Naval Fuels & Lubricants
Cross Functional Team
Research Report

F-76 LUBRICITY IMPROVER ADDITIVE EVALUATION

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LIST OF ACRONYMS/ABBREVIATIONS

NCT .... Naval Coalescence Tester

HFRR .... High Frequency Reciprocating Rig

LIA .... Lubricity Improver Additive

ASTM .... American Society for Testing and Materials

ppm .... parts per million

MSEP .... Micro Separometer

SDA .... Static dissipater additive

ULSD .... Ultra low sulfur diesel

FFP .... Fit-for-purpose

CONUS .... Continental United States

NAVSEA .... Naval Sea Systems Command

DEFINITIONS

Availability .... Ability to procure a fuel to a specified requirement.

F-76 .... NATO code for ship diesel fuel.

EXECUTIVE SUMMARY

Both on and off road fuels have seen a recent decline in their sulfur concentrations throughout the last several years. A decrease in the sulfur limits of fuel has resulted in the effective reduction of sulfur compound emissions in addition to important technological advances in vehicle emission research. However, the processing necessary in order to remove these sulfur compounds also removes other molecules important to the fuel's lubricity, and could lead to undesirable interactions with diesel engine components. In order for the Navy to further reduce sulfur levels below its current 0.1 mass% limitation, a lubricity additive specification and qualified product list must be established to allow procurement of LIA for injection at select defense supply points.

The objective of this investigation is to expand and diversify the number and type of diesel lubricity additives that are available for use in the Navy's distillate F-76 fuel. By increasing the number of approved LIA's and diversifying the chemistries of these additives, the Navy will be prepared to compensate for the decrease in lubricity as ultra low sulfur fuels shift into focus while still retaining the fuel's water separability traits.

Test and evaluation of the additives focused on three main sections of the evaluation segment of the Navy draft fuel and additive approval process: specification, fit-for-purpose, and rig testing. A local ultra low sulfur diesel (ULSD) was clay treated in order to mimic ultra low sulfur fuels that would potentially be acquired by the Navy in the future. Additives were tested at two times the manufacturer's recommended treat rate for both specification and fit-for-purpose testing, and several of these tests focused on the effects of the additives to the fuel's water separation properties. Rig testing was conducted on the Naval Coalescence Tester only with additives that had failed crucial water separability testing such as demulsification and diesel MSEP. It was determined that if the worst possible case passed NCT testing, then all fuels additized with LIA that produced passing specification and fit-for-purpose results would pass as well.

A total of eleven additives were submitted for testing. The additives completed all specification, fit-for-purpose, effectiveness and compatibility testing. The additives demonstrating the poorest water separability characteristics were submitted for NCT rig testing in order to conduct a more in depth investigation into the effect of the additive on the fuel's water separability capabilities. Eight of the additives tested exhibited favorable properties for use in the Navy's distillate F-76 and will be recommended to NAVSEA for further testing in accordance with the draft F-76 lubricity additive specification. The three remaining additives were found unsatisfactory due to their low flash point.

F-76 LUBRICITY IMPROVER ADDITIVE EVALUATION

1.0 BACKGROUND

Over the last several years, the sulfur content of on-road diesel has steadily decreased to become the ultra low sulfur diesel fuel that is commonly used today. While this has resulted in significant reductions in sulfur compound emissions and technological advances in overall vehicle emission reduction, the extra processing required to create these fuels also removes additional compounds that influence a number of the fuel's properties, including lubricity. More recently, regulations are beginning to take effect to lower the sulfur content of off-road fuels. To date, the Navy specification for F-76 still allows up to 0.1 mass % sulfur. However, to lower the F-76 specification sulfur concentration to the current 15 ppm "ultra low" level will require the addition of diesel lubricity improving additive (LIA) to return fuel lubricating compounds. In a previous study conducted in 2009, four additives were down selected and recommended for use in the Navy's F-76. Testing results from these four additives, along with additional additives screened in this test program, will form a basis of experience for the Navy's Lubricity Improver Additive Specification, reference (a) and Qualified Products List currently in development.

2.0 OBJECTIVE

The objective of this test program is to diversify the Navy's set of usable ultra low sulfur diesel lubricity (ULSD) additives through test and evaluation of candidate additives as a means of screening candidate additives for full evaluation against the requirements of the draft F-76 lubricity additive specification, reference (a). As the Navy follows the trend of shifting to ULSD's, additives that work in the Navy's at-sea environment must be found to compensate for the decrease in lubricity that is often found in ULSDs, reference (b).

3.0 APPROACH

The testing for this investigation closely mirrored the 2009 study that down selected four lubricity additives for Naval use. Major additive manufacturers were contacted and asked for samples of diesel lubricity additives that they thought would perform acceptably in a water laden environment and would not interfere with the fuel's water separability or coalescence properties. A total of seven additive samples from six manufacturers were obtained for testing. Four more additives were later received from an additional manufacturer and added to the testing regimen as well.

The Navy's fuel and additive approval process shown in Figure 1 was used in order to develop an appropriate test plan to evaluate the additives. The processes used to evaluate these lubricity additives were concentrated on the yellow evaluation division of the flow chart, with emphasis on the specification, fit for purpose, and rig testing sections.

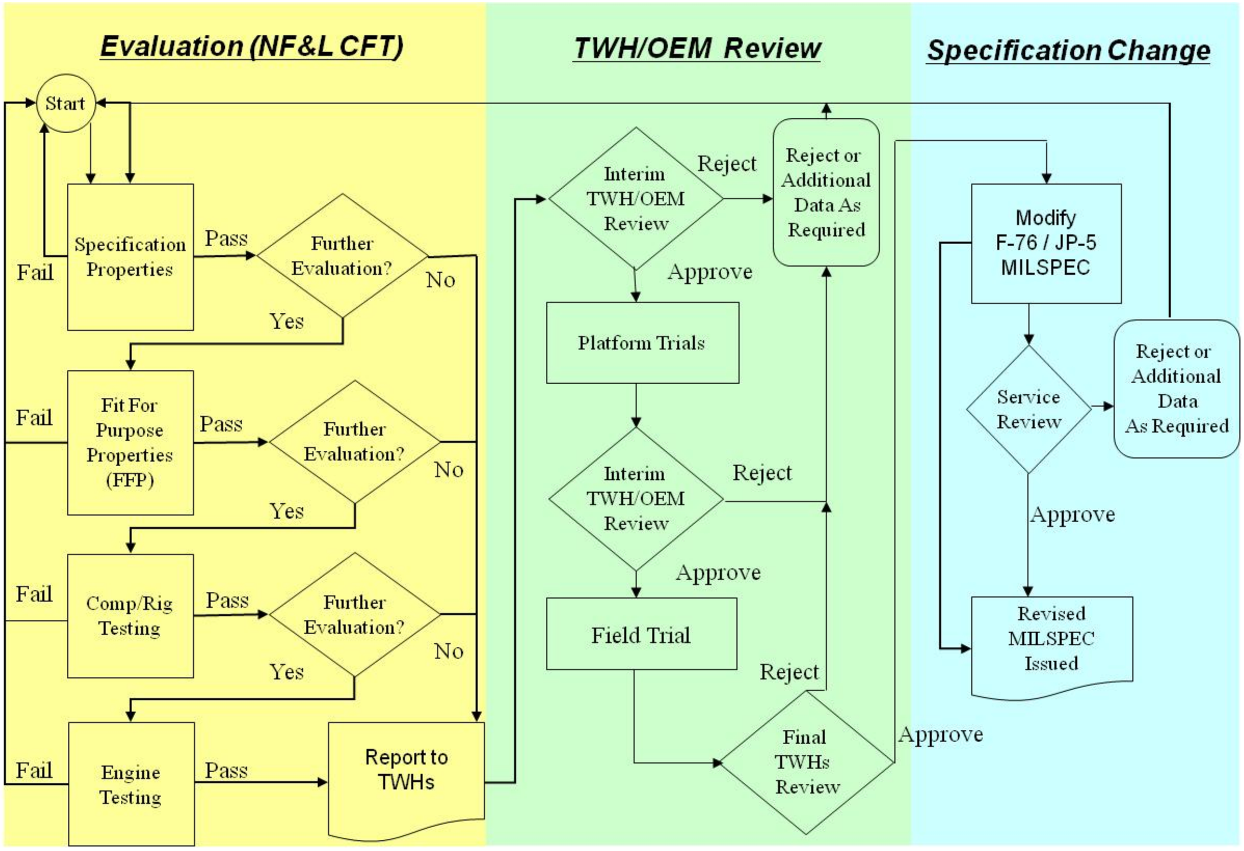

Figure 1: Navy Fuel and Additive Approval Protocol

3.1 Specification Testing

All eleven lubricity samples were initially submitted for specification testing. A local ULSD was obtained and clay treated to ensure removal of any existing lubricity additives in the fuel. The fuel was then additized at two times the manufacturer's recommended treat rate and submitted for eight selected specification tests. The base ULSD was also submitted for the same tests to ensure that any deviations in results would be due to the lubricity additive. The eight specification tests are listed below.

1. Cloud point
2. Cu strip corrosion 100°C
3. Demulsification @ 25°C
4. Flash point
5. Pour point
6. Storage stability
7. Sulfur, total
8. Total acid number

3.2 Fit-for-Purpose Testing

Following completed specification testing, the additized fuels were then submitted for fit-for-purpose testing. Fit-for-purpose properties (FFP) are the chemical and physical aspects of a fuel that are not typically measured for petroleum derived fuels, but still impact the performance, materials compatibility, handling, and safety properties of the fuel. The purpose of testing FFP properties was to ensure that there were no unintentional consequences when lubricity additives were added to naval distillate fuels. The fit-for-purpose tests that were performed are listed below.

1. Interfacial tension
2. Water reaction
3. Diesel micro-separometer (MSEP)

3.3. Additive Compatibility

Once fit-for-purpose testing was completed, additive compatibility studies were also conducted. Testing was performed using the procedure listed, with minor variations, as identified in ASTM D4054 Annex A2. LIA compatibility with static dissipater additive, ignition improver, middle distillate flow improver, antioxidants, and corrosion inhibitor/lubricity improver additives was tested. Instead of cooling the additive mixtures to the specification temperature of -17.8°C (0°F), the test was carried out at 0°C due to the freezing point of diesel fuel. These five additives were selected because they are commonly found in diesel fuels used by the Navy. If the additive had more than one chemistry type, then one additive representative of each chemistry type was included in testing.

Additionally, the LIAs were tested for compatibility with diesel engine lubricant oils due to the amount of mixing that occurs during diesel engine operations onboard ship. Particulate matter formation caused by the unfavorable reaction of these LIA's and engine lubricant would be detrimental to diesel engine operation. A testing protocol used in the 2009 lubricity additive evaluation obtained from the UK Navy was used for this investigation. 50 mL of fuel additized at two times the recommended treat rate was blended with 50 µL of lube oil and stored in a compatibility oven at 80°C for one week. After the samples were removed and allowed to cool, they were examined for signs of particulate matter, cloudiness, or other signs of incompatibility between the two additives. Five lubricant oils were selected for compatibility testing: Shell Gadinia, MIL-PRF-23699, MIL-PRF-9000, MIL-PRF-17331, and MIL-PRF-32353. A testing matrix was developed in order to confirm that the diesel lubricity additives were each exposed to all five lube oils.

3.4 Additive Effectiveness

Additive effectiveness was also investigated by additizing fuel samples at different LIA treat rates and recording their respective average HFRR wear scar diameters. Additives were tested at the manufacturers recommended treat rates, or to 200 parts per million, which was greater. These relationships were plotted out and are shown in Appendix A.

3.5 Rig Testing

Rig testing was determined as the final stage of the lubricity additive evaluation process. Like the previous 2009 study, it was determined that the Naval Coalescence Tester (NCT), reference (c), was the only rig necessary to complete the survey of the lubricity additives undergoing testing. The NCT is a fit-for-purpose test which allows for fuel flow through a fuel coalescer, while using a small amount of fuel. The objective of this test is to determine the water shedding, or coalescence properties, of the test additive. A known amount of free water is injected upstream of the test element and upstream and downstream free water levels are measured and compared to the saturated water level in the fuel. A passing test shall give downstream total water measurements that are comparable to that of the saturated water level of the fuel, instead of the upstream levels where additional water had been added.

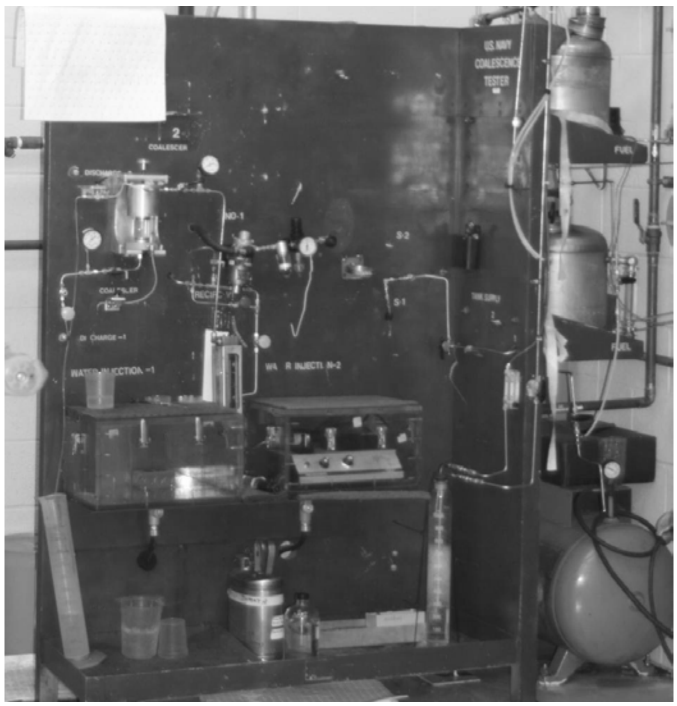

Figure 2: Naval Coalescence Tester

4.0 DISCUSSION

4.1 Specification Testing

Table 1 shows the manufacturer's recommended treat rates for all additives evaluated in this study. Specification and FFP testing was done at 2x recommended treat rate also shown in Table 1. Results from the diesel lubricity additive specification testing are shown below in Table 2. All additives were treated at two times the manufacturer's recommended treat rate. Discrepancies were found for the flash points of Additives E, F and G. However, after consulting with industry and manufacturer contacts, it was determined that the flash point of the additized fuel could be increased to the 60°C minimum by requesting that a less volatile solvent be used when manufacturing the additives. Additive K failed the demulsification test at twice maximum treat rate, but was deemed acceptable at 1x treat rate. All additives proceeded to fit-for-purpose testing.

Table 1. Additive Treat Rates.

Additive	Suggested treat rate (ppm)	Actual treat rate (ppm)
Additive A	200	400
Additive B	75	300
Additive C	200	400
Additive D	75	150
Additive E	75	150
Additive F	100	200
Additive G	100	200
Additive H	100	200
Additive I	100	200
Additive J	100	200
Additive K	100	200
Base ULSD	---	---

Table 2. Specification Test Results

	ASTM method	Units	Requirement	Base ULSD	Additive A	Additive B	Additive C	Additive D	Additive E
Cloud point	D5773	°C	-1 (max)	-11.8	-11.9	-14.2	-12	-11.8	-11.8
Cu Strip Corrosion @ 100°C	D130	--	No. 1 (max)	1a	1a	1a	1b	1a	1b
Demulsification @ 25°C	D1401	min	10 (max)	1	2	2	2	4	3
Flash Point	D93	°C	60 (min)	60	62	**57.5**	64	60	**59**
Pour Point	D5949	°C	-6 (max)	-21	-21	-21	-21	-21	-21
Storage Stability	D5304	mg/100ml	3.0 (max)	0.7	0.7	0.3	0.35	0	0
Sulfur, Total	D5453	mg/kg	15 (ULSD max)	12.9	11.6	6.4	10.2	9.9	9.7
Total Acid Number	D974	mg/g	0.3 (max)	0.011	0.008	0.025	0.005	0.025	0.039
	ASTM method	**Units**	**Requirement**	**Additive F**	**Additive G**	**Additive H**	**Additive I**	**Additive J**	**Additive K**
Cloud point	D5773	°C	-1 (max)	-11.6	-11.8	-11.8	-11.9	-11.8	-12.1
Cu Strip Corrosion @ 100°C	D130	--	No. 1 (max)	1a	1a	1a	1a	1a	1a
Demulsification @ 25°C	D1401	min	10 (max)	3	3	3	3	2	**19**
Flash Point	D93	°C	60 (min)	**59**	**57.5**	60.5	60	60	61
Pour Point	D5949	°C	-6 (max)	-21	-21	-21	-21	-21	-24
Storage Stability	D5304	mg/100ml	3.0 (max)	0.15	0.35	0.95	1.6	2.3	1.55
Sulfur, Total	D5453	mg/kg	15 (ULSD max)	9.4	9.3	11.4	10.8	11.2	11.9
Total Acid Number	D974	mg/g	0.3 (max)	0.053	0.061	0.045	0.044	0.003	0.011

4.2 Fit-For-Purpose Testing

Data obtained from fit-for-purpose testing of the 11 additives are shown in Table 3. These tests were also conducted at two times the recommended manufacturer concentration used for specification testing. The interfacial tension, water reaction and diesel MSEP tests were run in order to give a more in-depth look at the water separation abilities of the additized fuels. All additives successfully passed the interfacial tension test. Additive B failed both the water reaction and diesel MSEP tests, and three additional additives (A, C, and K) failed the diesel MSEP. The additives that received borderline diesel MSEP ratings were tested again at the original recommended treat rate. It was found that this decreased treat rate brought the MSEP rating to acceptable levels, with the exception of Additive C.

Table 3. Fit-for-purpose testing

	Interfacial tension	Water reaction--(separation), interface	Diesel MSEP
ASTM method	D971	D1094	D7261
Units	Dyn/cm	---	---
Requirement	Min 20	Max (0) 1	90
Base ULSD	40.3	(-1) 1	97
Additive A	21.2	(-1) 1	**83**
Additive B	39.6	**(-2) 2**	97
Additive C	20.8	(-1) 1b	**59, 58**
Additive D	43	(-3) 1	97
Additive E	32.7	(-1) 1	97
Additive F	41.3	(-3) 1	98
Additive G	43.3	(-1) 1	99
Additive H	44	(-1) 1	99
Additive I	44.3	(-1) 1	97
Additive J	21.8	(-1) 1	91
Additive K	22.5	(-1) 1	**51**

4.3 Additive Compatibility

After fit-for-purpose testing, additive and lube oil compatibility testing was conducted in accordance with procedure described in section 3.3. Candidate lubricity additives exhibited no signs of incompatibility with other common fuel additives. Samples were clear and bright with no visible particulate after cold and warm storage. Candidate lubricity additives also showed no signs of degradation or unfavorable interaction with lube oils stored at elevated temperatures. These results show that all candidate lubricity additives interact favorably with the lube oils as well as other common additives.

4.4 Additive effectiveness

Additive effectiveness was also tested using four treat rates, beginning at 50 ppm and increasing in increments of 50 until reaching 200 ppm, in order to examine the relationship between additive concentration and lubricity. The results obtained from Additive A are displayed in Figure 3. The horizontal red line marks the maximum 460 µm average wear scar diameter threshold. The remainder of the additive effectiveness data can be found in Appendix A.

Table 4. Additive A effectiveness data

Treat Rate (ppm)	50	100	150	200
WSD (µm)	550	500	360	340

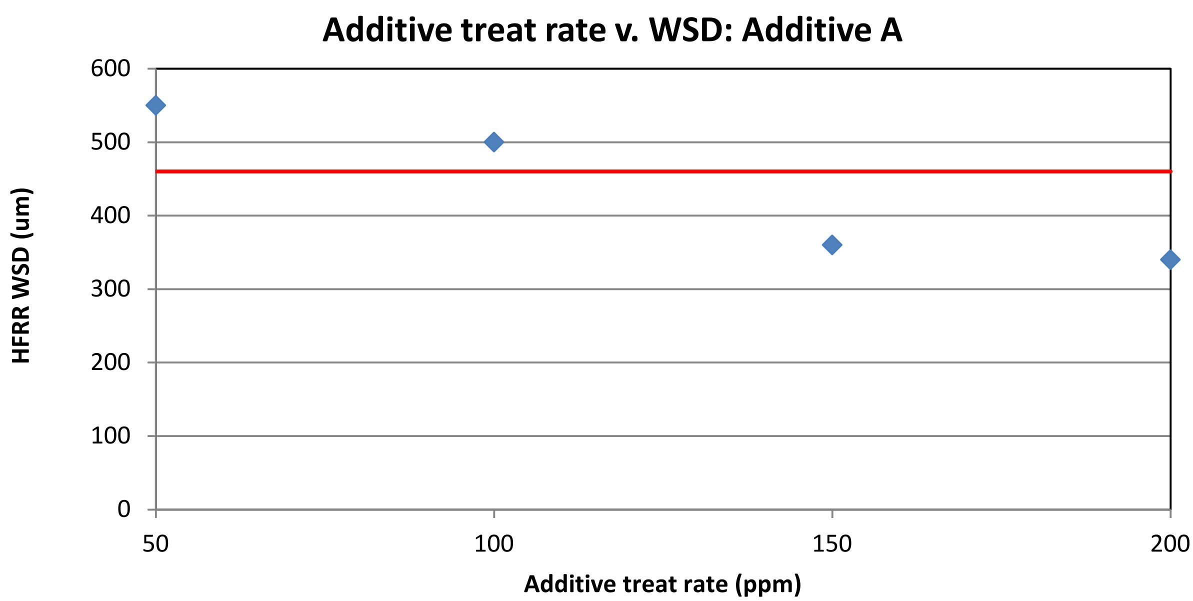

Figure 3. Additive A effectiveness chart

4.5 Naval Coalescence Tester

Once all specification, fit-for-purpose, compatibility and effectiveness testing was completed, results were evaluated in order to determine which additives would be submitted for NCT testing. The majority of the additives passed all testing, demonstrating that the additized fuel did not negatively affect the fuel's water separation or specification properties. Rather than incurring the cost and time of testing all additives, additives were down selected to represent the additives least likely to pass NCT. Additive A was selected for NCT because it was an ester-based chemistry. Ester based additives were not previously tested by the Navy and were suspected of having poor water separability. Additives C and K failed the diesel MSEP and demulsification tests, which are indicators of possible poor water separability. Fuels additized with a single of the three additives at two times the manufacturer's recommended concentration were submitted for NCT. Differential total water readings were taken of water saturated fuel prior to free water injection and subtracted from total water reading post-coalescer. A passing fuel will have no more than three sequential differential readings above 100 ppm. Differential water readings for NCT of fuels containing additive A and C are shown in figures 4 and 5 respectively. Additives A, C, and K passed NCT at 2x recommended concentration. Full NCT test report for these fuel denoted by references d, e, and f.

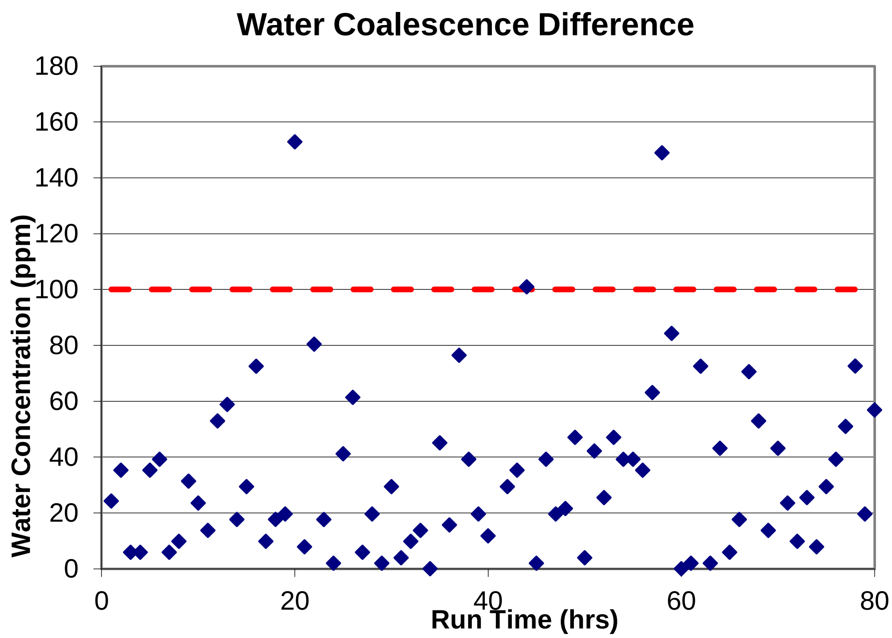

Figure 4. NCT differential water readings between saturated and downstream results from fuel with 2x recommended dosage of Additive A. Red dashed line denotes maximum acceptance level of 100 ppm.

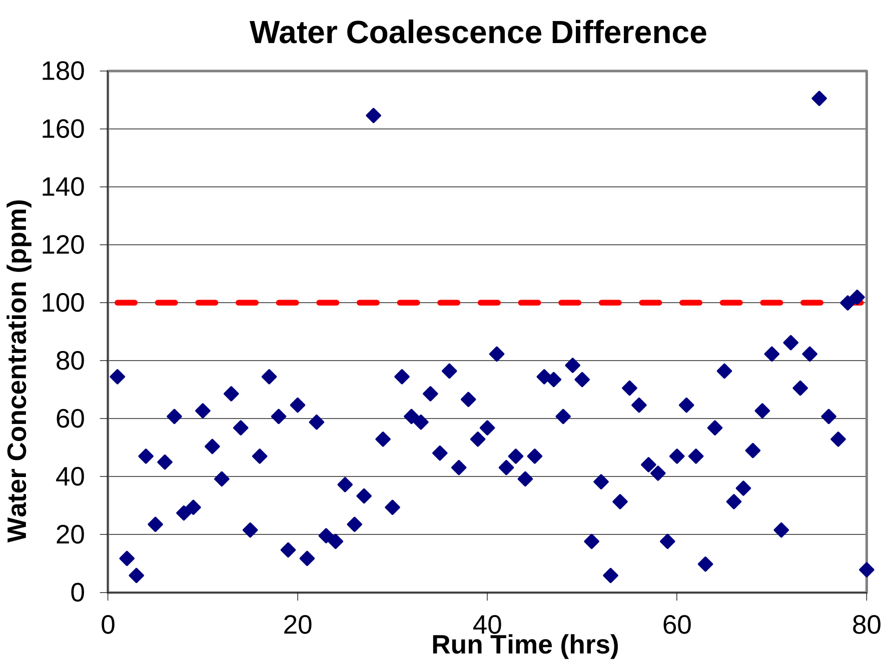

Figure 5. NCT differential water readings between saturated and downstream results from fuel with 2x recommended dosage of Additive C. Red dashed line denotes maximum acceptance level of 100 ppm.

5.0 CONCLUSIONS

1. Additive C was determined from the specification and FFP testing to have the greatest adverse impact on water separation. This additive was selected to be the surrogate for the other additives for NCT testing. Additive C at twice the recommended additive treat rate passed the NCT test.
2. Additives A, C, D and H-K passed all the requirements of the test protocol.
3. Additives B, E, F, and G passed all tests with the exception of the flashpoint. Discussions with the manufacturers of those additives have indicated that changing the additive's solvent to a less volatile compound with mitigate the test failure.

6.0 RECOMMENDATIONS

1. Additives A, C, D and H-K continue testing in accordance with MIL-PRF-xx646 (reference 6). It is recommended that results of this report be applied toward the qualification of these additives.
2. Request manufacturers of additives B, E, F and G resubmit a candidate with a less volatile solvent for retesting.

7.0 REFERENCES

a. MIL-PRF-xx646, Performance Specification, Additive, Lubricity Improver, Diesel, Draft.
b. NF&LCFT Report 441/09-001 Ultra Low Sulfur Impact on F-76
c. SWP44FL-005 Appendix A-13 Naval Coalescence Tester
d. NF&L CFT Report 441-12-015 Laing, C. Navy Coalescence Test Report. June 20, 2012
e. NF&L CFT Report 441-12-017 Laing, C. Navy Coalescence Test Report. June 20, 2012
f. NF&L CFT Report 441-13-xxx Dickerson, T. Navy Coalescence Test Report. July 20, 2013. Report in progress.

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APPENDIX A: Additional Laboratory Data

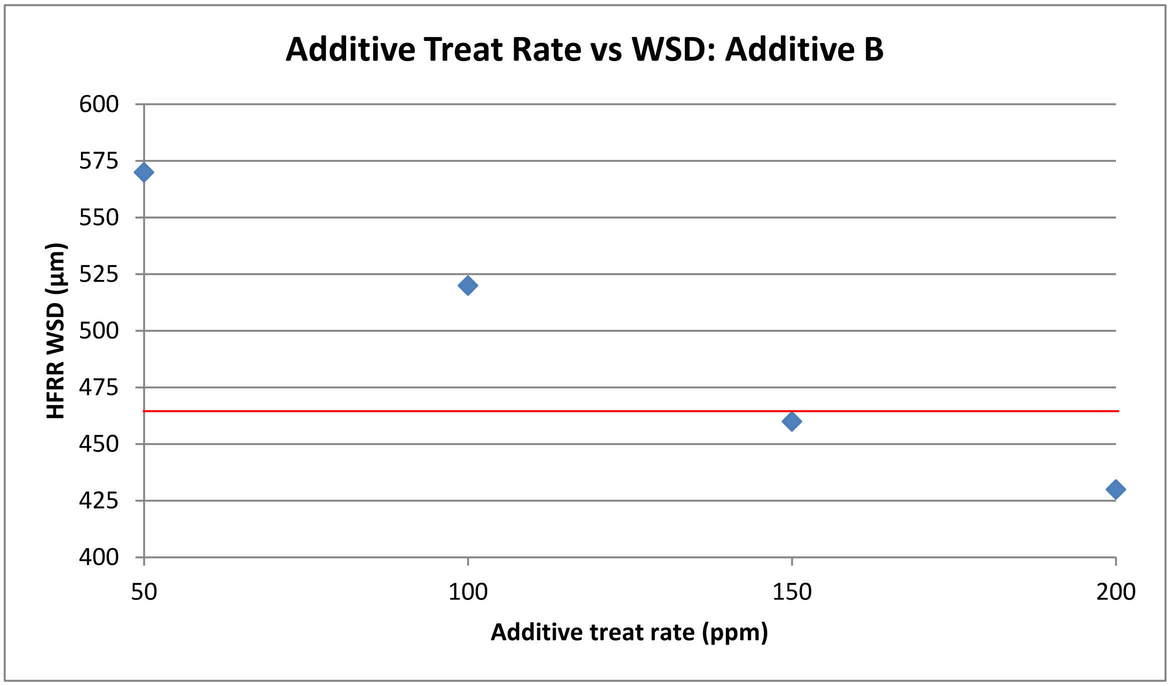

Figure A-1. Additive B Effectiveness Chart

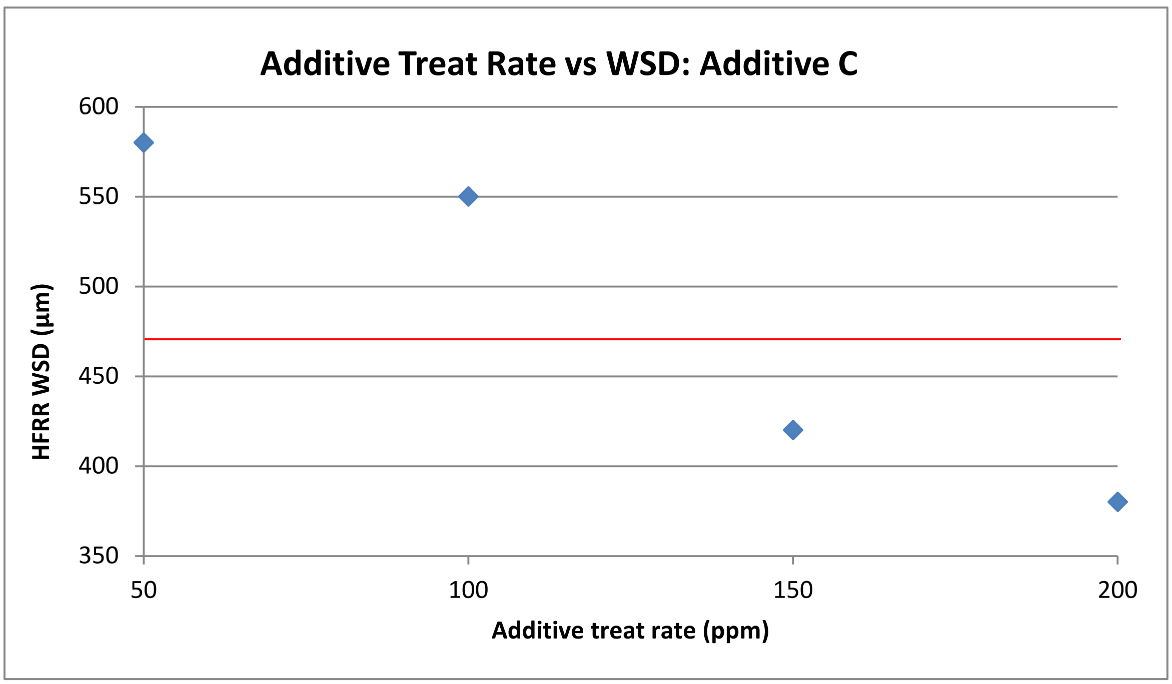

Figure A-2. Additive C Effectiveness Chart

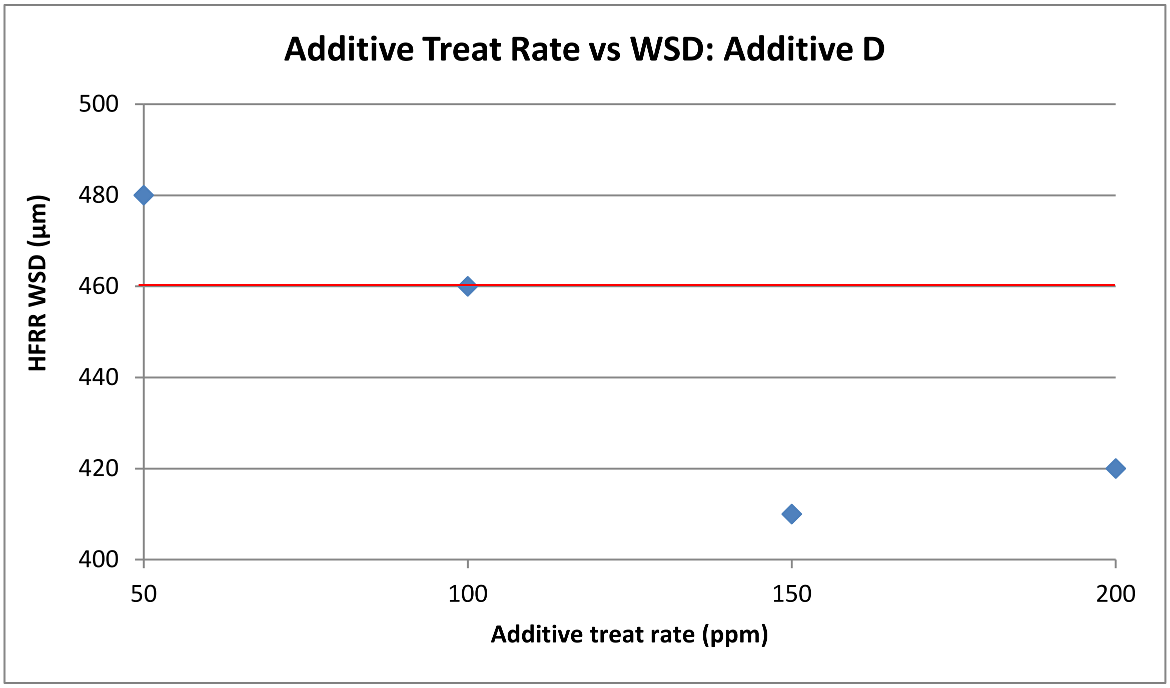

Figure A-3. Additive D Effectiveness Chart

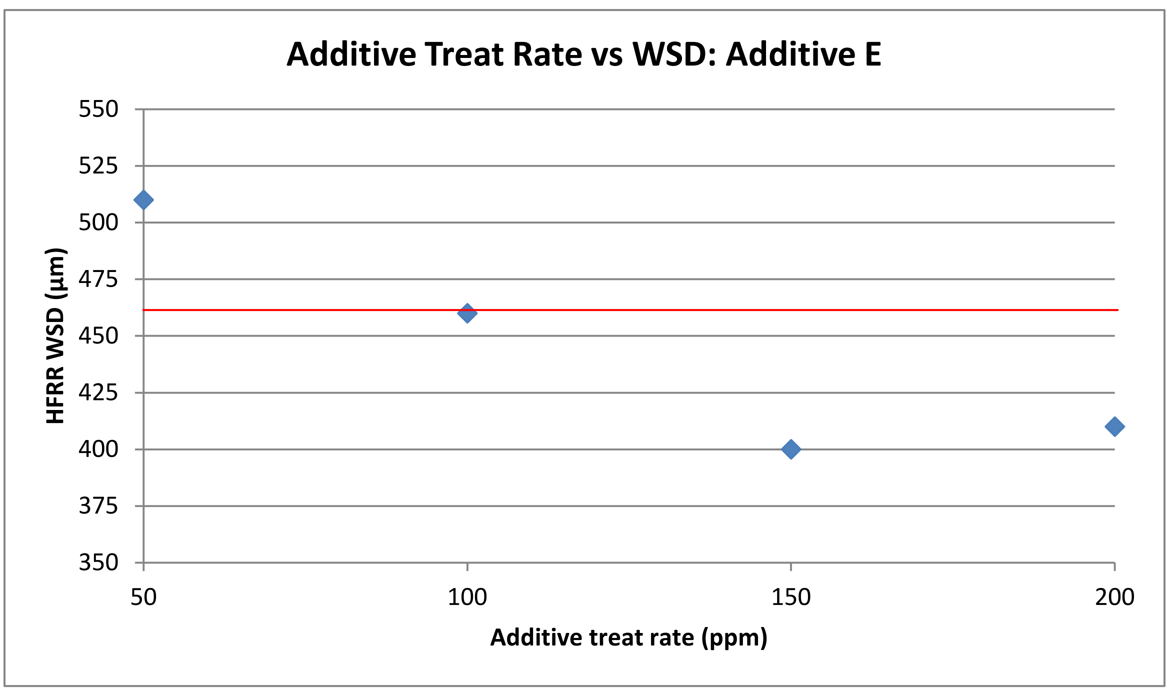

Figure A-4. Additive E Effectiveness Chart

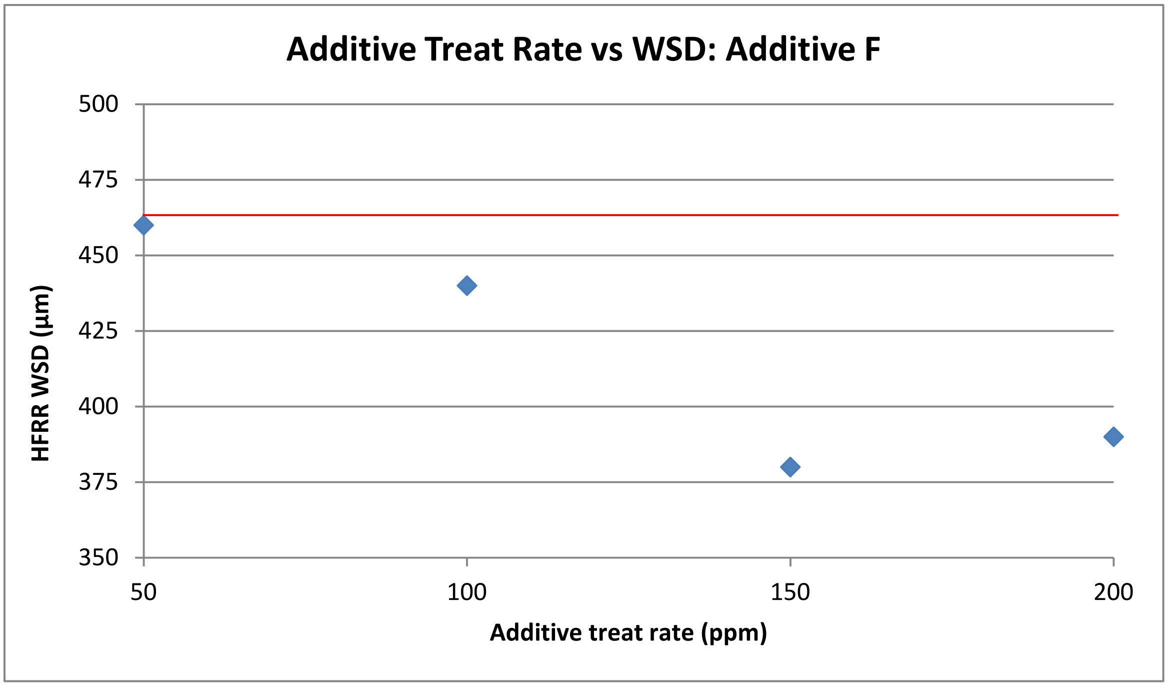

Figure A-5: Additive F Effectiveness Chart

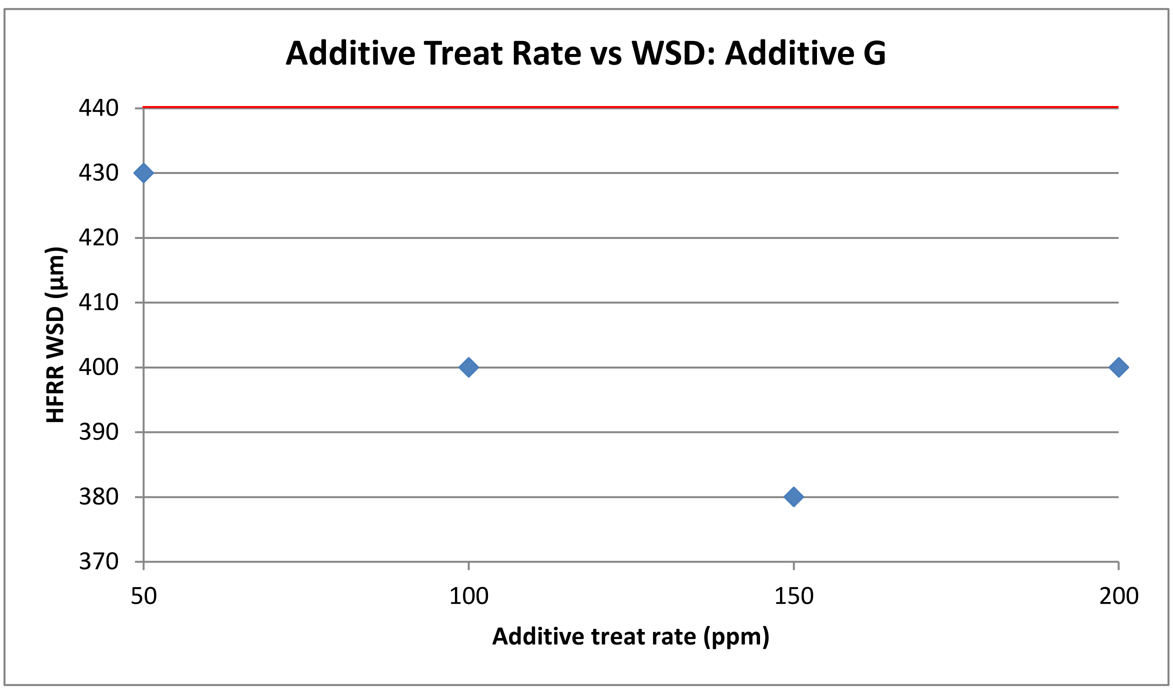

Figure A-6. Additive G Effectiveness Chart

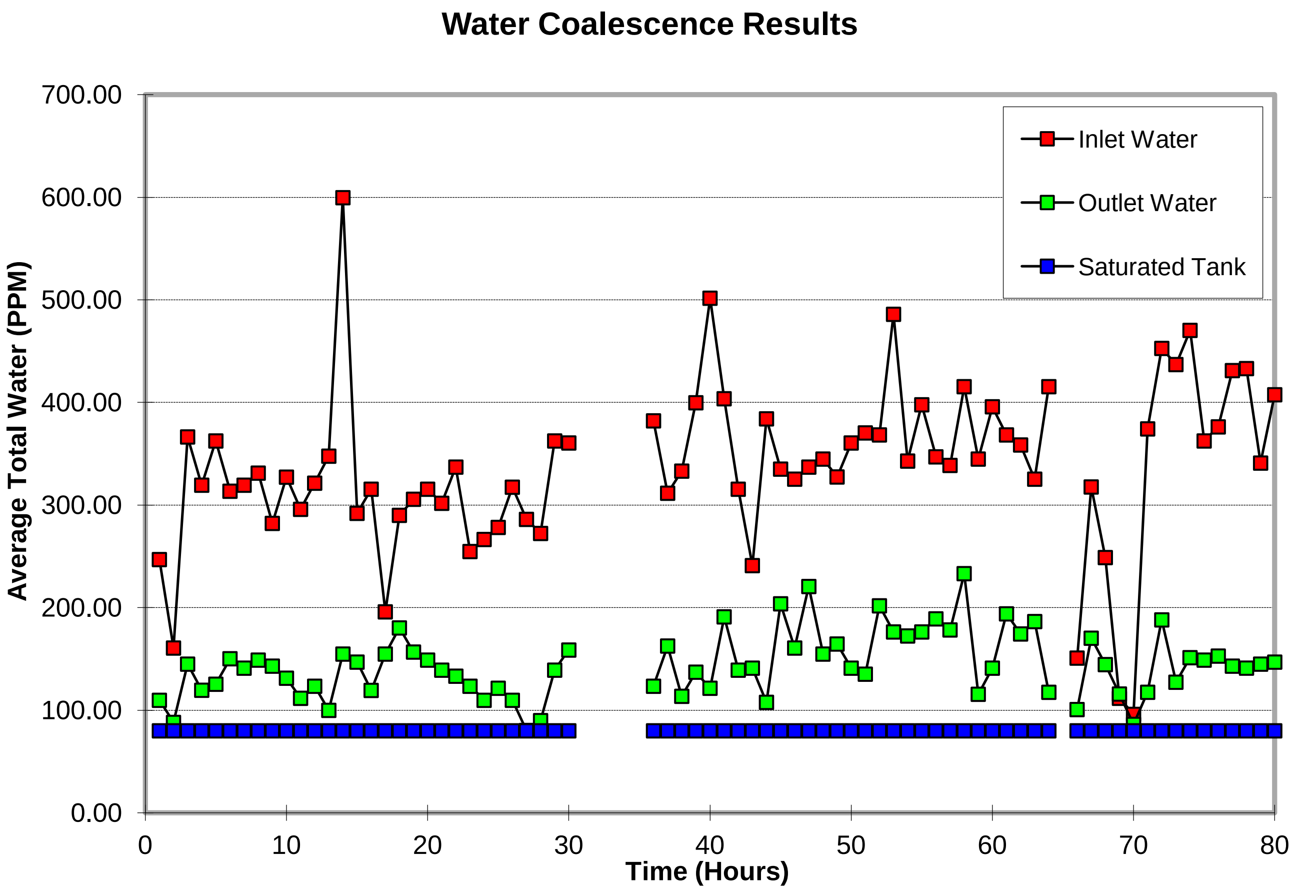

Figure A-7. NCT Results for Additive C- Passing results

APPENDIX B: Naval Coalescence Tester

NAVAL COALESCENCE TEST (NCT)

B.1 SCOPE

B.1.1 Scope. This appendix outlines the protocol used to simulate water coalescence effects in aviation and naval distillate fuels. This appendix is a mandatory part of the specification. The information contained herein is intended for compliance.

B.1.2 Summary of the method. The NCT is a fit-for-purpose test, which allows for fuel flow through a fuel coalescer while using a small amount of fuel. The objective of this test is to determine the water shedding, or coalescence properties, of the candidate additive on the filter. A known amount of free (undissolved) water will be injected upstream of the test element and upstream and downstream free and dissolved (total) water levels will be measured and compared to the saturated water level in the fuel. A passing test shall give downstream free water measurements that are comparable to that of the saturated water level of the fuel.

B.2 MATERIALS AND APPARATUS

B.2.1 Apparatus. The test apparatus is shown schematically in Figure B-1. It consists of the following:

a. Nitrogen sparger

b. Test capsule

c. Kent Scientific syringe pump

d. Control system and panel

e. Localized valves, including the rotameter control valve, fuel feed valves, discharge valves, and air supply valve

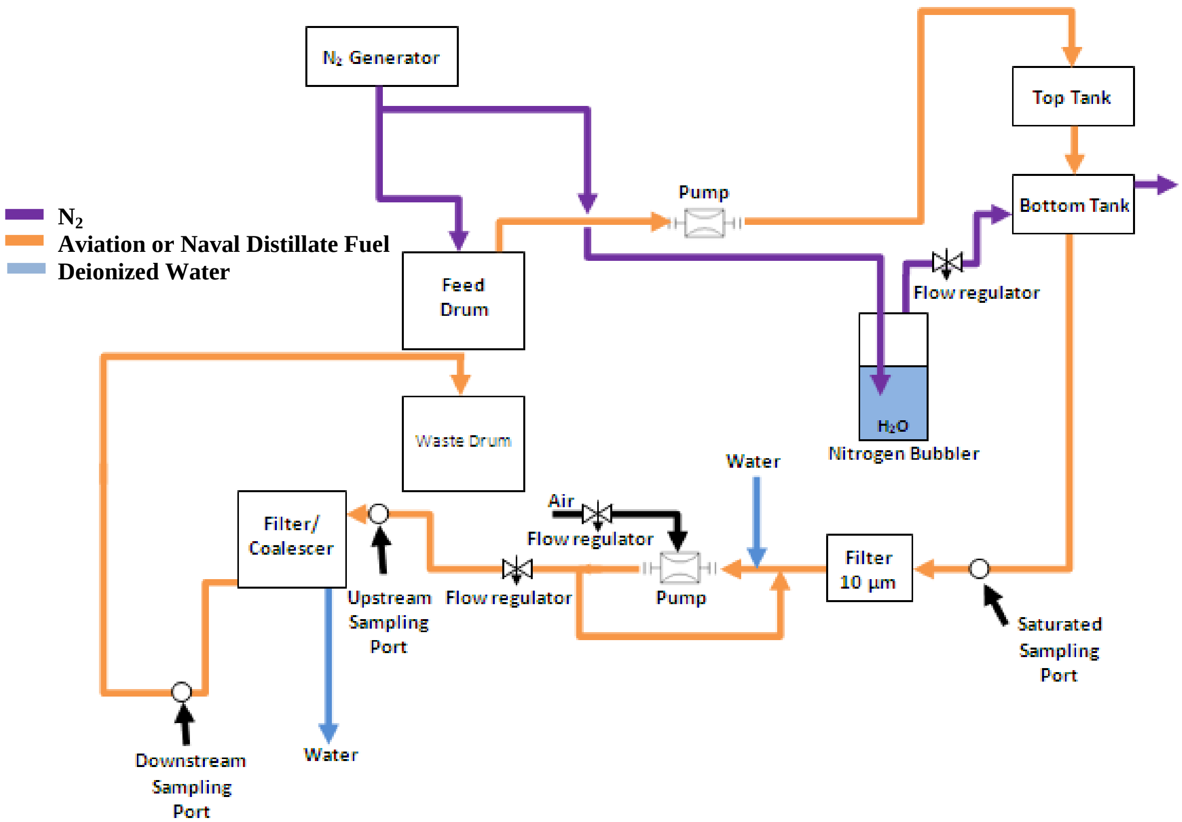

FIGURE B-1. NCT schematic.

B.2.2 Materials. The materials shall consist of the following:

a. 100 gallons of referee fuel with candidate additive
b. Latex gloves
c. Syringe, 1 mL
d. Solvent dispenser squeeze bottle, 500 mL
e. Graduated cylinder, 500 mL
f. Water injection needle, #33
g. Syringe, 50 mL
h. Tweezers, flat tipped
i. Distilled water

B.3 TEST PROCEDURE

B.3.1 Test preparation. A new, epoxy/phenolic lined 55-gallon drum is filled with filtered test fuel. A steel 8-inch extender tube is attached to the large bung of the drum and a stainless steel, air driven pump is placed into the extender tube. The drum pump outlet line is then attached onto the 90-degree stainless steel fitting on the small bung of the drum and the additized referee fuel is recirculated through the drum pump at 7 gallons per minute (GPM) for 3 hours. If no flowmeter is available, then the air on the pump is turned halfway on and then slowly closed until the flow contains no bubbles. The inlet and outlet drum lines are then attached to the portable filter separator apparatus and the additized referee fuel is recirculated for 16 hours. Finally, a 1-gallon sample of additized referee fuel is collected and submitted to a laboratory for filtration time and particulate matter analysis.

B.3.2 Test operation. The NCT shall be cleaned and built up prior to running each additized fuel and a new NCT element shall be inserted into the capsule holder prior to beginning a new test. The separator is tested for water beading efficiency, and the system is flushed with additized referee fuel for 60 minutes. The nitrogen generator must be properly configured and distilled water shall be filled to the 800-mL mark in the 1000-mL graduated cylinder nitrogen sparger. The injection needle is inspected and the fuel flow set to 33 mL/minute. The water injection valve is then opened and observed until water drops appear at the outlet of the filter separator. Time is recorded as soon as the first drop is seen. The syringe pump is set to approximately 8.25 μL/min and the fuel flow is maintained at 33 mL/minute using the appropriate valves. This results in an undissolved water concentration of 250 ppm in the test fuel. Normal test duration is 80 hours. The following shall be collected:

a. The inlet total water content is measured, in triplicate, according to ASTM D6304, once per hour from a sample obtained from upstream sampling port.

b. The outlet total water content is measured, in triplicate, according to ASTM D6304, once per hour from a sample obtained from downstream sampling port.

c. The total water content shall be measured, in triplicate, according to ASTM D6304, once per hour by extracting a sample from the saturated water port. Differential pressure across the filter/separator and fuel temperature shall be recorded every hour. Accumulated water in the housings shall be drained and bled off as needed, and documentation shall continue for 80 test hours.

B.4 DATA EVALUATION

B.4.1 Acceptance criteria. The test shall be considered passing if the difference between the outlet total water concentration and saturated total water concentration does not exceed 100 ppm for 4 consecutive hours. A differential pressure across the filter/separator shall remain less than 3 pounds per square inch (psi) during the entire 80-hour test.

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14. ABSTRACT
The objective of this investigation is to expand and diversify the number and type of diesel lubricity additives that are available for use in the Navy's distillate F-76 fuel. By increasing the number of approved LIA's and diversifying the chemistries of these additives, the Navy will be prepared to compensate for the decrease in lubricity as ultra low sulfur fuels shift into focus while still retaining the fuel's water separability traits. Test and evaluation of the additives focused on three main sections of the evaluation segment of the Navy draft fuel and additive approval process: specification, fit-for-purpose, and rig testing. A local ultra low sulfur diesel (ULSD) was clay treated in order to mimic ultra low sulfur fuels that would potentially be acquired by the Navy in the future. Additives were tested at two times the manufacturer's recommended treat rate for both specification and fit-for-purpose testing, and several of these tests focused on the effects of the additives to the fuel's water separation properties. Rig testing was conducted on the Naval Coalescence Tester only with additives that had failed crucial water separability testing such as demulsification and diesel MSEP. It was determined that if the worst possible case passed NCT testing, then all fuels additized with LIA that produced passing specification and fit-for-purpose results would pass as well. Eight of the eleven additives tested exhibited favorable properties for use in the Navy's distillate F-76, while the three remaining additives were found unsatisfactory due to their low flash point.

15. SUBJECT TERMS
F-76, Fuel Lubricity, Lubricity Improving Additive

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