions recommended to prevent microbiological growth. Eliminating water from the tank bottom is important as all microorganisms require water to survive. A consistent fuel tank draining program backed by a biocide treatment will adequately reduce the risk of biological fouling. Testing for the presence of microbiological growth should be performed only as required when a problem is suspected and not on a periodic frequency. At this time, there are no ASTM standard methods for microbe screening and testing for microbiological contamination. Testing on a periodic frequency is not a recommendation of NUREG 1.137. Testing for microorganisms should be performed using a field microbe test kit and the test results should be interpreted based on the vendor's literature.

Ultra Low Sulfur Diesel Fuel (ULSD) – Aging, Heat Content, Lubricity

Because the sulfur has been stripped from the long chain hydrocarbon molecules by cracking or by treating, ULSD is said to be very **polar***. As diesel fuel ages hydrogen-peroxides form which can become soluble gums when cooled. At 90 degrees C or less, hydrogen peroxides are stable, but at 100 degrees C or greater they are highly unstable. The diesel fuel that runs through an engine fuel system is typically heated >100 degrees C and most of it returns to the fuel tank. The aging concern for ULSD is with varnish and sludge or soluble gums. The aging concern with higher sulfur fuels was the generation of particulates in the form of waxes and asphalts. When ULSD is oxidized, it smells like turpentine or paint remover. This condition was particularly noticed at SRS when cleaning the 715-2N fuel storage tanks, Spring 2008.

There are several properties that may be affected by the severe (hydro) cracking of diesel fuel. Because sulfur is stripped off of the hydrocarbon chains, heat content may be lower in BTU/gallon, viscosity may be lower, the cetane index (an anti knock indicator) may be low. Lowered lubricity and wear of injection equipment has been evaluated. ASTM D975 now requires a minimum lubricity for all diesel fuel grades. Lubricity is typically brought into specification using additives. The property should be tested periodically using method D6079 to ensure minimum lubricity requirements are being met.

***Polarity** underlies a number of physical properties including surface tension, solubility, and melting- and boiling-points. Polar chemicals are ones that possess an overall positive charge on one end of the molecule and a negative charge on the other end. This charge can be determined by drawing out the structure and determining the dipole moments (area of greater electro-negativity). For example, water has the "mickey-mouse" ear shape, and because oxygen has a greater tendency to attract electrons, the positive polar region is around the oxygen. ULSD being polar will bond with water and oxygen readily.

Bio-Diesel

Bio-diesel is an unsaturated fatty acid methyl (or ethyl) ester (FAME) and is also a very polar material. A 5% mixture of bio diesel and 95% petroleum based diesel fuel is called ***"B5"***. As stated above, ULSD is also made of polar molecules and binds easily with available oxygen and with bio-diesel. Hydrogen peroxides lead to formation of carboxylic acids which attack metals and cause buna-N or nitrile rubber o-rings to become brittle. B5 can have a cleaning effect that loosens accumulated sediment in fuel oil storage tanks that previously stored conventional diesel fuel. This sediment can then plug filters and injection equipment in the fuel oil system. To prevent the buildup of sediment, SRS facilities may decide to clean fuel oil storage tanks before putting B5 in them and upgrade the filters in the fuel oil system. Expect to change and/or clean filters more frequently especially during the early stages of B5 use.

Water

B5 contains entrained particles of water from the manufacturing process. This water will, in time, fall out of suspension and form "dirty water" in the fuel oil storage tank which eventually leads to the formation and growth of microbes. To prevent the formation of dirty water and the subsequent growth of microbes, take the following actions: Use moisture dispersant and biocide in fuel oil storage tanks containing B5. Add a fuel/water separator to the fuel oil system. Keep fuel oil storage tanks topped off to minimize in-tank condensation.

Biodegradation

B5 is biodegradable, and the presence of water, heat, oxygen, and other impurities accelerate the degradation of the fuel supply. Unsaturated fatty acid esters are more susceptible to oxidation, and as a result bio-diesel oxidizes readily in the presence of air during storage. To avoid damage caused by fuel degradation, consider not using B5 if it has been stored for 3 to 6 months without an oxidation stabilizer.

Material Incompatibility

Brass, bronze, copper, lead, tin, and zinc in tanks and fittings may accelerate the oxidation process of B5, creating fuel insolubles or gels and salts. Facilities should avoid using zinc linings, galvanized pipe fittings, copper pipes and fittings, and brass regulators with B5. Verify that elastomeric materials, such as hoses, gaskets, and O-rings, and their inspection and maintenance, are compatible with B5 and its effects.

Temperature Protection

Bio-diesel components have higher cloud points (the temperature at which wax or gel begins to form) than standard (petroleum) diesel components. The cloud point also varies

considerably with the source of the bio-diesel component, which is not specified in B5 blends. Clouding may also combine with suspended particles of water and exacerbate adverse cold temperature concerns. Consequently, evaluate and ensure adequate low temperature protection for all diesel generator system components. Monitor the cloud point of incoming shipments periodically.

Heat Content

A 5% blend of bio-diesel and petroleum distillates is expected to have an impact on BTU/gallon of fuel oil burned in the diesel engine. Predictions range from 2-10% reduction when using ULSD, B5, and high aromatics. The California Air Resources Board (CARB) mandated low aromatic diesel fuel use several years ago. This is fuel oil that has been hydro-cracked to remove benzene primarily. The West Coast nuclear industry used the high limit of 42 degrees API to calculate heat content reduction of 12-14%. Is low aromatic diesel fuel coming to the East Coast ? API gravity and / or specific gravity are primary indicators of heat content and they should be monitored periodically.

Other Additives

Some distributors are adding detergents to diesel fuel which also tend to bond to ULSD and form a jelly-like substance. Glycol is added at some terminals as a winterizer; it will also bond with ULSD to form soluble gums. Ethylene glycol (monoethylene glycol (MEG) is an alcohol with two -OH groups (a diol), a highly polar chemical compound widely used as an automotive antifreeze. Ask your distributor what chemicals are being added at the terminal because detergents and glycols will cause problems.

V. SAMPLING TECHNIQUE

Manual Sampling of SRS Diesel Fuel Storage Tanks

Representative sample
A portion extracted from the total volume that contains the constituents in the same proportions that are present in the total volume. This would be much like taking samples every 1 or 2 feet in the tank and mixing them in a common container then decanting into the sample bottle.

All level samples

Obtained by submerging the thief to a point as near as possible to the draw off level then opening the sampler and raising it at a rate such that it is approximately ¾ full as it emerges from the liquid

Outlet sample
A spot sample taken with the inlet opening of the thief at the level of the bottom of the tank outlet. Also called a dispensing level sample.

Upper sample
A spot sample taken from the middle of the upper 1/3 of the tanks contents.

Top sample
A spot sample obtained 6 inches below the top surface of the liquid.

Clearance sample
A spot sample taken with the inlet opening of the sample thief 4 inches below the bottom of the tanks pump suction line.

Bottom sample
A spot sample collected from the material at the bottom of the tank.

Sampling with the Bacon Bomb Thief

- Do not use an automatic all level sampler because the results will not be repeatable. The level at which the auto thief captures the sample is dependent on the rate of travel down into the fuel tank

- Use a spot sampler (thief) having a lowering chain and a fill valve chain so that different level samples can be taken depending on what information is required. Sampling for TSR needs to be defined… Do you want clearance sample, outlet sample, or representative samples?

- Many commercial nuclear power plants prefer the multi level or representative sample to ensure the oil sample is representative of the entire tank contents. However, many will draw a clearance sample which is considered worst case scenario for TSR.

- Transportation Fuel Management draws their samples from the dispensing level (an outlet sample) in order to determine the quality of fuel being delivered to their customers. During the annual PM, a bottom sample is taken to determine whether there is water or sludge buildup to pump out.

- Since day tanks are being monitored for sediment, water and particulates, it is recommended that the all level (representative sample) be used to determine operability.

Particulate, sediment and water will typically be discovered in the day tank before storage tanks.

- Take clearance and bottom samples when you want to know how stored fuel is aging. Since you have very little clearance volume, I remove all thief extensions and grab a bottom sample periodically to determine the "health" of the fuel tank.

- Tools recommended for sampling include a 1-1/2 or 2 gallon stainless steel bucket, thief with actuator chain, absorbent material, sample bottles, gloves, apron, and other PPE as required.

- It cannot be stressed too fervently: Sample thief, sample bottles and caps, gloves, any item coming in contact with the fuel to be tested must be rinsed in clean fuel oil prior to capturing a sample for test.

- **Sampling Procedure:**

1. Clean the bucket and the sample thief with alcohol and lint free cloths
2. Ensure the bucket is visibly clean, wipe with lint free cloths
3. Lower the thief into the tank and grab several top or upper samples of fuel and pour them in the bucket. This fuel will be used for rinsing parts
4. Put sample bottles in the bucket of fuel and rinse them several times, put the tops back on and set aside on absorbent towels
5. Disassemble the thief completely and rinse all parts in the bucket of fuel
6. Re assemble the thief ensuring the gloves worn were rinsed in clean fuel before handling the thief
7. Once re-assembled, take a representative, outlet, clearance, or bottom sample as required
8. Holding the sample bottle over the bucket, pour from the thief into the sample bottle until full,
9. Pour the dregs from the thief into the pail after filling the sample bottle. Do not pour off the last few fluid ounces from the thief into a sample bottle as it may not be representative. Put the top on the sample bottle(s)
10. Continue to pull samples and pour into sample bottles over the waste bucket
11. Do **not** discard the rinsate fuel back into your fuel tank. The rinse pail contents are usually poured into a double walled oily waste can at the sampling site. You can buy them, identify them, and have them emptied periodically by site forces.

Periodic Testing

The recommended periodic tests and test frequencies are shown in Table 1. The following properties have been added:

Fuel Stability and Oxidation D6468

Presence of Bio-diesel D7371
Acid Number D664
Lubricity D6079
Conductivity D2624
Aromaticity D1319
Cetane Number D613

VI. RECOMMENDED PRACTICES

Receipt Sampling

All fuel for safety related diesel engines should be sampled on receipt. When the fuel shipment is sampled and how the sample is obtained will depend on the logistics of delivering the fuel at each facility. A facility can request that a vendor wait while the initial analysis is performed or a facility may sample a fuel delivery ahead of time and control the batch until it is unloaded at the site. It is essential that a representative sample of all fuel is taken and the load of fuel is monitored or secured from the time it is sampled until the time it is unloaded. If the fuel is delivered by truck or by rail a composite sample consisting of equal parts from the lowest point of each compartment should be obtained. ASTM D4057 can be used as a reference for sampling methods. Approximately one to two liters of fuel is required for laboratory testing. The exact quantity depends on the laboratory that performs the fuel analysis.

Five properties should be tested upon receipt prior to off loading: 1) Sediment / Water and 2) Visual Appearance are checked to detect contaminated fuel oil, 3) Kinematic Viscosity and 4) API Gravity are checked to verify that the product has the appropriate lubricity and energy content as there is a direct correlation between API Gravity and the Heat of Combustion, 5) Flash point is measured to ensure requirements for handling a Class II combustible product are met. All other properties required from receipt sampling are as specified by the Standard Specification for Diesel Fuel Oils, ASTM D975 and ANSI/ANS-59.51, Appendix B, per the recommendation of NUREG. 1.137.

The diesel fuel should appear "Clear and Bright" as described in ASTM D4176. The ASTM distributes a card to be used as a visual guide. The card is placed behind the fuel sample as an aide in grading a sample's clarity. The card is very helpful in quantifying this inspection per D4176 on a scale from 1 to 5. Using the D4176 card, a fuel equal to or higher than a 3 should be rejected.

Long Term Storage

Long term stability additives should be used to extend the shelf life of the fuel on site (e.g., Fuel Quality Services LTSA-35A , MSDS # 28291) - 1. Furthermore, stability additives typically contain metal de-activators to prevent reactive metals like copper and zinc from

serving as an oxidation catalyst, dispersants to prevent filter plugging by reducing the size of suspended particulate, and corrosion inhibitors to protect the diesel engine fuel system components.

LTSA-35A is a multifunctional diesel fuel stabilizer additive package containing an antioxidant, corrosion inhibitor, dispersant, and metal deactivator that is specifically designed, tested, and approved under MIL-S-53021 for use by all Departments and Agencies of the Department of Defense. LTSA-35A stabilizer additive is one part of a two part package that also includes the microbiocide Kathon® FP1.5. They are intended to be added to diesel fuel to retard or prevent the formation of fuel deterioration products such as gums, sludge, particulates that result from auto-oxidation processes, to reduce the potential for microbiological growth, and to provide for corrosion protection of fuel-wetted surfaces. Primarily, this product is for the treatment of fuel in depot facilities where vehicles and equipment are in re-build or storage, pre-positioned material at locations involving storage of equipment partially or fully fueled, and fuel stocks intended for intermediate storage of 6-24 months or long-term storage of 25-60 months. LTSA-35A has also found wide spread use in a number of non-military commercial applications such as long term storage of middle distillate fuels for utilities, standby diesel generators, home heating oil, and bio-diesel blend (B5/B20). Suggested treat rate: 1 gallon of LTSA-35A to 5,000 gallons of fuel.

Test DF2 periodically for thermal stability (D6468). This will provide information about petroleum based fuel's potential for asphaltene dropout, one of the most common causes of fuel filter plugging. Asphaltenes are tar-like, resinous substances that "drop out" of suspension and tend to agglomerate at high engine operating temperatures. Thermal stability measures the fuel's resistance to permanent changes in properties caused by heat. In determining thermal stability, a percentage range is assigned to the fuel based on its tendency to produce asphaltenes. The higher the fuel's thermal stability, the lower its potential for producing asphaltenes. According to Polaris Laboratories LLC, fuel with a Thermal Stability of 80% or greater should not cause filter clogging. Fuels between 60% and 80% could have a marginal affect and values less than 60% will significantly reduce filter life. If thermal stability is less than 60%, consider treating the fuel with an asphaltene conditioner, which will prolong suspension.

Test B5 periodically for oxidation and long term storage stability (EN 14112 Rancimat method). Six different ASTM test methods were evaluated between 2005 and 2007 to determine what test for bio-diesel oxidation stability would be best. All test results and reports, both the USA and EU indicate that the Rancimat method would be the most sensitive and was best used on fuel with fatty acid methyl esters (FAME).

Remove / drain water from tank bottoms and monitor particulates either monthly or quarterly. Take bottom samples periodically and compare with the clear and bright test. When sediment or particulates are found, test for microbes.

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