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Development of a Fuel Lubricity Haze Test (FLHT) for Naval Applications

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14. ABSTRACT

Highly processed diesel fuels are now monitored for lubricity by specially developed mechanical tests. Because these mechanical tests are imprecise, difficult to run, expensive, and difficult to relate to actual field conditions, a chemical test for lubricity has been developed. This Fuel Lubricity Haze Test (FLHT) has been miniaturized, and can be run in the laboratory or field. It has been found to be an objective, precise test that is capable of determining the complete range of fuel lubricities. It can also detect small changes in lubricity, and is very responsive to fuel lubricity additives. The FLHT involves the addition of a small volume of aqueous base to a small volume of fuel, followed by mechanical shaking for a short time, and after a specific settling time the fuel haze is determined by a hand held turbidimeter. The FLHT has been related to current mechanical lubricity testers and by extension to components such as diesel fuel pumps and injectors. No interferences to the FLHT method were found, including fuel color, most fuel dyes, the presence of water and seawater, the addition of biofuel or synthetic fuels, or typical fuel additives.

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CONTENTS

ACRONYMS

ACS	American Chemical Society
AD	Arctic Diesel
ASTM	American Society for Testing and Materials
BF	Baseline Fuel
BOCLE	Ball on Cylinder Lubricity Evaluator
CI	Cetane Improver (additive)
CT	Clay Treated
DCI	DuPont Corrosion Inhibitor (additive)
DESC	Defense Energy Supply Center
EPA	Environmental Protection Agency
FLHT	Fuel Lubricity Haze Tester
FOA	Fuel Oil Additive
FSII	Fuel System Icing Inhibitor (additive)
FT	Fisher Tropsch
FY	Fiscal Year
GC	Gas Chromatograph
HFRR	High Frequency Reciprocating Rig
ID	Identification
JF	Jet Fuel
JFTOT	Jet Fuel Total Oxidation Tester
LCO	Light Cycle Oil
LSDF	Low Sulfur Diesel Fuel
MDFI	Middle Distillate Flow Improver (additive)
MIL-DTL	Military Detail
MSC	Military Sealift Command
NTU	Nephelometric Turbidity Units (dimensionless)
NRL	Naval Research Laboratory
SD	Standard deviation
SDA	Static Dissipater Additive
SE	Standard error
SLBOCLE	Scuffing Load Ball on Cylinder Lubricity Evaluator
SwRI	Southwest Research Institute
ULSD	Ultra Low Sulfur Diesel
USCG	United States Coast Guard
WSIM	Water Separation Index Modified

DEVELOPMENT OF A FUEL LUBRICITY HAZE TEST (FLHT) FOR NAVAL APPLICATIONS

1.0 BACKGROUND

Fuel lubricity is the term that denotes the ability of fuel to reduce friction and wear to moving metal parts and has been of interest to commercial and military users particularly for the past several decades. This is because environmental regulations have become stricter and now even diesel fuels must undergo intense refining processes and treatments in order to remove sulfur species which contribute to sulfur oxide emissions. In the process of such sulfur removal trace surfactant components that provide the naturally occurring fuel lubricity are also removed. The resulting low lubricity fuels can exhibit wear and sometimes mechanical failure in critical fuel system components such as fuel pumps, fuel injectors, and flow controllers.

Several mechanical tests have been developed to test lubricity in fuels but these tests are quite difficult to perform and invariably have very poor precision. Another potential solution used by the commercial marketplace to ensure adequate lubricity has been to add lubricity enhancing additives, which are surfactants consisting of organic acids or esters or other mild surface active materials in order to restore this degraded fuel property. The mechanical lubricity tests have also been used to monitor the addition of these surfactants and this can be another area where these tests may not be useful or applicable. Since the mechanical tests tend to be unresponsive to lubricity additives, this approach can result in adding too much.

In 2000 research was conducted to determine the chemical nature of lubricity of diesel fuels. [1, 2] Hughes developed a chemical test for diesel fuel lubricity that included a base extraction, acidification, a back extraction, and analysis with gas chromatography/mass spectroscopy in order to qualitatively and quantitatively determine the most surface active species in the fuel. The observation was made that during the base extraction and acidification steps, fuels with good lubricity consistently had a visible haze while fuels with poor lubricity consistently lacked the visible haze and appeared to be clear.

This led to efforts in 2003 to investigate this observation by scaling down the original extraction volumes and trying to develop a scheme to visually evaluate the haze caused by the presence of surface active components that formed water in oil emulsion haze in a fuel of a given lubricity [3]. The effects of different concentrations and ratios of reagents were explored, the acidification step was eliminated, and standard viewing conditions were specified. However, in 2004 the decision was made to switch from a visual evaluation of the haze that used a gray scale to a more objective evaluation using a small turbidimeter to measure the haze in a more quantitative way. This report describes the final development of this potentially important test method and also reports the first extensive data of a wide variety of fuels and fuel types including severely processed diesel fuels, bio diesel, coal derived liquid fuels, solvent mixtures, and additives. In addition, a number of possible interferences were investigated including fuel color, fuel dyes, the effects of seawater contamination, and the effects of glassware cleanliness. This report focuses on poor lubricity diesel fuel that has generally been observed to cause rather severe component wear effects such as adhesive and scuffing wear on fuel-wetted moving parts

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of pumps and fuel controls. Although the FLHT was developed to screen middle distillate diesel fuels, it also appears to have some possible usefulness when evaluating middle distillate jet fuels.

In the final year of development (FY 2006) a test plan was developed (see Appendix A). The primary task in that plan was to develop an objective quantitative method and to determine its precision. The precision of the test was to be compared to the three standard mechanical tests, the Scuffing Load Ball-on-Cylinder Lubricity Evaluator (SLBOCLE) (ASTM Standard Test Method D6078), the High-Frequency Reciprocating Rig (HFRR) (ASTM Standard Test Method D6079) and the Ball-on-Cylinder Lubricity Evaluator (BOCLE) (ASTM Standard Test Method D5001). This latter test was developed only for jet fuel use and is included in the matrix for comparison purposes only. The first two tests were specifically developed for diesel fuel evaluation.

An additional task in the FY 2006 test plan was to investigate any potential interferences on the FLHT final test method. This included an evaluation of all possible middle distillate diesel fuel additives that are known to be present in Navy military specification diesel fuel (NATO F-76 MIL DTL 16884), singly and in combination. The final task was to determine the linearity of the FLHT for intermediate additive concentrations using several poor lubricity fuels and a lubricity enhancing additive from the commercial marketplace.

One goal of the test plan was to determine shipboard acceptability of the method since it employs sodium hydroxide at 0.2 M concentration. This was accomplished by corresponding with the applicable ship safety offices. There appears to be no impediment to the use of pre packaged small quantities of sodium hydroxide and there are standard shipboard procedures in place for its safe disposal.

2.0 EXPERIMENTAL AND FUELS

2.1 Summary and Detailed Description of the Final FLHT Method

This test is intended to evaluate the lubricity of diesel fuel (number 1 or number 2) regardless of sulfur content, by means of extracting the surfactant materials and forming an emulsion. The emulsion is measured on a small bench-top turbidimeter with a range of 0.00 to 1,100 nephelometric turbidity units (NTU). The log of the turbidity reading is used to compare fuels. Higher lubricity fuels will have more surfactants and thus a greater amount of emulsion formed and higher turbidity ratings.

Using disposable borosilicate serological pipettes, 12.0 mL of the test fuel and 2.0 mL of 0.2 M NaOH (in 50/50 methanol and water solvent) are transferred into a turbidimeter vial (e.g., LaMotte 2020 Tube, Code 0286, Chestertown, MD). The sample vials are 15 mL optically selected glass turbidity tubes specially designed for this purpose. The Teflon lined caps, which must be purchased separately, are then tightened on the sample vials.

The sodium hydroxide and methanol are ACS Grade, and the water is from a Milli-Q system or equivalent.

If a vial is new, no cleaning is necessary. If a vial is being reused, it should be rinsed in heptane and trisolvent (1:1:1 methanol/acetone/toluene) and filled with deionized water and allowed to stand for 24 hours followed by drying in an oven.

The vial is placed in a vortex mixer (e.g., Fisher #12-810-3). In order to ensure that a reproducible emulsion is produced a rubber septum sleeve (about 1 inch diameter and about 2 inches long) is placed over the top of the vial so that the distance between the base of the mixer and the larger diameter opening of the septum cap is 2 3/8 inch. The septum cap is clamped at 3 3/16 inch from the base of the mixer to the bottom of the clamp (around the smaller diameter end of the septum cap). This arrangement should allow free rotation of the vial in the mixer during the mixing and also the formation of a vortex in the liquid and good emulsion formation.

The mixer speed is pre-set to 1600 rpm. The sample is allowed to mix for 30 seconds (plus or minus 5 seconds). When the time has elapsed, the mixer is turned off and the vial is removed.

The outside of the vial is carefully cleaned with a lint free tissue. After waiting 15 minutes (plus or minus 1 minute), the vial is placed in the turbidimeter (e.g., LaMotte 2020, Code 1799, Chestertown, MD). Immediately record the turbidity.

Given the optical path height in the turbidimeter used, the fuel haze layer is being analyzed. In earlier work using a gray scale, both the fuel and the water layer were analyzed initially and finally only the fuel layer was assessed for haze by the gray scale method.

Using this final version of the FLHT, good lubricity fuels will have turbidity readings of greater than 10 (or log 10 = 1.00). Poor lubricity fuels will have turbidity readings of less than 10 (or log 10 = 1.00). The turbidimeter should be calibrated daily with the LaMott standard (10 NTU). A set of FLHT standards made of Isopar M and DCI-4A, for example, at concentrations to give turbidities of 10, 20 and 30 (or log 10 = 1.00, log 20 = 1.30, and log 30 = 1.48, respectively) when properly emulsified as described above should be considered and included in any future method development. This will ensure interlaboratory reproducibility and lack of bias.

This final test method actually does not have as good a precision or as high turbidity readings as the originally devised manual shaking method. However, the manual shaking method contained definite operator biases and in order to exclude this bias a mechanical shaking method was developed. The mechanical method precision is not as good as manual shaking, probably because the positioning of the vials in the vortex mixer is so sensitive and the mixing of the two layers in the vial is being done right on the lower limit edge of good emulsion formation. This turns out not to be problematic since the precision of poor lubricity fuels by the final FLHT method is still quite good and the good lubricity fuels have generally quite high turbidities and thus the poor precision does not impact the ability to sort pass and fail fuels. This will be discussed further in the Results and Discussion section below.

2.2 Description of All of the Test Samples in the Program

A complete list of all 203 samples prepared during the 5 year program, which was performed at 3 different laboratories, is included as Appendix B. This list is annotated in detail and coded with a letter code which is carried out in all of the data tables and figures. The actual listing of the samples is chronological and reflects some of the early goals of the test development and the evolution of the testing as improvements were made and new understandings were gained.

Samples 1, 2, 6, 13, 18, 26, 27, 37, 48, 63, 70, 74-80, and 157-161 (a total of 15 different fuels some in replicate) constituted the base fuels from which most of the test mixtures were made. These constitute a very wide range of lubricities and fuel types including high sulfur petroleum diesel, severely processed very low sulfur petroleum diesel fuels, processed tar sands fuels, biodiesels, Isopar M, Fisher Tropsch (FT) fuel, and several jet fuels.

The first 25 samples prepared generally focused on trying to include a very high volume fraction of a very poor lubricity fuel when developing the FLHT method. This line of sample preparation was abandoned in favor of making 10% increment blends of poor and good lubricity fuels to determine lubricities completely throughout the range of possible lubricity values. This work used samples 26 through 57 and 81 through 89 for several mixtures.

The biodiesel blend work was done using samples 58 through 63. The initial work with additives used samples 64 through 69 and 90 through 108, and 113 through 148. The base fuel samples are described in samples 74 through 80, and 109 through 112. Samples 149 through 157 were used near the end of the manual shaking method development and will be discussed below for their role in uncovering operator bias.

The remaining samples 158 through 203 were prepared in order to test out precision of the final method, possible additive interferences, and linearity of the method towards additives at the very end of the test program.

2.3 Specification Tests of Several Base Fuels

Appendix C contains the full specification test results for 9 of the petroleum derived diesel or jet fuels used as blending stocks throughout the program. These fuels are coded as B, BM, BN, BO, BP, BQ, BR, BU, and DA. Six of these are diesels and 3 are jet fuels. There were actually 15 fuels used as blending stocks and these are grouped in Table 2.3.1 along with a description of the fuel type. Although the full specification for 6 of these samples was not determined, two of them were very highly processed and poor lubricity petroleum and tar sands diesels (codes A and F), one was Isopar M (code M), one was a typical soy-derived biodiesel (code BF), and two were typical high sulfur DF-2 diesels (codes EV and EW).

Table 2.3.1 Base blending stocks and fuels for the entire study.

NRL ID Code	Sample ID	Type of Sample
A through A(5)	Arctic Diesel #1	Poor lubricity diesel
B through B(4)	Baseline Fuel #2	Good lubricity diesel
F through F(3)	Arctic Diesel #2	Poor lubricity diesel
M through M(3)	Isopar M	Isoparaffin solvent mixture
BF	Biodiesel from soy	Biodiesel
BM	Clay treated Baseline Fuel #2	Poor lubricity diesel
BN	Clay treated Jet A	Poor lubricity jet fuel
BO	Synthetic fuel from natural gas	Poor lubricity jet fuel
BP	Jet A	Poor lubricity jet fuel
BQ	ULSD diesel #1	Good lubricity diesel
BR	ULDS diesel #2	Poor lubricity diesel
BU	EPA No. 2 Diesel	Good lubricity diesel
DA	Mixture of diesels (F and BR)	Poor lubricity diesel
EV	SwRI No. 2 Diesel	Good lubricity diesel
EW	Arco No. 2 Diesel	Good lubricity diesel

The determination of good or poor lubricity of each of these samples was made on the basis of their SLBOCLE and HFRR results and the data will be introduced in the next section. The number in parenthesis in the Table above indicates separate aliquots of the same sample drawn at different times during the testing from a bulk storage container.

3.0 DISCUSSION OF RESULTS

3.1 Precision and Bias of the Final FLHT Method

There were 4 primary variables that were initially investigated in the test method development. These were sample size, partition coefficient (base to sample relative volumes), base strength, and times of emulsion formation and settling. A factor that was examined only near the final development of the method was the severity of mixing.

In order to establish the best test conditions, we must first define the entire range of possible lubricity. This has been done in this study by using a very good lubricity, high sulfur distillate diesel fuel, code B (also known as Baseline Fuel #2) which gives very high SLBOCLE weights near the maximum possible and very low wear scars on HFRR near the minimum possible for that method. Next, a poor lubricity, very low sulfur, highly processed diesel fuel such as code A (Arctic Diesel #1), or code BO (synthetic jet fuel from natural gas), or code M (Isopar M) was selected. These gave the lowest weights possible on SLBOCLE and nearly the maximum wear scars possible using HFRR. Finally, volumetric blends of very good and poor lubricity base stocks were made in 10% volume increments.

Using typical 10% volume increment blend series described, the amount of time needed to form the emulsion by manual shaking and the settling time were determined. Initially a wide range of manual shaking times between 30 seconds and 5 minutes was investigated. The shaking of the

vials was using strokes about 10 inches long and a repetition rate of about 1 second per shake. Selected data from this study are shown in Table 3.1.1 for mixtures of Fuel Codes A and B.

Table 3.1.1 Manual shaking time 5.0 minutes, fuel is %B in A, single determinations, NTU is dimensionless units (nephelometric turbidity units).

	100% B	80% B	60% B	40% B
Wait time, min.	NTU	NTU	NTU	NTU
3.5	1097	177	68	19
5	869	122	54	18
6.5	703	107	50	17
8	592	101	46	16
9.5	511	94	44	15
11	449	86	41	15
13	320	80	36	15
15	236	74	36	15
20	197	69	36	15
25	194	62	34	15
35	163	54	31	14
45	159	51	28	14
60	156	47	25	14

Manual shaking time 1.0 minute, fuel is %B in A, single determinations, NTU is dimensionless units (nephelometric turbidity units).

	100% B	70% B	50% B
Wait time, min.	NTU	NTU	NTU
10	936	279	57
15	820	228	33
20	716	169	39
25	643	145	41
30	616	136	42
35	569	108	41
40	501	129	41

Manual shaking time 30 seconds, fuel is %B in A, single determinations, NTU is dimensionless units (nephelometric turbidity units).

	100% B	70% B	50% B
Wait time, min.	NTU	NTU	NTU
10	1100	256	107
15	920	183	58
20	778	118	53
25	703	111	57
30	619	128	60
35	548	114	63
40	435	110	61

Inspecting this data, it is clear that regardless of manual shaking time, with 10.0 mL fuel and 2.0 mL, chosen and the base strength, 0.2M, chosen), that after 20 minutes there is no further change in haze rating for the 70% B and lower turbidity samples. In order to make the final standardized test as easy and rapid as possible, it is necessary to choose shaking and wait/settling times as short as practicable. A conservative shake time of 1.0 minutes was chosen initially for the manual shake method and a very conservative wait time of 10.0 minutes for all of the subsequent **manual shake** work reported in this paper. Later the **mechanical shake** time development data showed that the shake time could be reduced to 30 seconds and the wait time increased to 15.0 minutes for the remainder of the work reported.

There is one further problem in a timed method such as this and that is whether the operators will observe the timing precisely. Requiring long wait times tends to minimize this potential problem, since a test that relies on short, precise times is more liable to operator differences than a test with longer, more imprecise "timing." However, the final determination of standardized shake and wait times will involve the selection of the actual NTU reading that constitutes a pass/fail criterion. This will be discussed below.

The precision of the manual shake test was found to be excellent. Generally a standard error of about 15% was easily achievable throughout the range of turbidity, except for very low turbidity (< 15 NTU; <1.18 LOG NTU) where the precision could be poor as 50% standard error (SE = (SD/Mean) x 100%). However as shown in Table 3.1.2 the manual shake method could exhibit a clear operator bias through the range of turbidities.

The first 4 entries in the Table are for poor fuels with additives at various concentrations (see Appendix B for details). The final 5 entries in the Table (Codes EQ through EU) are for mixtures of good and poor lubricity fuels without any additives.

Large differences in operator averages for very high turbidity/lubricity samples (above 100 NTU) are really of no consequence. But for the 3 lowest turbidity samples, 2 samples gave differences which could be very problematic (Fuel Codes EM and EU) in terms of using the test to differentiate good and poor lubricity fuels. It was this data that led us to consider development of a mechanical shaking method to remove this operator bias.

Table 3.1.2 Comparison of Manual Shake mode of the FLHT between two operators using a "blind" sample set. Shake time of 1.0 minutes, wait time of 10.0 minutes.

Sample Code	**Operator #1 Average of 2 Runs**	**Operator #2 Average of 2 Runs**
EL	3.2	1.8
EM	18	6.7
EN	60	84
EP	273	310
EQ	320	304
ER	119	65
ES	60	50
ET	49	46
EU	16	3

The development of a mechanical mixing alternative to manual shaking began with the use of a standard vortex mixer. Initially, a method was developed using a clamp to hold the vial in place, but this proved to be unsuitable for eventual shipboard use as it still required an adjustment to the clamp by the operator. The final mechanical mixing method used a sleeve stopper also called a septum cap from Wheaton Scientific (part #224100-320), which is held at a set height above the vortex mixing well. The top of the sample vial fits into the sleeve stopper and the bottom of the vial fits into the vortex mixer so that the vial is loosely held vertically and can rotate freely during mixing. The height of the sleeve stopper is fixed so that a vortex is observed in the vial during the mixing.

This is the configuration of the FLHT method that was used during the final period of the program for the testing outlined in Appendix A. This method used a 30 second mixing and a 15 minute wait time. Earlier work using the manual shaking method (using a 1 minute shake time and a 10 minute wait time) will also be reported below. Much of that work was done to compare the FLHT with the mechanical tests, to show the method worked on a wide variety of fuel types, and to perform some of the earlier interference studies with dyes, darker colored fuels and seawater.

Table 3.1.3 The standard deviation of 7 different samples, each one run by two operators, each operator using 3 separate aliquots for a total of 6 determinations per sample.

Table 3.1.3. Precision and standard error % for the final FLHT method. Average of 6 runs.

Sample Code	**NTU Avg ± SD (SE %)**	**Log (Avg NTU)**
68% A + 32% EW	144 ± 44 (31%)	2.16
Z	124 ± 95 (76%)	2.09
BU	52 ± 25 (48%)	1.72
EZ	22 ± 8 (35%)	1.34
EX	17 ± 7 (40%)	1.23
EY	14 ± 2 (13%)	1.15
96% A + 4% EW	6.8 ± 1.1 (16%)	0.83

From the Table above it can be seen that the standard deviation (SD) improves steadily as the average turbidity value decreases. The standard error (SE %) is much worse than the original mechanical shaking method, especially for the higher turbidity samples. When the actual values of the manual shaking and **final** mechanical shaking methods are compared (see Table 3.1.4) it can be seen that the final mechanical method appears to be much less severe in the forming of the emulsion since the values are two to three times less for the latter method. This is probably contributing to the much poorer precision of the final mechanical shaking method. Essentially some of the method precision is sacrificed in order to ensure that there is no operator bias. In Table 3.1.5 three samples are used to compare the final mechanical shaking method to the initial friction mechanical shaking method for NTU values and precision. NTU values are much lower for all three samples indicating continued lessening of severity for emulsification formation. This also contributes to somewhat worse precision at higher NTU values.

Later the pass/fail criterion is discussed for the FLHT and compared to the accepted mechanical lubricity tests. In general the manual shaking method pass/fail was nearer to 20 to 30 NTU (on a log basis 1.30 to 1.48) and in the **final** mechanical shake method this pass/fail was closer to 10 NTU (on a log basis 1.00). This also was consistent with a less severe emulsion forming step regardless of the actual times of mixing and waiting before measuring the turbidity.

Table 3.1.4. Comparison of manual shaking and final mechanical shaking. Single determination for manual and average of 6 determinations for mechanical shaking.

Sample Code	**Manual Shaking NTU**	**Mechanical Shaking NTU**
EZ	61	22
E(2)	30	17
Z	168	124

Table 3.1.5. Comparison of Friction Mechanical shaking to Final Mechanical shaking. All samples are the average of 6 determinations. Average, SD and SE% are given.

Sample Code	**Friction Mechanical Shaking NTU**	**Final Mechanical Shaking NTU**
BU	181 $\pm$ 39 (21.5%)	52 $\pm$ 25 (48%)
96% A + 4% EW	14 $\pm$ 1 (6%)	6.8 $\pm$ 1 (16%)
E(2)	36 $\pm$ 4 (12%)	17 $\pm$ 7 (40%)

Using three samples (Code EX, EY, and EZ) that were all prepared to simulate fuels that would be considered to be just at the margin of good lubricity, the precision of the FLHT, the HFRR, the SLBOCLE and the BOCLE were each run twice in an attempt to compare the precision among all of these methods. The raw data for the duplicate runs using separate aliquots are presented in Table 3.1.6 for each test method. The actual sample aliquots are from a single source. In order to give a basis for comparing the 4 test methods a "precision" definition was adopted such that the difference in the two results for each test and sample is divided by the average of the sum of the two results and multiplied by 100. Thus the lower the value obtained for each sample and each test, the more "precise" that test method. Table 3.1.7 gives this "precision" calculation for each test and sample and the sum of the values for each test method.

The lowest and best "precision" value is for the BOCLE test. This is because the BOCLE was really developed as a very sensitive test method for jet fuel lubricity differentiation, and since these diesel fuel samples were made up to have fairly good lubricity, they would be considered very good lubricity by BOCLE standards and thus would be expected to give very low scars and very good "precision" by BOCLE. The problem with the BOCLE test is that even for very poor lubricity diesel fuels in many cases it continues to give very low scars and so it really cannot be considered as a valid test methodology for diesel fuels. The same can be said in reverse for SLBOCLE and HFRR which were developed for diesel fuels and cannot be used to evaluate jet fuel lubricity. However, as discussed below, using FLHT it may be possible to evaluate both diesel and jet fuels provided it is known which fuel type is being evaluated for lubricity.

Comparing the "precision" of the remaining three test methods in Table 3.1.7, we see that the FLHT is about twice as precise (for these good to marginal lubricity diesel fuels) as the HFRR

and that HFRR is about twice as precise as the SLBOCLE. The FLHT is thus about 4 times more precise as the SLBOCLE. All three of these tests are without operator bias, but FLHT is the most precise by far.

Table 3.1.6. Raw data for repeatability of two separate aliquots using 4 test methods. The pass for FLHT (final version, log NTU) is >00, the pass for BOCLE is ≤0.620 mm, the pass for SLBOCLE is ≥3,200 grams, and the pass for HFRR is ≤520 um.

Fuel Code	FLHT log NTU	BOCLE (mm)	SLBOCLE (g)	HFRR (um)
EX	1.34	0.592	3150	577
	1.34	0.602	3800	536
EY	1.30	0.496	2950	544
	1.36	0.533	3400	501
EZ	1.53	0.609	3150	577
	1.41	0.614	4000	510

Table 3.1.7. Comparison of "precision" repeatability. Lowest numbers are best "precision". All values are dimensionless.

Fuel Code	FLHT	BOCLE	SLBOCLE	HFRR
EX	0	2	19	7
EY	5	7	14	11
EZ	8	1	24	12
Sum	13	10	57	30

3.2 Tests of Possible Interferences to FLHT

After the manual shaking method with turbidity quantification had been developed and the precision and bias determined, a series of experiments were performed to begin to assess the potential for interferences to the FLHT method. At this point, since the FLHT was an optical method the most important possible interferences were fuel dyes and dark fuels. In addition since FLHT was being developed as a possible field method in addition to a laboratory method, the effect of temperature of the fuel on FLHT was investigated. Finally, the effect of entrained seawater on the FLHT method was investigated since many field samples are taken that include water or seawater. In past lubricity studies with low sulfur diesel fuels seawater it was found to exert a small lubricity enhancing effect on fuel samples when tested in mechanical testers. It is not known whether or not this lubricity effect in the test methods actually enhances lubricity on actual components in the field.

Dye Effect. The most common diesel fuel dyes are red and blue. These dyes are normally added to fuels at concentrations of 10 to 30 ppm. When added to fuels that are light yellow to clear, they produce the desired color that is easily observed by the naked eye. Two fuels were chosen and two dye concentrations for these two dyes. The data are given in Table 3.2.1. It can be seen that in the low haze fuel (Code A) that there was a negligible effect of either dye at both concentrations. For the high haze fuel (Code W) there was also a negligible effect of either dye

at both concentrations. Thus for typical dyes and concentrations there is no interference with FLHT turbidity measurements.

Temperature Effect. The temperature of one high haze sample (Code W) was varied down to 10 degrees C and up to 30 degrees C to simulate the effect of fuel samples in the field that might not be temperature controlled. Once the sample and the extracting base solution were at temperature the emulsion was formed and the sample held at the desired temperatures (10 or 30 degrees C) until the turbidity was measured. It was found from these limited experiments that there was no statistically significant variation (outside of typical manual precision) in the FLHT results throughout this temperature range.

Water Effect. Two aliquots of a low haze rating fuel (again, Code A) were prepared. One contained 50% v/v seawater and the other 0.5% v/v seawater. These samples were thoroughly mixed by manual shaking and immediately a 10.0 mL aliquot was taken and the base added. The manual hand shaking method was employed to form the emulsion. It was found that there was no effect on the turbidity readings of either of these samples compared to Code A fuel without any seawater present. Thus we conclude that the presence of emulsified seawater in any sample will not cause any interference in the FLHT rating.

Table 3.2.1**.** Results of FLHT manual shaking turbidity for red and blue dyes.

Sample Code	**Dye Concentration (ppm)**	**Log NTU**
A	0	0.30
W	0	1.99
A	16 (Blue)	0.60
A	32 (Blue)	0.60
W	16 (Blue)	2.00
W	32 (Blue)	1.91
A	16 (Red)	0.00
A	32 (Red)	0.00
W	16 (Red)	1.90
W	32 (Red)	1.85

Color Effects. Next two sets of blends were made with dark (ASTM D1500 color ≥3.0) fuels. These dark blending fuels were refinery blending stocks of unhydrotreated light cycle oils and are not found in the sample Appendix B. They are coded as LCO 2421 and LCO 84-20 and were filtered through glass fiber filters before blending. Since these dark fuels are also very high in polar organics and surfactants it is expected that their lubricities would be excellent (high) and that their FLHT ratings would also be very high. The data in Table 3.2.2 for the neat LCO stocks show that the FLHT ratings are indeed very high. First 10-15% v/v of LCO 2421 is added to low haze Code A fuel to obtain a color of 3.0. Then about 15-20% of LCO 2421 is added to low haze Code A to obtain a color of >3.0. This is repeated using the high haze Code W fuel.

Next mixtures were made of Code A and Code W fuels with LCO 84-20 which is much lighter in color than LCO 2421 and also has a much lower FLHT rating. The mixtures were made to color rating of 3.0 and >3.0 in both fuels. The FLHT ratings for all 8 mixtures indicated that using

fuels with colors as high as 3.0 to 4.5 (ASTM D1500 rating) did not prevent the turbidimeter from reporting reasonable values. Thus dark fuel color (below 4.5) does not cause interference in the use of the FLHT method in assessing lubricity.

Reused/Clean Vial Effects. Next, surface effect of the glass turbidity sample containers was investigated for possible interferences of the FLHT manual method. During the original development of the method before turbidity rating was introduced, very inexpensive vials were the sample containers. These were always used new from the supplier and not cleaned before use. They were also disposed immediately after a single use. At one point, in an attempt to reuse these vials, they were cleaned with a soap and water solution, followed by water rinse and drying. When the cleaned vials were re-used, false passes resulted when visually evaluating low lubricity fuels.

The turbidity vials are much more expensive and so development of a cleaning method was of interest so that the vials could be reused. The cleaning procedure consisted of rinsing the inside of the tubes and Teflon lined caps with heptane to remove any fuel from the surface, followed by trisolvent (1:1:1 v/v of toluene, acetone and methanol) to remove any residual polar surfactants from the glass surface. Finally, the vials and caps were rinsed with deionized water and allowed to soak in deionized water for about 24 hours. After this, the vials and caps were air-dried and inspected for any surface residue before reuse.

Table 3.2.2. Results of the manual FLHT rating using "dark" ASTM D1500 color fuel blends. Recall that the pass for FLHT Log NTU is estimated >1.48 for the manual method.

Sample Code/Description	**ASTM Color**	**Log NTU**
LCO 2421	7.5	>3.04
A	<0.5	0.00
W	<1.0	1.86
A + 10-15% v/v LCO 2421	<3.5	1.3
A + 15-20% v/v LCO 2421	4.5	1.81
W + 10% v/v LCO 2421	3.0	2.25
W+ 15% v/v LCO 2421	4.5	2.36
LCO 84-20	4.5	2.42
A + 50% v/v LCO 84-20	<3.5	1.82
A + 70% v/v LCO 84-20	4.0	2.25
W + 45% v/v LCO 84-20	3.0	1.80
W + 60% v/v LCO 84-20	<4.0	2.12

The tubes that were washed using the above method were compared to new tubes by testing a series of blends of Code B and Code S fuels in 10% volume increments. The FLHT turbidities were identical within the precision of the method for both new and cleaned vials.

A third set of tubes was compared to these two sets. This third set had been used once and then simply washed with deionized water only and allowed to air dry. The readings from this water-rinsed set were fairly close up to NTU rating of about log 20 = 1.30. Above this turbidity value, the water-rinsed set gave generally much lower turbidity. This is probably due to scattered light absorption of the more poorly cleaned surfaces. See Table 3.2.3 for the results.

Thus, although vial surface cleaning exerts a lowering effect at higher turbidity ratings, this should not interfere with the usefulness of the method provided that standardized cleaning procedures are in place, or reuse of sample vials is prohibited, or if the pass criterion for the method is set low enough. This latter solution to vial surface effects on turbidity may actually be feasible since in the final mechanical shaking version of the FLHT, the pass criterion will be able to be set at a low value. This will be discussed in 3.4 below.

Table 3.2.3. Comparison of new vials with properly and improperly cleaned vials for the entire range of turbidity readings from the blend of Code B and Code S fuels in 10% volume increments. Manual shake method used to generate values.

Sample Code	**Log NTU, New Vial**	**Log NTU, Well cleaned, used vials**	**Log NTU, Poorly cleaned, used vials**
CD	0.30	0.30	0.30
CC	0.48	0.70	0.60
CB	0.90	0.90	0.90
CA	1.08	1.15	1.00
BZ	1.30	1.46	1.23
BY	1.60	1.67	1.53
BX	1.92	1.99	1.79
BW	2.28	2.32	1.96
BV	2.63	2.58	2.22

Additive Effects. The final effort in investigating potential interferences to the FLHT method was carried out near the end of the program, after the final mechanical shaking was instituted and after the changes to shaking time (30 seconds) and wait time (15 minutes) were made. A large matrix of typical fuel additives in a typical additive-free Jet A (Code BP(2)) or in a typical low lubricity diesel fuel (Code A(4)) was evaluated.

In the jet fuel the only military additive tested was Fuel System Icing Inhibitor (FSII) which is diethylene glycol monomethyl ether. This was included because it is a very polar additive, added at rather high concentrations (0.10% v/v minimum is typical). The military lubricity additives to jet fuel were not tested since these additives are easily determined by FLHT and do not pose an interference problem. The Air Force jet fuel additives Static Dissipator Additive (SDA) and the Thermal Stability Additive (JP-8 + 100) which is coded as the Betz additive were included in this investigation. The Air Force additive results are tabulated separately. The military antioxidants (primarily hindered phenols) were not tested given their low surface activity and their low concentrations in fuel.

For diesel fuel the approved stability additive (coded FOA-3 in this study) was investigated. Also a typical Middle Distillate Flow Improver (MDFI) additive was included in the study. The MDFI additives are not allowed by the military but are not controlled in commercial middle distillate fuel purchases by the military. They are slightly surface active but added in very high concentrations (typically above 1,000 ppm v/v). The approved cetane improver additive is very polar but not surface active and was also included in the study. Finally, a typical lubricity additive commercially available for low lubricity diesel fuels was included in the test matrix.

Much more data on this and other commercial lubricity additives are included below in Section 3.3.

A typical evaluation of an additive was to add it to the fuel and test its effect on FLHT with and without the presence of the commercial middle distillate fuel lubricity additive (Coded R650 in this study). Then the additive effect on water separation was measured in most cases by Water Separation Index Modified (WSIM), which is ASTM D3948, and in many cases by ASTM D1401 Water Reaction Test. The data for the study of possible interferences to FLHT by fuel additives are given in Table 3.2.4.

Table 3.2.4. Investigation of Possible Interferences from Fuel Additives on the Final FLHT Method and Water Separation Results. ASTM D1401 values are in minutes to complete separation, where the military pass for diesel fuels is 10 minutes maximum. WSIM values are in percent transmittance where high values indicate excellent water separation properties and values below 60 are considered problematic for water coalescence. FLHT values (final mechanical shaking method) are log NTU reading and values greater than 1.00 would indicate acceptable lubricity. ND is not determined. See text for additive codes, concentrations, and details.

Sample Code	**FLHT (log NTU)**	**WSIM (%)**	**D1401 (minutes)**
BP(2) Jet A	0.15	ND	ND
FA Jet A + FSII	0.18	97	1
FC Jet A + R650	1.98	99	ND
FB Jet A + FSII + R650	1.91	95	ND
A(4) AD-1	0.32	ND	ND
FD AD-1 + MDFI	1.41	54	1
FG AD-1 + R650	2.41	92	ND
FH AD-1 + R650 + MDFI	2.89	66	ND
FK AD-1 + FOA3	0.00	96	ND
FM AD-1 + R650 + FOA3	2.11	88	ND
FL AD-1 + CI	0.08	96	1
FN AD-1 + R650 + CI	2.13	84	1
FE AD-1 + MDFI + CI	1.54	60	3
FI AD-1 + R650 + MDFI + CI	2.93	64	ND
FF AD-1 + MDFI + CI + FOA3	1.53	56	2
FJ AD-1 + R650 + MDFI + CI + FOA3	2.92	50	ND
FQ 50:50 AD-1 + Jet A	0.11	98	ND
FO 50:50 FA + FF	0.81	71	1
FR 50:50 BP(2) + A(4) + R650	1.98	97	1
FP 50:50 FA + FF + R650	2.39	68	ND
FU AD-1 + SDA	0.40	83	1
FS Jet A + Betz 254	0.23	ND	1
FT Jet A + Betz 7	0.00	ND	1

Poor Lubricity Jet Fuel. For the Jet A in Table 3.2.4 no lubricity additive is present and so it would not pass the FLHT criterion (for this Table the pass value is log NTU ≥1.00) for use as a

diesel fuel. The presence of FSII indicates that it has no lubricity enhancing properties and it does not cause any problems with the WSIM or D1401 results. When the commercial diesel lubricity additive was added to the Jet A at 100 ppm it imparted significant lubricity and easily passed the FLHT criterion, without negatively impacting the water separation of the Jet A. Finally, there was no interference in the FLHT result from adding both the FSII and the R650 additives at 0.10 % v/v and 100 ppm, respectively.

Low Sulfur Diesel Fuel; Poor Lubricity. We see that the LSDF sample Code A(4) also failed the FLHT criterion, but when 1,000 ppm of the MDFI additive is added there is a clear pass result for lubricity. The WSIM result, however, would be considered very problematic for water separation of this diesel fuel. The neat fuel WSIM result is at least 96 (although it was not run, we can infer this from the fuel plus FOA3 or the fuel plus CI results). Thus the MDFI significantly degrades the WSIM result but does impart significant lubricity when added to a very poor lubricity fuel. This is the first time that there has been a report of the surface activity of this type of additive that might contribute to lubricity of poor lubricity fuels. Earlier Navy work [4] concluded that MDFI had no effect on lubricity of diesel fuel, however, the MDFI additives tested (including the one tested here) were added to fuels that had acceptable lubricity already. The MDFI did not show any increase in lubricity of these already acceptable lubricity fuels. When R650 commercial diesel lubricity additive was added at 100 ppm to the poor lubricity diesel (Code FG in the table) the result was an excellent lubricity value for FLHT, and only a minor degradation of the WSIM value. When both MDFI and R650 are added (Code FH) there was an even higher value obtained for FLHT and also a low, problematic WSIM value.

The diesel stability additive used as a reference in stability additive testing by the Navy (FOA3) was added at 25 ppm to the Code A(4) diesel. Clearly this additive has no lubricity enhancing properties, only minor WSIM degrading properties, and causes no interference with the FLHT result. When FOA3 and R650 are present together (Code FM) there is the expected FLHT high result, somewhat more, but acceptable degradation of the WSIM result, and no interference to the FLHT result (compare FN and FG).

The approved but rarely used cetane improver (CI) additive in F-76 is examined in Table 3.2.4. This additive was added at 0.2% v/v and is 2-ethyl hexyl nitrate. The additive shows no lubricity enhancing properties, only minor WSIM degrading properties, and causes no interference with the FLHT result. When CI and R650 are present together (Code FN) there is the expected FLHT high result, somewhat more, but acceptable degradation of the WSIM result, and no interference to the FLHT result (compare FN and FG).

Diesel additive combinations were tested such as MDFI plus CI (Code FE) and MDFI plus CI plus FOA3 (Code FF). In both of these cases the results are governed by the presence of MDFI, with or without R650 and we can conclude that these additives in all of their combinations do not interfere with the interpretation of lubricity by FLHT. In addition, we can conclude that MDFI in all of these combinations is the contributor to water separation degradation.

Because the Navy sometimes downgrades JP-5 jet fuel for use in shipboard diesel engine applications and platforms, the effects of 50:50 mixtures of poor lubricity jet and diesel fuel and additives on FLHT and WSIM were investigated. Code FQ results show that combining additive

free, poor lubricity jet and diesel give the expected FLHT and WSIM results. Code FO combines jet plus FSII with diesel plus MDFI, CI and FOA3 additives and gives the expected results for FLHT and WSIM. When this mixture was combined with R650 commercial diesel lubricity enhancer additive (Code FP), we also saw the expected results of very good LFHT lubricity and degraded WSIM. We can compare this result with Code FR (the neat jet and diesel plus R650 at 100 ppm) results and see the expected very good FLHT and very good WSIM results.

Finally, the two Air Force jet fuel additives are examined for interference with the FLHT result. Code FU is additive-free LSDF with Static Dissipator Additive (SDA) added at 1 ppm. This shows that the SDA has no lubricity effect and somewhat degraded WSIM as expected. The AF JP-8+100 thermal stability additive (coded as Betz herein) was originally intended for universal use in the AF, but now appears to be used very infrequently. This additive's use in the Navy is not allowed due to its tendency to emulsify water and disarm coalescers, which could have disastrous results for naval fuel systems and aircraft. For completeness, this thermal stability additive (coded Betz for JP-8 + 100 additive) at concentrations of 254 and 7 ppm in Code A(4) jet fuel was examined for potential interference effects. The result was unexpected. There was no interference since the additive did not cause a haze rating for the FLHT test. Although the WSIM was not run, this additive at these concentrations is well known to cause significant degradation to WSIM. This additive operates because it is such a powerful surfactant/detergent, so it was expected to falsely pass the FLHT. It is known that the Betz additive at these lower concentrations has no measurable effect on lubricity when examined using mechanical lubricity tests. What was found was that the strong base extraction was essentially partitioning the Betz additive into the aqueous layer and thus removing it from the fuel and its ability to cause water-in-fuel emulsions. This explains the very low FLHT values and because of the nature of the FLHT test, these additives do not cause any interference in using FLHT to determine lubricity.

Table 3.2.5. Comparison of FLHT results with 3 mechanical lubricity test devices. See text for additional fuel sample details.

Sample Code	**FLHT log NTU**	**HFRR (microns)**	**SLBOCLE (grams)**	**BOCLE (mm)**
FS Jet A + Betz 254	0.23	723	2,600	0.54
FT Jet A + Betz 7	0.00	678	2,100	0.52
FU AD-1 + SDA	0.40	639	1,875	0.60

The last 3 FLHT results from Table 3.2.4 are found in Table 3.2.5 in order to compare the FLHT results with 3 of the standard mechanical lubricity testers. Given the nature of the base fuels and their additives (none of them are known to have any significant surfactant or lubricity characteristics) the FLHT results are well below a pass criterion for the test (1.00). The HFRR and the SLBOCLE also give failing lubricity results for all three samples. All three samples pass using the BOCLE including the two samples that are jet fuel based. This simply means that the BOCLE test gives pass ratings to fuels intended for jet turbine hardware (pumps and fuel controls), but that this test and its results are clearly unable to correctly assess typical diesel engine hardware associated with fuel pumping and metering.

3.3 Effects of Lubricity Additives on the FLHT Test and Water Separation

3.3.1. Lubricity Additive Concentration and the Impact on Water Separation.

Table 3.3.1 gives the results for a series of six fuels, three jet fuels and three diesel fuels to which have been added 3 distinct chemical types of lubricity enhancing additives at 3 concentrations. The 3 additives are coded as follows: the ester type surfactant is R690, the mono acid type surfactant is R650 and the diacid type surfactant is DCI-4A. The additive concentrations in each fuel are 15, 100 and 500 ppm. The lowest concentration represents what might be found in a typical military jet fuel regarding lubricity enhancing additive concentrations. The next highest concentration represents what is typically being considered by the commercial marketplace as a level to treat typical poor lubricity diesel fuels. The highest concentration is to simply push the limits and might represent an accidentally overdoped additive situation. The point of using this concentration range of additives is to look at their effects on water separation.

Water separation is a very important fuel property that is not well controlled for diesel fuels by the diesel fuel side of the Navy. The air fuel side does control this property using the WSIM test described previously. Values for this test range in an index from 100 for perfect water separability down to zero for no possible water separation. Values around 70 are highly problematic for jet fuels. There is no known problem value for diesel fuels but similar values of 70% are likely to be problematic for water separation using full scale coalescer elements.

First, it should be noted that no WSIM or ASTM D1401 water reaction tests were run for the additive free jet or diesel fuels in Table 3.3.1. These additive-free fuels all had values of 99% or 100% for WSIM since the lowest concentration of additive gave results at least that high. The usefulness of ASTM D1401 in assessing the water separability for fuels (jet or diesel) that have lubricity enhancing additives added is highly questionable. All additive and fuel combinations at all additive concentrations easily passed the D1401 minimum separation time of 10 minutes. The only thing that can be said is that the DCI-4A additive appears to be more problematic using this test for water separation than the R690 or R650 lubricity additives, because the time to separation is generally higher in all cases.

The WSIM test is much more sensitive to differences caused by additive type or concentration. It is clear from this set of results that the R650 is clearly superior to both of the other additives. Recall that the R650 is a mono carboxylic acid type of additive, the R690 is a mono ester type additive and the DCI-4A is a dicarboxylic acid type additive. At 15 ppm the DCI 4A has acceptable water shedding WSIM values for all 6 fuels. However, at 100 ppm and 500 ppm, only 2 of the 6 fuels have acceptable water shedding values. The R690 additive has unacceptable WSIM values for 1 fuel at 15 ppm, 3 fuels at 100 ppm, and 5 of the 6 fuels at 500 ppm. This suggests that only the R650 type of additive (the mono carboxylic acid type) would be acceptable for naval applications which required excellent water coalescence.

Table 3.3.1.1. Water Separability and WSIM Results as a Function of Additive Type in 3 Diesel and 3 Jet Fuels. In each block below for each additive/fuel combination, there are 6 numbers. The 3 numbers on the left are WSIM numbers for the additive at 15, 100, and 500 ppm concentration respectively. The 3 numbers on the right are the D1401 water separability numbers for that additive at 15, 100, and 500 ppm concentration respectively. The WSIM numbers range from 100 (excellent water separation) to 0 very poor water separation. There is no accepted pass/fail for diesel fuels as this test is for jet fuels only (see text), however, numbers below 70 should be considered problematic and subject to further investigation. The D1401 numbers are in minutes to separate and acceptable values would be lower than 10 minutes for this test. The term "lacy" refers to a water/fuel interface that is also indicative of potential separation problems. All of the unadditized fuel gave excellent results for both tests and are not listed.

Fuel	Diesel or Jet	R690		R650		DCI-4A	
ULSD	D	59	2	100	1	96	3
Composite (low		44	2	100	1	45	4 Lacy
lubricity) DA		0	1	96	3	53	2 Lacy
CT BF-2	D	99	2	99	2	95	4 Lacy
B-4		73	2	90	4 Lacy	57	4 Lacy
		65	1	97	4	0	3 Lacy
AD-1	D	74	1	100	2	75	2 Lacy
A-4		44	2	100	2	52	5 Lacy
		0	1	97	4	76	2 Lacy
Jet A	J	98	2	99	1	93	2 Lacy
BP		90	1	96	2	62	4 Lacy
		55	1	89	3	42	2 Lacy
S-8	J	99	2	99	1	98	1
BO		98	2	96	2	90	8 Lacy
		87	1	95	3	73	4 Lacy
CT Jet A	J	99	1	99	1	100	2 Lacy
BN		68	2	97	2	91	5 Lacy
		52	1	93	2	95	3 Lacy

3.3.2 The Effect of Lubricity Additives on the FLHT Test Method

Along with the effect of commercial lubricity additives of various generic chemical types on water separation, the effects of these additives on the FLHT results were investigated. The 3 diesel type fuel results are given in Table 3.3.2.1 and the 3 jet type fuel results are given in Table 3.3.2.2. In addition to the FLHT results, the lubricity of this set of samples was also investigated using the 3 standard mechanical tests BOCLE, SLBOCLE, and HFRR.

Table 3.3.2.1. Three "Diesel" Low Lubricity Samples with 3 additive types at 3 concentrations. No pass/fail criterion is established for diesel type samples and the BOCLE results. SLBOCLE pass is >3200 grams; HFRR pass is ≤520 microns; FLHT pass is >15 NTU not yet firmly established). NTU are nephelometry turbidity units. It is important to note that the FLHT results are using the manual shake method for 1.0 minutes followed by a 10 minute wait. The proposed pass criterion for this version of the FLHT would be around 15 (or, log 15 = 1.18). See page 11 above where possible pass values for FLHT were around 20 NTU or log 20 = 1.30).

Sample	BOCLE (mm)	SLBOCLE (grams)	HFRR (microns)	FLHT (NTU)
ULSD Low (code DA)	0.67	1,925	690	1.9
+ R690 15 ppm	0.71	2,375	415	27
+ R690 100 ppm	0.62	2,775	378	241
+ R690 500 ppm	0.56	6,050	300	1,062
+ R650 15 ppm	0.53	2,450	700	12
+ R650 100 ppm	0.48	2,700	390	221
+ R650 500 ppm	0.46	4,625	250	94
+ DCI 4A 15 ppm	0.58	2,500	685	34
+ DCI 4A 100 ppm	0.52	2,800	490	235
+ DCI 4A 500 ppm	0.50	4,525	220	500
CT BF-2 (code B-4)	0.72	2,575	560	4
+ R690 15 ppm	0.60	3,950	535	24
+ R690 100 ppm	0.56	3,500	465	308
+ R690 500 ppm	0.52	5,875	255	>1,100
+ R650 15 ppm	0.52	2,775	555	70
+ R650 100 ppm	0.47	3,025	380	935
+ R650 500 ppm	0.47	3,125	260	55
+ DCI 4A 15 ppm	0.59	3,625	485	600
+ DCI 4A 100 ppm	0.52	3,375	435	>1,100
+ DCI 4A 500 ppm	0.50	4,425	325	>1,100
AD-1 (code A-4)	0.65	1,700	685	0.5
+ R690 15 ppm	0.61	2,650	690	7
+ R690 100 ppm	0.58	3,600	445	44
+ R690 500 ppm	0.53	4,250	263	172
+ R650 15 ppm	0.54	2,575	580	9
+ R650 100 ppm	0.48	2,800	410	240
+ R650 500 ppm	0.46	2,975	270	144
+ DCI 4A 15 ppm	ND	2,100	600	5
+ DCI 4A 100 ppm	ND	3,100	540	20
+ DCI 4A 500 ppm	ND	5,500	240	176

Table 3.3.2.2. Three "Jet" Low Lubricity Samples with 3 additive types at 3 concentrations. BOCLE pass is ≤ 0.62mm; SLBOCLE pass is ≥3200 grams; HFRR pass is <520 microns; FLHT pass is >15 NTU(not yet firmly established). NTU are nephelometry turbidity units. It is important to note that the FLHT results are using the manual shake method for 1.0 minutes followed by a 10 minute wait. The proposed pass criterion for this version of the FLHT would be around 15 (or, log 15 = 1.18). See page 11 above where possible pass values for FLHT were around 20 NTU or log 20 = 1.30).

Sample	BOCLE (mm)	SLBOCLE (grams)	HFRR (microns)	FLHT (NTU)
Jet A (code BP)	0.52	1,350-1,800	625	0.7
+ R690 15 ppm	0.50	2,825	608	3
+ R690 100 ppm	0.53	3,225	518	13
+ R690 500 ppm	0.52	3,275	195	81
+ R650 15 ppm	0.49	2,975	563	11
+ R650 100 ppm	0.46	3,100	350	135
+ R650 500 ppm	0.46	3,825	508	84
+ DCI 4A 15 ppm	0.53	2,825	625	10
+ DCI 4A 100 ppm	0.52	2,700	460	139
+ DCI 4A 500 ppm	0.48	3,675	230	275
S-8 (code BO)	0.90	1,050	630	0.2
+ R690 15 ppm	0.63	1,550	768	0.5
+ R690 100 ppm	0.55	3,975	733	1.6
+ R690 500 ppm	0.49	2,975	295	65
+ R650 15 ppm	0.49	1,525	740	2.6
+ R650 100 ppm	0.44	2,525	388	54
+ R650 500 ppm	0.41	3,200	188	5.3
+ DCI 4A 15 ppm	0.61	2,325	730	4
+ DCI 4A 100 ppm	0.53	1,850	700	78
+ DCI 4A 500 ppm	0.48	3,425	230	42
CT Jet A (code BN)	0.81	750	710	0.1
+ R690 15 ppm	0.63	1,450	700	3
+ R690 100 ppm	0.58	3,550	495	3
+ R690 500 ppm	0.55	3,450	210	53
+ R650 15 ppm	0.53	3,250	675	5
+ R650 100 ppm	0.44	2,300	415	106
+ R650 500 ppm	0.44	3,900	285	105
+ DCI 4A 15 ppm	0.61	1,600	705	14
+ DCI 4A 100 ppm	0.56	2,925	460	186
+ DCI 4A 500 ppm	0.50	5,325	240	250

From the data in Table 3.3.2.1 for the 3 diesel fuels, all 3 unadditized fuels would fail even the jet fuel criterion for lubricity using the BOCLE (maximum scar = 0.62 mm). Only in one case does 15 ppm of any additive cause a failure using the BOCLE test and in all cases 100 or 500

ppm cause BOCLE passes. All 3 fuels also fail the SLBOCLE, the HFRR and the FLHT test criteria for lubricity. Generally, all 3 additives begin to improve lubricity using these 3 tests even at 15 ppm, however, the great sensitivity to additive concentrations is clearly exhibited in the FLHT over the SLBOCLE or the HFRR tests. The SLBOCLE is generally much less sensitive to additives than the HFRR and in many cases the addition of 100 ppm of additive still shows a failure by SLBOCLE and a clear pass by HFRR testing.

In Table 3.3.2.2 for the jet fuels, most of the test methods gave much lower additive-free fuel results than diesel. The exception is code BP, the Jet A fuel, that gives a pass using BOCLE but is clearly problematic for the other 3 tests. Again the addition of all additives begins to show great improvement in lubricity even at the lowest concentration added, 15 ppm. However, for one of the tests even at the highest concentration of additive, a pass on the SLBOCLE is not achieved. In general the additives are less beneficial in the jet fuels than in the diesel fuels. Again, the clearly greater sensitivity of the FLHT test to additives over the other mechanical lubricity tests is apparent.

Recall that at and above 100 ppm only the R650 additive gave acceptable water shedding properties, so it is important to note that at 100 ppm this additive gave lubricity values by most of the mechanical tests (especially the FLHT) that were as good or better than the other two additive types. This means that this particular type of additive gives the best performance for two critical properties that would normally be inversely related.

One final thing to note in the above two tables is that in some cases there is a definite drop off in FLHT results when increasing the concentration from 100 to 500 ppm. This is explained by the fact that at the higher (really over additized) concentrations, an interface forms where the emulsion that would normally be suspended uniformly in the fuel layer is now concentrated. This is why the haze rating is much lower. This is actually an additional benefit to the use of FLHT over the other lubricity tests, since it is simple way to determine if too much additive has been added.

3.3.3 The Effect of Intermediate Additive Concentrations of FLHT

Finally, with regard to lubricity enhancing additives, the response of the FLHT was examined throughout the range of concentrations from 15 to 100 ppm. The data for two different extremely poor lubricity jet fuels are given in Table 3.3.3.1 for the FLHT in all 3 of its versions (manual shaking for 1.0 minute and 15 minutes wait, first mechanical friction shake version with shake time of 0.5 minute and wait of 10.0 minutes, and final mechanical shake version with 0.5 minute shake time and 10.0 minutes wait time), plus comparison of the FLHT values (log of NTU) to HFRR, SLBOCLE and BOCLE.

Table 3.3.3.1. Effect of Intermediate Additive Concentrations of R650 (mono carboxylic acid type) Commercial Lubricity Additive on two Poor Lubricity Jet Fuels. FLHT values are log NTU. See text for discussion of pass values of all tests.

Sample Code ID	**R650 ppm**	**Manual FLHT (Log NTU)**	**First Mech. FLHT (Log NTU)**	**Final Mech. FLHT (Log NTU)**	**HFRR (Micron)**	**SLBOCLE (Grams)**	**BOCLE (mm)**
BP(2)	0	0.11	0.11	0.20	625	1,750	0.52
FV or GG	15	0.54	0.54	0.48	651	2,900	0.49
FW	25	0.90	1.15	0.95	651	3,075	0.49
FX or GH	50	1.2	1.43	1.30	442	3,250	0.46
FY or GI	75	1.54	1.63	1.63	397	3,000	0.44
FZ or GJ	100	1.67	2.02	2.03	374	3,275	0.44
BO	0	0.00	0.00	0.00	630	1,050	0.90
GA	15	0.00	0.00	0.00	696	2,438	0.49
GB or GK	25	0.30	0.30	0.04	706	2,775	0.50
GC	50	0.85	0.85	0.60	531	2,853	0.47
GD or GL	75	0.95	0.95	0.94	434	3,375	0.44
GE or GM	100	1.18	1.18	1.11	417	3,375	0.44

For these very poor lubricity jet fuels, the 3 versions of the FLHT test appear to be rather similar. It is only in moderate to high lubricity fuels that there are differences in the 3 versions. If the proposed pass value for the final mechanical FLHT version is log NTU = 1.00, then for the Jet A (Code BP(2)) fuel the additive concentration to achieve this is just above 25 ppm and in the S-8 synthetic jet fuel from natural gas (Code BO) this pass is achieved at an additive concentration just above 75 ppm. This latter fuel is by far the poorest possible LSDF that would require an additive to achieve adequate lubricity in diesel type equipment for the Navy. This last statement is awaiting the final result of naval hardware testing at SwRI which will tie together the results of this study and provide final guidance on pass criteria for the FLHT test and for the use of additives.

The best commercial diesel fuel lubricity additive for naval use (R650) was chosen for the intermediate additive concentration work. It is interesting to note that for this additive in these very poor lubricity jet fuels, that the SLBOCLE and the HFRR tests also showed that proper minimum additive concentration to achieve a pass in each test was also just above 25 ppm in the Jet A fuel. For the worst case fuel, S-8 (Code BO), both tests suggested that a minimal concentration of 75 ppm was required, even though the FLHT suggested that this minimal concentration was above 75 ppm.

It is important to note that because of the optical nature of the FLHT test (its inherent non-linearity), that the log of the intermediate additive concentrations gives an excellent straight line fit throughout the entire range of concentrations. This is not seen in the SLBOCLE or HFRR tests.

Looking at the BOCLE results, we see that except for the neat S-8 fuel all of the lubricity values pass this test. These fuels may be adequate for jet hardware applications, but clearly, this is another reason why the BOCLE is not able to be used for any diesel hardware applications. It was not only developed just for the jet airframe hardware, but that hardware is apparently much more forgiving than diesel hardware for poor lubricity fuels.

3.4 Comparison of Mechanical Test Data with FLHT Data

3.4.1. Comparing a Range of Additive-Free Fuels' Lubricity Using a Variety of Methods.

When the development of a potential chemical test was first suggested during quantitative analysis of fuel surfactants, it was thought that the original quantitation by gas chromatography (GC) would be the best indicator of degree of success in developing a chemical test method to determine fuel lubricity. Thus, originally a set of 11 additive-free fuels was assembled and the total amount of surfactant content of each of them was determined by the original GC method [1]. Included in this sample set were a very wide range of fuel lubricities. In fact, this range was essentially guaranteed by the selection of some very highly processed jet and diesel fuels in addition to the selection of a number of high sulfur, minimally processed diesel fuels. Table 3.4.1.1 gives the results for the 11 fuels by GC analysis and for comparison by SLBOCLE, HFRR, and FLHT. The results are ranked in order of SLBOCLE results from best lubricity to poorest lubricity. BOCLE results are included simply for completeness, but are of little value for this fuel set. Two of the fuels are actually 50/50 mixtures made in order to achieve intermediate SLBOCLE values.

Within this data set is the original 3 fuels which were to be included in the lubricity hardware testing going on at this time at Southwest Research Institute (SwRI) under a separate contract effort. These fuels were coded in the hardware testing as Fuel 3000, Fuel 2000, and Fuel 1000 to indicate their respective SLBOCLE rating ranges. In fact, Fuel 3000 was originally Code BU, but this was subsequently replaced by another fuel (Code EV in our sample set). Fuel 2000 is actually Code BP in our fuel set (Jet A) and Fuel 1000 is actually Code BO in our data set (S-8 synthetic jet from natural gas).

In Hughes' earlier work (1), six different excellent lubricity diesel fuels have GC values of greater than 0.42% w/w (up to 0.71% w/w). All the remaining 24 of the poor lubricity fuels gave GC values of less than 0.25% w/w and most of these were less than 0.10% w/w. This earlier trend appears to hold for the present 11 fuels examined in this study. The first 4 entries in Table 3.4.1.1 are all well above 0.25% w/w and the bottom 7 are all well below 0.25% w/w. Although Hughes did not try to correlate the SLBOCLE and HFRR values of the 30 fuels in that data-set directly with the GC analysis, it could be seen that all 6 of the excellent lubricity fuels correlated with pass criterion for both mechanical tests. However, the SLBOCLE results gave one false pass (if it is accepted as the quantitative chemical extraction test for surfactants as the primary

test) for the 24 poor lubricity fuels and the HFRR test gave one false pass (not the same fuel as the SLBOCLE false pass).

Examining Table 3.4.1.1 SLBOCLE results, we see that the currently defined pass value of ≥3,200 grams is probably set too high for this additive-free set of fuels. It appears that a better pass value could be set for SLBOCLE as low as 2,800 grams minimum or perhaps even a bit lower. The reason for the current much higher pass criterion is probably due to the inherently very poor precision of the method even at around 2,800 to 3,200 grams. Although the actual precision of the GC extraction method was not determined, the values in Table 3.4.1.1 are actually the average of 2 separate extractions of each fuel sample and the duplicates are usually within 15% standard error throughout the range of analyses. This means that at 0.25% w/w the duplicate analysis would be within 0.04% w/w.

Table 3.4.1.1. Comparison of Various Methods to Determine Lubricity for a Wide Range of Additive-Free Fuels and Fuel Mixtures. Mixtures are volume mixtures. Ranking in the Table is by SLBOCLE from best to worst lubricity. ULSD = ultra low sulfur diesel fuel. SLBOCLE pass ≥3,200 grams; HFRR pass ≤ 520 microns; this version of FLHT pass >1.30; BOCLE pass ≤ 0.62 mm.

Sample ID Code	**Sample**	**GC Result (% w/w)**	**SLBOCLE (grams)**	**HFRR (Microns)**	**FLHT Log (NTU)**	**BOCLE (mm)**
B(4)	BF-2	0.51	3,875	410	2.70	0.58
BQ	High Lubricity ULSD	0.40	3,050	540	2.51	0.56
BU	#2 Diesel EPA	0.64	2,950	280	2.20	0.54
BT	50/50 BF-2/ AD-1	0.39	2,850	515	1.55	0.60
BM	CT BF-2*	0.11	2,575	560	0.60	0.72
DA	Low Lubricity ULSD	0.04	1,925	575	0.34	0.71
BP	Jet A	0.12	1,800	625	0.00	0.52
A(4)	AD-1	0.14	1,700	685	0.00	0.65
BS	50/50 Jet A/CT Jet A*	0.08	1,475	735	0.48	0.55
BO	S-8	0.05	1,050	630	0.00	0.90
BN	CT Jet A*	0.03	750	710	0.00	0.81

*CT refers to the original fuel that was Clay Treated to remove polar and surfactant material.

Examining Table 3.4.1.1 HFRR results, all of the poor lubricity fuels are correctly and easily determined by HFRR. However, it is troubling that one of the very good lubricity fuels (Code BQ) gives a fail result. This is despite the fact that all of the other tests easily passed this fuel for lubricity. This result was obtained from duplicate analysis and when re-examined later on a separate fuel aliquot was affirmed.

The FLHT results in the Table 3.4.1.1 easily correlate with the GC result and this is not surprising since the FLHT method is based on the original GC extraction result. In summary, all

4 methods, 2 chemical and 2 mechanical methods, (with one notable exception for HFRR) agree for all 11 fuels regarding lubricity, even for fuels that are clearly near the cut off of pass/fail for all of the methods.

The BOCLE results for Table 3.4.1.1 are included just for completeness and it is noteworthy that at least two results for the 7 poor lubricity fuels show pass values (Code BP and Code BS). This again demonstrates that the BOCLE results simply cannot and should not be used when trying to determine a fuel's fitness for lubricity in diesel hardware equipment.

A second, more systematic look at the entire range of fuel lubricity to compare all of the methods above and to take a closer look at pass/fail criteria for the various methods was made by preparing a mixture of excellent and poor lubricity fuels. The data in Table 3.4.1.2 were generated from taking Code B(4) and Code BO and making a series of 10% blends (Code BV through Code CD). A very similar set of data was generated using Code F(3) as the poor lubricity fuel blended into Code B(3).

Table 3.4.1.2. Comparison of Various Methods to Determine Lubricity for a Series of 10% v/v Blends of an Excellent and a Poor Lubricity Fuel, Additive-Free. Ranking in the Table is based on the volume blends, from best to worst lubricity. SLBOCLE pass ≥3,200 grams; HFRR pass ≤ 520 microns; this version of FLHT pass >1.30; BOCLE pass ≤ 0.62 mm.

Sample ID Code	**GC Result (% w/w)**	**SLBOCLE (grams)**	**HFRR (microns)**	**FLHT Log (NTU)**	**BOCLE (mm)**
B(4)	0.51	3,875	410	2.70	0.58
BV	0.28	3.750	355	2.44	0.60
BW	0.29	4,625	405	2.05	0.59
BX	0.29	4,100	410	1.73	0.59
BY	0.26	4,100	465	1.58	0.59
BZ	0.25	3,125	510	1.28	0.59
CA	0.18	3,125	535	0.95	0.60
CB	0.17	3,000	540	0.90	0.60
CC	0.13	2,400	560	0.54	0.60
CD	0.13	1,675	675	0.59	0.61
BO	0.05	1,050	630	0.00	0.90

Examining the GC data from Table 3.4.1.2, the criteria developed from Hughes' original work plus the data from Table 3.4.1.1 where poor fuels are defined as 0.25% w/w or lower it appears that Sample Code BZ through Code BO should be poor lubricity fuels and Code BY should be marginal and Code B(4) through Code BX should be good lubricity fuels. However, it is now clear that the GC quantitative data are also not linear simply by comparing the GC data with a known linear method such as HFRR. This is most likely because the GC extraction also carries along fuel components that are not the surfactants responsible for lubricity. Thus as more dilute blends of the good lubricity fuel (defined by GC extraction) were made the expected drop off does not occur because this is masked by the constant amount of non-surfactant material which is included in the total GC analysis of the extract. This does not detract from Hughes' finding that fuel of 0.25% w/w and lower has poor lubricity since a very large data set was examined to reach

that conclusion and regardless of the GC result for this one series of fuel blends, it would be justified in predicting lubricity fails for the last 6 or 7 blends in the entire series.

Examining the SLBOCLE data from Table 3.4.1.2, this mechanical test correlates very well with the GC results. The only exception is that the two best fuels Codes B(4) and BV give somewhat lower weights than the next 3 fuels in the series. But in every case the SLBOCLE predicts that the first 5 fuels should exhibit excellent lubricity and the remaining 6 fuels should have much less lubricity, provided the pass criterion is ≥=3,200 grams. Recall that for the fuels in Table 3.4.1.1 some of the clear GC predicted passes had SLBOCLE results that were clearly below the pass criterion of ≥3,200 grams. This is probably more due to the poor precision of the SLBOCLE especially when running different fuels (ASTM reproducibility) and not because different fuels might carry along differing amounts of non-surfactants in the GC extraction. This would tend to mitigate against using SLBOCLE results as a means of evaluating fuels that weren't very poor or very good (by SLBOCLE or any other discriminating test method). This simply means that SLBOCLE is probably not the test method of choice for fuel lubricity that may be considered "marginal" for diesel hardware equipment.

The HFRR data in Table 3.4.1.2 for the most part show good agreement both with the GC and SLBOCLE results in general. One fuel, Code BZ, does show as a pass using the 520 micron maximum pass currently being used for this test, whereas all of the other tests show this to be just in the failing range. This also points out that the HFRR is very good at distinguishing very good and very poor lubricity fuels, but is much less able to predict fuels that lie in the "marginal" lubricity range. As with the SLBOCLE test this is most likely due to the poor precision of the HFRR method, more than anything else. Both of these methods will have difficulty at their current stated pass/fail criteria.

The FLHT data in Table 3.4.1.2 are very interesting in light of the discussion above. This test appears to be more discriminating than the GC test upon which it is based. This is because unlike the GC test which includes some of the non-surfactants in its % w/w result, the optical FLHT includes only the contribution of surfactants that cause a haze under the test conditions. Thus FLHT results are clearly superior to GC results in predicting lubricity. The FLHT results agree in general with both SLBOCLE and HFRR in distinguishing very good and very poor lubricity fuel. Where the FLHT is superior to these two is in its ability to distinguish much more accurately the "marginal" lubricity fuels or those in the mid range of lubricity. This is partly due to the much better precision of the FLHT method, but is also due to the fact that the FLHT is both a chemical and an optical method, both aspects of which tend to concentrate and focus upon the actual fuel components that contribute most to the property of fuel lubricity. Finally, the log (NTU) plot of this series vs the increasing volume % of good lubricity component gives a straight line throughout the entire range of lubricity with an R^2 value of 0.99.

3.4.2 Use of FLHT with Marginal Lubricity Fuels and with Different Fuel Types Including Biodiesel

As indicated in the previous section the FLHT appears to be the best way possible of rating fuel lubricity when approaching the mid range or what could be called "marginal" range of lubricity by bench test methods. Of course, these mid range or "marginal" lubricity fuels must be

examined by component tests and finally full scale hardware tests, or actual use to determine actual useful minimal criteria for the laboratory-scale tests, such as FLHT. However, during the early evaluation of the FLHT with manual shaking for 1.0 minute and a 15.0 minutes wait time, a series of 6 different fuel blend stocks was used to try to get as close to a mid range lubricity value as possible to see if any particular blending component would be problematic when examined at two different blends near the mid range lubricity. Recall that for this version (manual shake) of the FLHT that a pass would be about log 30 = 1.48.

Table 3.4.2.1. FLHT data from various component blend stocks near the mid range of lubricity. "Good Fuel" = 60% B(4)/40% A(2). Pass criterion for this version of FLHT manual is log 30 = 1.48.

Fuel Blend Description	**Log (NTU)**
25% Hydrotreat 4/75% "Good Fuel"	1.67
75% Hydrotreat 4/25% "Good Fuel"	1.26
25% Isopar M/75% "Good Fuel"	1.54
75% Isopar M/25% "Good Fuel"	1.04
25% Shell FT/75% "Good Fuel"	1.32
75% Shell FT/25% "Good Fuel"	0.48
25% Sasol FT/75% "Good Fuel"	1.32
75% Sasol FT/25% "Good Fuel"	0.70
25% Hydrotreat 3/25% "Good Fuel"	1.48
75% Hydrotreat 3/75% "Good Fuel"	1.15
25% Hydrotreat 4/ 75% Biodiesel 5% in A(2)	1.79
50% Hydrotreat 4/50% Biodiesel 5% in A(2)	1.57
75% Hydrotreat 4/25% Biodiesel 5% in A(2)	1.00

For the poor lubricity blending components, there are two hydrotreated diesel, petroleum based blend stocks in Table 3.4.2.1 and one iso-paraffin solvent mixture (Isopar M) and two synthetic diesel blend stocks (one from natural gas and the other from coal). It is clear that the ability of the FLHT to discriminate good and poor lubricity fuels in the mid range is independent of blend stock source. In addition it is clear that using a poor lubricity petroleum based blend stock (Biodiesel 5% v/v in Code A(2)) that contained the very high lubricity material, biodiesel from soy beans, can impart significant lubricity to the blend, however, the volume % of this 5% biodiesel mixture must be quite high (around 50%) to achieve this lubricity.

The use of the FLHT to determine a wide variety of fuel types (not blended) was also investigated and the data are shown in Table 3.4.2.2. This Table also includes the soy bean derived biodiesel and a variety of blends of this biodiesel with a poor lubricity fuel, Code A(2).

Table 3.4.2.2. Results for FLHT using the manual shake version and a wide variety of fuel types. Proposed pass for this version log 30 = 1.48. All are in % v/v.

Fuel Description	**Log NTU**
Shell FT - Gas to Liquid Diesel	0.00
Sasol FT - Gas to Liquid Diesel	0.00
Isopar M	0.00
Hydrotreat 4 – Haifa	0.08
Hydrotreat 3 = Arco	0.66
60% BF-2/40% Arctic Diesel #1	1.70
100% Biodiesel from Soy	>3.04
20% Biodiesel/80% Arctic Diesel #1	>3.04
5% Biodiesel/95% Arctic Diesel #1	1.60
2% Biodiesel/98% Arctic Diesel #1	1.04
1% Biodiesel/99% Arctic Diesel #1	1.00
0.5% Biodiesel/99.5% Arctic Diesel #1	0.38

It is clear from the data that the FT liquids and the Isopar M solvent have no surfactants present. The hydrotreated petroleum diesel stocks have very little, but different, levels of surfactants present. The biodiesel from soy exhibits a high level of surfactant formation and thus should be an excellent lubricity blending stock from that point of view. However, it takes a significant fraction of biodiesel to give a pass rating to the very poor lubricity blend stock into which it was blended – 5% v/v. From 2% v/v and lower, biodiesel would not provide an acceptable level of lubricity based on FLHT results.

3.4.3 Comparison of FLHT to SLBOCLE data for diesel fuels

During the development of the FLHT test there were two studies occurring independently which gave the program access to a separate set of diesel fuels and a separate set of jet fuels. The first study was a set of 12 blind samples from an ASTM precision test of one of the mechanical lubricity testers. The second set was from a B.F. Goodrich study of jet fuel properties from around the world. The 12 ASTM diesel fuels were tested by SLBOCLE and by FLHT (manual version) and the data are presented in Table 3.4.3.1. For the manual version of the FLHT the proposed pass was log 30 = 1.48. The 12 diesel fuels track very well throughout the wide range of lubricities (some extremely poor, <1,000 grams). In addition, 4 of the 12 are deemed excellent by the FLHT although by the current SLBOCLE criteria (pass ≥ 3,200 grams) only the best fuel would have actually passed. This is the dilemma previously posed in Section 3.4.1 by the poor precision of the SLBOCLE which requires that a very conservative pass value must be set. The actual pass value for SLBOCLE suggested by the FLHT data would be somewhere between 2,500 and 2,950 grams as was suggested in Section 3.4.1.

Table 3.4.3.1. ASTM diesel fuel set comparison between the manual shake version of FLHT and SLBOCLE. Fuels are ranked by SLBOCLE from best to worst lubricity.

ASTM Diesel Code	SLBOCLE (grams)	FLHT (Log NTU)
1	3,500	>3.04
2	3,150	2.36
3	2,950	2.11
4	2,500	1.58
5	2,400	0.30
6	2,300	0.78
7	2,150	0.78
8	2,000	0.90
9	1,300	0.00
10	<600	1.08
11	<500	0.00
12	<465	1.23

From the jet fuel sample, 6 samples were chosen to exercise this comparison between FLHT and SLBOCLE. All these samples contained no lubricity improver additives. Recall that SLBOCLE was not developed for use with the lower viscosity jet fuels in general, and that is why these fuels were grouped together since all of them exhibit similar viscosity. The data are given in Table 3.4.3.2 and show the expected result that SLBOCLE values are extremely low with no passes using the diesel fuel criterion of ≥3,200 grams. Several of the jet fuels do show significant SLBOCLE ratings (#43 and #104). The FLHT values are also very low as expected (jet fuel naturally contains many fewer surfactants and polar components than diesel fuel in general). All of the FLHT values fail the manual version pass criterion. But it is interesting to note that even at these very low lubricities (from a diesel hardware point of view) the FLHT is still able to differentiate the 6 fuels very easily. This might be the basis of differentiating jet fuels for diesel applications and provide an easy way to minimize lubricity additives for such applications.

Table 3.4.3.2. Comparison of SLBOCLE and the manual shake version of FLHT for 6 typical additive-free commercial jet fuels. Fuels ranked by FLHT from best to worst.

B.F. Goodrich ID	FLHT (Log NTU)	SLBOCLE (grams)
42	1.04	1,700
43	0.95	2,500
30	0.60	1,200
104	0.48	2,650
306	0.00	1,750
7	0.00	<700

3.4.4 Possible use of FLHT to Determine Jet Fuel Lubricity Compared to BOCLE

The possibility exists to use the FLHT not just as a potential method to evaluate jet fuels for diesel applications, but also as a possible way to evaluate jet fuels for jet hardware application instead of the BOCLE mechanical method. The BOCLE test method is subject to the same problems as outlined above for the diesel fuel lubricity mechanical testers, including difficulty of

operation, imprecision, and inability to be used as a field test. The FLHT was developed to look at diesel type fuels and not jet fuels. The surfactants that provide lubricity in diesel fuels are identical in chemical type to those in jet fuel. However, in the future the FLHT may be modified to enhance the partition coefficient of the surfactants. For now, an attempt was simply made to take 6 randomly selected jet fuels from the B.F. Goodrich commercial jet fuel set and measure their BOCLE ratings while at the same time measuring the FLHT values for the manual version of the method.

Table 3.4.4.1. Comparison of BOCLE data to FLHT manual version. All samples are commercial jet fuels without additives. BOCLE is in mm (pass ≤ 0.62 mm). FLHT is in log (NTU). Pass for this manual version is >log 30 = 1.48. Fuels are ranked by FLHT data.

B.F. Goodrich ID	**FLHT (Log NTU)**	**BOCLE (mm)**
41	1.11	0.64
33	0.95	0.56
109	0.60	0.61
45	0.48	0.59
301	0.00	0.68
7	0.00	0.64

The BOCLE data show that most of the fuels are marginal regarding the pass criterion with 3 of the 6 failing and 3 passing. On the contrary, the FLHT results for these fuels again show a wide range of surfactant haze rating for these fuels and the ability to differentiate the fuels for possible additive use. Clearly all of the fuels are again deemed fail by the FLHT for diesel hardware uses. The lack of ranking comparison between the two methods can generally be explained by the fact that the BOCLE is very imprecise, the fuels are marginal (even by BOCLE rating) and the FLHT was not really modified to differentiate such low levels of surfactants.

The **ease** of measuring lubricity by the FLHT method, alone, is reason enough to pursue the possibility of modifying the procedure in the future as a laboratory specification method and/or as a field method for jet fuels.

4.0 CONCLUSIONS AND RECOMMENDATIONS FOR POSSIBLE FUTURE WORK

There are four major conclusions drawn from the above study. First, the FLHT final mechanical shake version which incorporates a mechanical shaking of 30 seconds and a wait time of 15.0 minutes is suitable for final development as a procurement specification test (either ASTM Committee D-2 or Navy specific test). The method could also be easily adapted for field work. The precision of the method is good throughout the range of total possible lubricity for LSDFs. There is currently no operator bias, however, the precision of the method was partly sacrificed to remove operator bias. The response (log NTU) is linear throughout the entire range of interest and, most importantly, the method is capable of differentiating small changes in lubricity, especially when these changes are induced by lubricity enhancing surfactant additives.

Second, the FLHT method as currently developed has no known interferences from fuel color, red or blue fuel dyes, the presence of seawater or water, the presence of biofuels or synthetic fuels, or any typical fuel additives. One fuel additive studied, MDFI, at its typical high concentration of 1,000 ppm was found to impart lubricity to very low lubricity fuels. This lubricity benefit of MDFI is far outweighed by this additive's tendency to emulsify water.

Third, the FLHT method appears to be very sensitive to all generic commercial lubricity additives. The method can be modified to determine if too much additive has been added. The method is sensitive to very small changes in additive concentration throughout the whole range of lubricity. This makes the FLHT especially attractive to fuel producers wanting to minimize costs by avoiding unneeded high concentrations of additive and to fuel users such as the Navy which cannot tolerate water emulsion problems in its fuel handling equipment or shipboard engines.

Fourth, the FLHT method does compare well to standard accepted mechanical ASTM tests methods such as SLBOCLE and HFRR, especially at very good lubricity and very poor lubricity ends of the scale. For the mid range of lubricity (the so-called "marginal" lubricity fuels) the FLHT appears to be much more capable than the typically imprecise and difficult-to-run mechanical lubricity test methods.

There are 9 recommendations for potential future work on the FLHT method. First, the use of the method for jet fuels that contain lubricity additives (such as JP-5 or JP-8) needs to be addressed as soon as possible. This is because the Navy already uses JP-5 that has been "down graded" to F-76 (the Navy's specification diesel fuel for ship use) and the question then becomes – is JP-5 of adequate lubricity to protect the diesel engine fuel injection system? In addition, answering this question would provide a rationale for possible use of FLHT in the JP-5 specification. This would be especially important if the Navy single fuel at sea initiative is developed in the future.

Second, and related to the above, is a need to determine how the use of both a FLHT requirement and mandatory additive levels of lubricity additives might affect the JP-5 procurement specification. It is already known that many JP-5 jet fuels have adequate lubricity from an air frame hardware requirement standpoint without the need for the mandatory additive. The potential use of an easy lubricity test at the refinery may allow producers to only add the additive to jet fuel that does not have adequate lubricity. The question of mixing JP-5 fuels in the field later also needs to be addressed.

Third, a better understanding of the trade-offs between increasing lubricity-enhancing additive concentrations and decreasing water separation and potential increases to gas turbine hot section corrosion need to be investigated. The FLHT could be the simple tool to study these trade-offs given its very high sensitivity to lubricity additives.

Fourth, standard methods should be developed to determine if FLHT can be used to protect from adding too much lubricity additive. It was noted that when this happens an interface tends to form which causes the fuel haze to actually decrease and therefore falsely shows fuel has poorer

lubricity than it actually has. Other than operators taking note of this effect, this is not part of the standard FLHT method. A more systematic study of adding increasing amounts of additive needs to be accomplished.

Fifth, it should be determined if the FLHT method can be used for other fuel quality field testing such as water separation or jet fuel thermal stability. Already, the method is similar to the WSIM method and the correlation does exist that as lubricity increases, water separation decreases whether due to natural fuel materials or additives. Generally as water separation decreases (worsens), this is due to increasing amounts of polar material and surfactants which tends to decrease thermal stability when it is measured as a separate property. Perhaps various techniques based on the FLHT method could be developed that could be correlated to these two properties. This would have tremendous benefits in that current field tests for these properties do not exist. In addition a simple test for polar materials that correlated well to thermal instability could be the basis for a replacement to the questionable JFTOT test current in the JP-5 procurement specification.

Sixth, given the great sensitivity of the FLHT method, a study should be made whether known very poor lubricity fuels have had a lubricity enhancing additive added in order to bring them to their current "marginal" status, rather than simply being marginal lubricity fuels without an additive present.

Seventh, it is recommended that the FLHT method as developed in this study be submitted to ASTM D-2 as a potential lubricity test for diesel fuels. Alternatively, the Navy could develop the test either for the F-76 diesel fuel procurement specification and/or as a field test for Navy/USCG/MSC use when purchasing commercial fuels.

Eighth, it is recommended that the FLHT method as it is currently written be used in on-going studies addressing the use of synfuel mixtures with petroleum derived fuels. Initial studies in this report are promising and should be expanded.

Ninth, and finally, it is recommended that other detergent-type additives (similar to the Betz additive in this study) be investigated as a function of their concentration in low lubricity base fuels as to their effect on the FLHT.

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6.0 ACKNOWLEDGMENTS

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APPENDIX A

FY06 Test Plan
Development of a Chemical Lubricity Test for Naval Fuels
(Fuel Lubricity Haze Test – FLHT)

Problem/Deficiency Being Addressed: Ultra low sulfur middle distillate fuels in the US and abroad may be achieved by severe refinery processing, which can lead to removal of natural surfactants in the middle distillate fuel and most likely, to poor lubricity performance. This lack of naturally occurring surfactants also applies to most fuels derived from gas-to-liquid processes such as Fischer-Tropsch as well as to aviation jet fuels.

Mechanical tests such as SLBOCLE, BOCLE, and HFRR are standardized but are imprecise, difficult to run, not amenable to field testing, and expensive. Also, when lubricity improver additives are present, these mechanical tests may produce results that are even more imprecise, and can lead to unnecessary additive overdoses.

If chemical tests can be devised, they will be simpler, quicker, more able to differentiate fuels (both with and without additives) than mechanical tests, and may be able to be performed in the field.

Deliverable: A rapid, simple chemical test method or methods which are shown to be quantitative and related to real-world data will be developed along with a report detailing the development and results of the simple test.

FY06 Task List

1. NRL to prepare Memo Report. Based on FY 04 - FY 06 work, including the below FY 06 work. *(May 2008; NRL Completed)*

2. Finalize FY06 test plan. *(June 2006; NRL/Pax completed)*

3. Precision Enhancement Tasks and Final Development of the Mechanical Fuel Lubricity Haze Test (FLHT).

   a. Buy one mechanical shaker with timer and variable shake severity
   (December 2005; NRL completed)

   b. Compare the standard FLHT method with manual shaking to the mechanical shaking version at several levels of shaking severity. Use two fuel mixtures of 90% BF-2 (Sample ID B(4)) and 10% Arctic Diesel #1 (Sample ID A(4)). AD-1); and 50% BF-2 and 50% AD-1. ***NOTE: During this task, the mechanical shake method (called the First Optimization/Friction Method) appeared unsuitable for possible shipboard implementation, therefore a "Second Optimization" termed the "Restrained Cap Method" became necessary to develop.***
   (January 2006; NRL completed)

c. Note the bubble effect from (b) above (January 2006). Document via written notes. (Jan*uary 2006; NRL completed)*

d. Using the Second Optimization/Restrained Cap Method (mechanical), determine the optimal mechanical shake time and wait time aiming to minimize the manual shake times from 1 minute and 15 minutes to as short as possible (e.g., 15 sec each). Use three (3) different fuels at two (2) different mixture levels, aiming for HRT levels of over 100 and around 30, for a total of six (6) samples. The high lubricity fuels BF-2 (Sample ID B(4)), Arco feedstock (Sample ID EW), and EPA diesel #2 (Sample ID BU) will be mixed with low-lubricity Arctic Diesel #1 (AD-1, Sample ID A(4)) to obtain these HRT levels. *(January 2006; NRL completed)*

e. Determine the precision of the mechanical shaker and new potential shake/wait times for the Second Optimization/Restrained Cap Method using the lower lubricity FLHT samples (approximate FLHT levels of 30) from (d). Also include EPA diesel #2 neat fuel (Sample ID BU) in this task because that fuel had a wide range of FLHT results when tested on the manual method several times on different days by the same operator. Also determine precision of the manual shaking FLHT using these samples for comparison. *(January 2006; NRL completed)*

f. Test the precision using 3 test samples for SLBOCLE, BOCLE and HFRR (all in duplicate for a total of 6 samples) for 50:50 BF-2 (Sample ID B(4)) and AD-1 (Sample ID A(4)), for 55:45 Sample EPA diesel #2 (BU) and AD-1, and 13:87 of Arco feedstock (Sample ID EW) and AD-1. These three were the most problematic mixtures when determining the final precision of the HRT method. NRL will prepare the mixtures and send samples to Pax for SLBOCLE (in duplicate) and BOCLE (in duplicate), and to SwRI for HFRR (in duplicate). Final data will be collected at NRL and included in the final report.

(May – June 2006; NRL, Pax and SwRI)

4. Investigation of Possible Interferences on the Final FLHT Method and Water Separation Tests

(May – July 2006; NRL, Pax,and SwRI).

a. Jet A (Sample ID BP) plus the minimum required concentration of 0.1% v/v Di-EGME (FSII) will be screened for any possible positive interference using the FLHT final mechanical method (Second Optimization/Restrained Cap) method above (in duplicate at NRL). Single determinations of WSIM and D1401 water shedding tests at Pax will be run. Finally, a sample from the remaining volume above will include 100 ppm of Infineum R-650 plus the FSII additive to obtain another set of results for the 3 tests (FLHT, WSIM (water separation index modified) and ASTM D1401),which will be compared to the previous results of the Jet A (Sample BP) plus R-650 alone results. R-650 is chosen throughout this task due to its previously determined superior water shedding and lubricity characteristics.

b. AD-1 (Sample ID A(4)) plus Middle Distillate Flow Improver (MDFI) at its maximum effective concentration will be screened for any possible positive interference using the FLHT final method above (in duplicate at NRL) by making a 100 mL volume sample. The MDFI additive used will be Keroflux from BASF at 1,000 ppm, which was chosen due to the effect that it had in degrading WSIM and D1401 (while still passing these tests) in previous work conducted at Pax on the additive. Single determinations of WSIM and ASTM D1401 water shedding tests will be made at Pax. Finally, a sample will be made up from the remaining volume above that will include 100 ppm of Infineum R-650 plus the MDFI additive and results of these 3 tests (HRT, WSIM and D1401) will be compared to the results of the AD-1 (Sample A(4)) plus R-650 alone results.

c. AD-1 (Sample ID A(4)) plus FOA-3 (dimethyl hexyl amine, a storage stability additive) at 25 ppm will be screened for any possible positive interference using the FLHT final method above (in duplicate at NRL). Single determinations of WSIM and D1401 water shedding tests will be made at Pax. Finally, a sample will be made up from the remaining volume above that will include 100 ppm of Infineum R-650 plus the FOA-3 additive and results of these 3 tests (FLHT, WSIM and D1401) will be compared to the results of the AD-1 plus R-650 alone results.

d. AD-1 (Sample ID A(4)) plus a Cetane Improver (CI) (2-ethyl hexyl nitrate) at 0.2% v/v will be screened for any possible positive interference using the FLHT final method above (in duplicate at NRL). Single determinations of WSIM and D1401 water shedding tests will be made at Pax. Finally, a sample will be made up from the remaining volume above that will include 100 ppm of Infineum R-650 plus the FOA-3 additive and results of these 3 tests (FLHT, WSIM and D1401) will be compared to the results of the AD-1 (Sample A(4)) plus R-650 alone results.

e. AD-1 (Sample ID A(4)) plus MDFI plus CI (both at their respective, individual concentrations from abaove) will be screened for any possible positive interference using the FLHT final method above (in duplicate at NRL). Single determinations of WSIM and D1401 water shedding tests will be made at Pax. Finally, a sample will be made up from the remaining volume above that will include 100 ppm of Infineum R-650 plus the MDFI and CI additives and results of these 3 tests (HRT, WSIM and D1401) will be compared to the results of the AD-1 (Sample A(4)) plus R-650 alone results.

f. AD-1 (Sample ID A(4)) plus MDFI plus CI plus FOA-3 (all at their respective concentrations individually above) will be screened for any possible positive interference using the FLHT final method above (in duplicate at NRL).Single determinations of WSIM and D1401 water shedding tests will be made at Pax. Finally, a sample will be made up from the remaining volume above that will include 100 ppm of Infineum R-650 plus the MDFI and CI additives and results of these 3 tests (FLHT, WSIM and D1401) will be compared to the results of the AD-1 (Sample A(4)) plus R-650 alone results.

g. The AD-1 (Sample ID A(4)) from 4b-4f above with the greatest positive interference effect on the FLHT will be chosen and combined with the Jet A (Sample BP(2)) plus FSII (4a, above) in a 50:50 blend of total volume about 500 mL. This blend will be screened for any possible positive interference using the FLHT final method above (in duplicate at NRL). This will be the Second Optimization/Restrained Cap method. Next, single determinations of WSIM and D1401 water shedding tests will be done at Pax. Finally, a sample will be made up from the remaining volume that will include 100 ppm of Infineum R-650 plus the MDFI and CI additives and results of these 3 tests (FLHT, WSIM and D1401, or similar to above) will be compared to the results of a 50:50 mixture of AD-1 (Sample A(4)) and Jet A (Sample BP(2)) plus R-650 alone.

h. The Betz additive for thermal stability of jet fuel (Betz 8Q405 which contains only the dispersant portion of the final Air Force (AF) additive package at 50% active ingredient in paraffinic solvent) will be evaluated in Jet A (Sample ID BP(2)) at the additive's required AF minimum concentration of 254 ppm of active ingredient and a separate sample containing 7 ppm, which is close to the Navy shipboard maximum allowed concentration for this additive. The higher concentration sample is expected to show very high haze by FLHT (to be run in duplicate at NRL) and both water separation tests (each run in single determination at Pax). A 500 mL sample volume will be prepared in order to send adequate sample to Pax River for duplicate SLBOCLE and BOCLE tests and to SWRI for duplicate HFRR tests. The purpose of running both the high and low concentration samples will be to ascertain if the Betz 8Q405 dispersant alone imparts any significant improvements in lubricity to the Jet A sample by comparing the lubricity results with those for Jet A (BP(2))alone by obtaining neat Jet A (Sample BP(2)) <u>single</u> determinations for SLBOCLE, BOCLE (at Pax), and a <u>single</u> determination for HFRR (at SwRI).

i. Static Dissipater Additive (SDA) (Octel Stadis 450) will be prepared in AD-1 (Sample ID A(4)) at 1 ppm active ingredient concentration in a 400 mL volume. The sample will be tested for positive interferences using the mechanical FLHT in duplicate at NRL, and WSIM and D1401 as single determinations at Pax. In addition, samples will be sent to Pax River for duplicate SLBOCLE and BOCLE determinations, and to SwRI for duplicate HFRR determinations. Pax River will also conduct a conductivity measurement to ensure proper additive addition. This testing is necessary because several NATO and ABCANZ nations add SDA to their F-76.

5. <u>The Effect of Intermediate Additive Concentrations on the Final Mechanical FLHT Test</u>

a. <u>By FLHT:</u> If necessary, up to three versions of the FLHT test will be used to evaluate intermediate additive effects in duplicate at NRL. The versions are: 1) Manual shaking (1 min shake, wait of 15 min) plus La Mott turbidimeter, 2) Original mechanical version (Jan 06) shake 1 min, 15 min wait, using ring stand friction technique called the First Optimization Friction Method, plus La Mott

turbidimeter, 3) Improved mechanical version (May 06) (shake/wait time TBD) using the confined vial technique, plus La Mott turbidimeter, called the Second Optimization/Restrained Cap Method. (*NOTE*: The turbidimeter must be set to read to the non-rounding setting (Non-EPA-mode)). The additive Infineum R-650 will be tested in Jet A (Sample ID BP(2)) and S-8 (Sample ID BO). The additive will be tested at active ingredient concentrations of 15, 25, 50, 75, and 100 ppm in addition to 0 ppm (neat fuels). *(May - July 2006; NRL)*

b. By HFRR, SLBOCLE and BOCLE: For the same blends in 5a above, conduct RR (in duplicate at SwRI, and SLBOCLE (in duplicate), and BOCLE (in duplicate) at Pax. *May – July 2006; Pax and SwRI)*

6. Determine shipboard acceptability of the HRT final method including use of sodium hydroxide at 0.2 M concentration. *(May and July 2006; NRL)*

7. Finalize the format of the FLHT Navy method. An ASTM Test Method may be developed in the future, if needed. *(September 2006; NRL and Pax)*

Future Work; FY07 if Needed

1. Obtain several petroleum derived poor lubricity fuels and several petroleum derived good lubricity fuels.

2. Precision round robin (using ASTM format) with at least six samples and five labs including additives in the sample matrix. May need to purchase up to three additional LaMott Turbidimeters and four additional mechanical shakers.

3. Implementation plan for the method (Navy/CG/MSC/DESC).

4. Prepare the FY07 Final Report.

5. Prepare journal articles for publication, based on the NRL Memo Report, above.

APPENDIX B

Chemical Lubricity Sample Identification and Notes

NOTES

1. Arctic Diesel #1 (NRL #02-21) – High-sulfur Arctic Diesel fuel from Advanced Engine Technology in Ottawa, Canada. Received at NRL on May 2, 2002 in one steel 55-gallon drum. Drum sent to Pax River in August 2002 and placed in the cold storage box.

2. Arctic Diesel #2 (NRL #02-22) – High-sulfur Arctic Diesel fuel from Advanced Engine Technology in Ottawa, Canada. Received at NRL on May 2, 2002 in one steel 55-gallon drum. Drum sent to Pax River in August 2002 and placed in the cold storage box.

3. BF-2 (NRL #02-19) – Obtained from tank storage in Baltimore (ST Services). Received at NRL on April 3, 2002 in a steel drum. Drum was then sent to Pax River in August 2002 and placed in the cold storage box.

4. Samples A through I (Samples #1 through #9) – Samples C, D, E, G, H, and I were blended at NRL in May 2002 in epoxy lined (with phenolic resin) 1-gallon cans. Samples A, B, and F were drawn off as neat samples at NRL in May 2002, also in epoxy lined (with phenolic resin) 1-gallon cans. Five hundred-mL aliquots were shipped to SwRI on May 22, 2002 in brown borosilicate bottles. Remaining samples A through I in the 1-gallon cans were shipped to Pax River in early August 2002 and placed in the cold box.

5. Samples J through M (Samples #10 through #13) – Samples J, K, and L were blended at Pax River in late November 2002 in 1-liter borosilicate bottles and shipped to SwRI during first week of December 2002. These blends used NRL #02-19, BF-2, obtained from storage in Baltimore (ST Services) and the Isopar M that John Colbert ordered in 2002 for the Reference Fluid for the SLBOCLE at Pax River. Sample M was a neat Isopar M sample, which was transferred into a 1-liter borosilicate bottle at Pax River in late November 2002 and shipped to SwRI during first week of December 2002, along with samples J, K, and L. An aliquot of the original Sample I, blended at NRL in May 2002 in epoxy lined (with phenolic resin) 1-gallon cans, was transferred into a 1-liter borosilicate bottle at Pax River and shipped to SwRI during the first week of December.

6. Samples B(2) through F(2) (Samples #14 through #26) – Samples #15-17, 19-21, and 23-25 were blended in 1-liter borosilicate bottles at Pax River on 27 January 2003. On January 27, 2003 a fresh sample of NRL #02-19, BF-2 (Sample B(2), sample #14), was drawn from the 55-gallon drum into 1-liter borosilicate bottle, at Pax River. Also, fresh samples of NRL #02-21, Arctic Diesel #1 (Sample A(2), sample #22), and NRL #02-22, Arctic Diesel #2 (Sample F(2), sample #26), were drawn from their respective 55-gallon drums into 1-gallon steel cans, at Pax River. These blends were made from these fresh samples that had just been drawn and the Isopar M (Sample M(2), sample #18), that was in storage in the Pax River laboratory freezer, purchased by John Colbert in September 1999 from Exxon. The Pax River SLBOCLE result for

this Isopar was 1500 grams. These samples were shipped to SwRI on 28 January 2003. Neat samples #14, 18, 22, and 26 were transferred into 1-liter borosilicate bottles at Pax River on 27 January 2003, wrapped in aluminum foil, and shipped to SwRI on 28 January 2003.

7. Fresh samples of NRL #02-19, BF-2, (Sample B(2), sample #14), NRL #02-21, Arctic Diesel #1, (Sample A(2), sample #22), and NRL #02-22, Arctic Diesel #2, (Sample F(2), sample #26) – Drawn from their respective 55-gallon drums into 1-liter borosilicate bottles, at Pax River. These clear bottles were then wrapped in aluminum foil. The three samples were taken to NRL on 31 January 2003.

8. Fresh samples of NRL #02-19, BF-2, (Sample B(3), sample #37), NRL #02-21, Arctic Diesel #1, (Sample A(3), sample #27), and NRL #02-22, Arctic Diesel #2, (Sample F(3), sample #38) – Drawn from their respective 55-gallon drums into 5-gallon steel cans on May 5, 2003 at Pax River.

9. Samples D(3) through Z (Samples #28 through #36) – Blended on May 6 and 7, 2003 in 1000-mL borosilicate bottles at Pax River and immediately wrapped in aluminum foil. Seven hundred-mL aliquots of each blend were shipped to SwRI on May 7, 2003. Neat samples #27 (Sample A(3)) and #37 (Sample B(3)) were transferred to 1-l liter borosilicate bottles at Pax River, immediately wrapped in aluminum foil and shipped to SwRI on May 7, 2003.

10. Samples H(3) through AG (Samples #39 through #47) – Blended on May 19 and 20, 2003 in 1000-mL borosilicate bottles at Pax River and immediately wrapped in aluminum foil. Seven hundred-mL aliquots of blends H(3) through AD (Samples #39 through #44) were shipped to SwRI on May 22, 2003. The remaining 3 samples AE through AG (Samples #45 through #47) were shipped to SwRI during the week of 7-11 July 2003. Neat sample F(3) (Sample #38) was transferred to a 1-liter borosilicate bottle, immediately wrapped in aluminum foil and shipped to SwRI on May 22, 2003.

11. Sample M(3) (Sample #48) – Neat sample of Isopar M from new shipment of Isopar M received at Pax River Lab on July 2, 2003. Ordered from ASTM as a reference fluid for the SLBOCLE. A 700-mL aliquot was transferred to a borosilicate bottle, wrapped in aluminum foil and sent to SwRI during week of 7-11 July 2003.

12. Samples K(3) through AN (Samples #49 through #57) – Blended on July 3, 2003 in 1000-mL clear borosilicate bottles at Pax River and immediately wrapped in aluminum foil. Seven hundred-mL aliquots of blends K(3) through AN (Samples #49 through #57) were shipped to SwRI during the week of 7-11 July 2003.

13. Samples BA through BE (Samples #58 through #62) – Blended on November 17 and 18, 2003 at Pax River in clear borosilicate bottles and immediately wrapped in aluminum foil. These samples were blended using Arctic Diesel #1 diesel fuel (NRL #02-21) from a 5-gallon can, which was taken from the 55-gallon drum on November 14, 2003 and a 5-gallon

can of Biodiesel from World Energy, which contains a stabilizer additive. Six hundred-mL aliquots of blends BA through BE (Samples #58 through #62) were transferred to clear 1 liter borosilicate bottles, blanketed with nitrogen and wrapped in aluminum foil, were shipped to SwRI during the week of December 1-5, 2003. A 50-mL aliquot of each blend was taken to NRL on November 25, 2003 for the new haze-rating test. None of the clear borosilicate vials taken to NRL were wrapped.

14. Sample BF (Sample #63) – Neat sample of World Energy Biodiesel. A 600-mL aliquot was transferred to a clear borosilicate bottle, immediately wrapped in aluminum foil, and shipped to SwRI during the week of December 1-5, 2003. A 50-mL aliquot was taken to NRL on November 25, 2003. The clear borosilicate vial taken to NRL was not wrapped.

15. Samples BG through BL (Samples #64 through #69) – Blended on January 6 and 7, 2004 using Arctic Diesel #1 (NRL #02-21) from a 5-gallon can which was taken from the 55-gallon drum on November 14, 2003 and a sample of DCI- 4A from the Pax River supply. Six different levels of the additive were used. Six hundred-mL aliquots of blends BG through BL (Samples #64 through #69), blanketed with nitrogen, and wrapped in aluminum foil, were shipped to SwRI during the week of January 12, 2004. Clear borosilicate bottles were used for the SwRI samples. Fifty-mL aliquots of each sample were taken to NRL during the week of January 12, 2004 for the new haze-rating test. None of the clear borosilicate vials taken to NRL were wrapped

16. Samples A(4) and B(4) (Samples #70 and #71) – NRL #02-21, Arctic Diesel #1 (Sample A(4)), and NRL #02-19, BF-2 (Sample B(4)). A fresh sample of each fuel was drawn from their respective 55-gallon drum into a 5-gallon steel can in mid-March 2005. A 400-mL sample of each fuel was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL sample of each fuel (in clear borosilicate glass bottles, wrapped in aluminum foil) was shipped to SwRI on May 5, 2005 for HFRR lubricity testing. No additional sample was required at NRL since a sufficient quantity already existed at NRL.

17. Sample BM (Sample #72) – Approximately 2-gallons of the freshly drawn BF-2 (Sample B(4)), stored at Pax River, was clay treated by allowing the fuel to drip slowly through a column of clay. A 400-mL aliquot of this sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of this sample was sent to SwRI on May 5, 2005 for HFRR lubricity testing. A 40-mL aliquot of this sample was taken to NRL for FLHT testing.

Note: The vial size was changed from 50 mL to 40 mL since it was a new stock of vials that were on-hand at the Pax River Lab. The vials were clear borosilicate glass and were not wrapped.

18. Sample BN (Sample #73) – Approximately 2-gallons of the Jet A Sample (BP) supplied by SwRI (fuel number AL-27069) and stored at Pax River, was clay treated at the Pax River lab by allowing the fuel to drip slowly through a column of clay. A 400-mL aliquot of this sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of this sample was sent to SwRI on May 5, 2005 for HFRR lubricity testing.

A clear borosilicate bottle was used for the SwRI sample. A 40-mL aliquot of this sample, in clear, unwrapped borosilicate glass, was taken to NRL for FLHT testing.

19. Sample BO (Sample #74) – A fresh sample of S-8 (SwRI AL-27074) was drawn from the 55-gallon drum stored at Pax River into a 5-gallon steel can in mid-March 2005. A 400-mL aliquot of this sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of this sample was sent to SwRI on May 5, 2005 for HFRR lubricity testing. A 40-mL aliquot of this sample was taken to NRL for FLHT testing. The sample for SwRI and NRL was stored in clear borosilicate glass. The SwRI sample was wrapped in aluminum foil, while the NRL sample was not. This is the sample in the SwRI hardware lubricity studies that is known as Fuel 1000. See note #49 and #50 below for the other two fuels in the hardware study.

20. Samples BP (SwRI AL-27069) and BQ (SwRI AL-26927) [Samples #75 and #76] – A 5-gallon sample of each fuel was shipped to Pax River from SwRI. Sample BP is non-additized Jet A, and Sample BQ is a high lubricity ULSD fuel. It is not known whether the high lubricity ULSD fuel contains a lubricity additive, or if it has high lubricity due to the type of processing used to remove the sulfur. A 400-mL aliquot of each sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was shipped to SwRI on May 5, 2005 for HFRR lubricity testing. A 40-mL aliquot of each sample was taken to NRL for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

21. Sample BR (Sample #77) – A composite sample consisting of numerous low lubricity fuels from the SwRI severely hydrotreated samples that were stored at NRL. The samples came from a set of six different F-76-type fuels from various locations around the world, all of which had very good, initial fuel lubricity characteristics. These fuels were severely hydrotreated at SwRI, to four different hydrotreatment levels, each succeeding level more severe than the previous one, which reduced the lubricity levels dramatically. The hydrotreatment samples that NRL chose to make up the composite were from hydrotreatment levels 3 and 4 (HT3 and HT4), which were the levels exhibiting the poorest lubricity characteristics. The final sample volume was generated at NRL by pouring them all into a 5-gallon unlined steel can that was provided by Pax River. Some minimal, remaining quantities of HT3 and HT4 were saved at NRL. The total final composite volume was approximately 2-1/2-gallons. A 400-mL aliquot of this composite sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of this sample was shipped to SwRI on May 5, 2005 for HFRR lubricity testing. A 40-mL aliquot of this sample was taken to NRL for FLHT testing. The sample for SwRI and NRL was stored in clear borosilicate glass. The SwRI sample was wrapped in aluminum foil, while the NRL sample was not.

22. Sample BS (Sample #78) – A blend of 50% Jet A (Sample BP) and 50% clay treated Jet A (Sample BN) was prepared in the Pax River lab. A 400-mL aliquot of this sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot was shipped to SwRI for HFRR lubricity testing. A 40-mL aliquot of this sample was

taken to NRL for FLHT testing. The sample for SwRI and NRL was stored in clear borosilicate glass. The SwRI sample was wrapped in aluminum foil, while the NRL sample was not. The sample was shipped to SwRI in late March/early April 2005. During this same time frame, the sample was taken to NRL.

23. Sample BT (Sample #79) – A blend of 50% BF-2 (Sample B(4)) and 50% Arctic Diesel #1 (NRL #02-21, Sample A(4)) was prepared in the Pax River lab in early May 2005. A 400-mL aliquot of this sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of this sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of this sample was taken to NRL for FLHT testing. The sample for SwRI and NRL was stored in clear borosilicate glass. The SwRI sample was wrapped in aluminum foil, while the NRL sample was not.

24. Sample BU (Sample #80) – A 5-gallon sample of an EPA emissions certification Grade No. 2 Diesel Fuel with good lubricity (SwRI AL-27070) was shipped to the Pax River Lab from SwRI. A 400-mL aliquot of this sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing in early May 2005. A 400-mL aliquot of this sample was sent to SwRI for HFRR lubricity testing. An aliquot of approximately 1-liter was taken to NRL for FLHT testing in late August 2005, and might be used for additional testing at NRL. The sample for SwRI and NRL was stored in clear borosilicate glass. The SwRI sample was wrapped in aluminum foil, while the NRL sample was not.

25. Samples BV through CD (Samples #81 through #89) – Blends of S-8 (Sample BO) and BF-2 (Sample B(4)) in 10% increments were prepared at Pax River in early May 2005 beginning with Sample BV (10% S-8 and 90% BF-2) and ending with Sample CD (90% S-8 and 10% BF-2). A 400-mL aliquot of each sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was sent to SwRI for HFRR lubricity testing in May 2005. A 40-mL aliquot of each sample was taken to NRL in late August 2005 for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

26. Samples CE through CH (Samples #90 through #93) – Blends of 15 ppm, 100 ppm, 200 ppm and 500 ppm respectively of Infineum R-690 additive (active ingredient *assumed* to be 100%) in S-8 (Sample BO) were prepared at Pax River in early June 2005. A 400-mL aliquot of each sample was submitted to the Pax River Lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of each sample was taken to NRL for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

27. Samples CI through CK (Samples #94 through #96) – Blends of 15 ppm, 100 ppm and 500 ppm respectively of Infineum R-690 additive (active ingredient *assumed* to be 100%) in Jet A (Sample BP) were prepared at Pax River in early June 2005. A 400-mL aliquot of each sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was sent to SwRI for HFRR lubricity testing. A 40-mL

aliquot of each sample was taken to NRL for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

28. Samples CL through CN (Samples #97 through #99) – Blends of 15 ppm, 100 ppm and 500 ppm respectively of Infineum R-690 additive (active ingredient *assumed* to be 100%) in Composite Sample (Sample BR) were prepared at Pax River in early June 2005. A 400-mL aliquot of each sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of each sample was taken to NRL for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

29. Samples CO through CQ (Samples #100 through #102) – Blends of 15 ppm, 100 ppm and 500 ppm respectively of Infineum R-690 additive (active ingredient *assumed* to be 100%) in Arctic Diesel #1 (Sample A(4)) were prepared at Pax River in early June 2005. A 400-mL aliquot of each sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of each sample was taken to NRL for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

30. Samples CR through CT (Samples #103 through #105) – Blends of 15 ppm, 100 ppm and 500 ppm respectively of Infineum R-650 additive (active ingredient *assumed* to be 100%) in S-8 (Sample BO) were prepared at Pax River in early July 2005. A 400-mL aliquot of each sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of each sample was taken to NRL for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

31. Samples CU through CW (Samples #106 through #108) – Blends of 15 ppm, 100 ppm and 500 ppm respectively of Infineum R-650 additive (active ingredient *assumed* to be 100%) in Jet A (Sample BP) were prepared at Pax River in early July 2005. A 400-mL aliquot of each sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of each sample was taken to NRL for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

32. Sample CX (Sample #109) – A blend of 50% S-8 (Sample BO) and 50% Jet A (Sample BP) was prepared at Pax River in early July 2005. A 400-mL aliquot of this sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of this sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of this sample was taken to NRL for FLHT testing. The sample for SwRI and NRL was stored in

clear borosilicate glass. The SwRI sample was wrapped in aluminum foil, while the NRL sample was not.

33. Sample CY (Sample #110) – A blend of 50% S-8 (Sample BO) and 50% clay treated Jet A (sample BN) was prepared at Pax River in early July 2005. A 400-mL aliquot of this sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of this sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of this sample was taken to NRL for FLHT testing. The sample for SwRI and NRL was stored in clear borosilicate glass. The SwRI sample was wrapped in aluminum foil, while the NRL sample was not.

34. Sample CZ (Sample #111) – A blend of 50% clay treated Jet A (Sample BN) and 50% Composite Fuel from NRL (Sample BR) was prepared at Pax River in early July 2005. A 400-mL aliquot of this sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of this sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of this sample was taken to NRL for FLHT testing. The sample for SwRI and NRL was stored in clear borosilicate glass. The SwRI sample was wrapped in aluminum foil, while the NRL sample was not.

35. Sample DA (Sample #112) – A blend of approximately 10% Composite Fuel (Sample BR) and 90% Arctic Diesel #2 (Sample F(3)) was prepared at Pax River in late July 2005 to be used as a low lubricity replacement for Sample BR. Sample BR was nearly depleted and there was not enough to complete all of the required sample blending. A 400-mL aliquot of this sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of this sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of this sample was taken to NRL for FLHT testing. The sample for SwRI and NRL was stored in clear borosilicate glass. The SwRI sample was wrapped in aluminum foil, while the NRL sample was not.

36. Samples DB through DD (Samples #113 through #115) – Blends of 15 ppm, 100 ppm and 500 ppm respectively of DCI-4A additive (active ingredient *assumed* to be 100%) in S-8 (Sample BO) were prepared at Pax River in late July 2005. A 400-mL aliquot of each sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of each sample was taken to NRL for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

37. Samples DE through DG (Samples #116 through #118) – Blends of 15 ppm, 100 ppm and 500 ppm respectively of DCI-4A additive (active ingredient *assumed* to be 100%) in Jet A (Sample BP) were prepared at Pax River in late July 2005. A 400-mL aliquot of each sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of each sample was taken to NRL for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

38. Samples DH through DJ (Samples #119 through #121) – Blends of 15 ppm, 100 ppm and 500 ppm respectively of Infineum R-690 additive (active ingredient *assumed* to be 100%) in clay treated Jet A (Sample BN) were prepared at Pax River in late July 2005. A 400-mL aliquot of each sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of each sample was taken to NRL for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

39. Samples DK through DM (Samples #122 through #124) – Blends of 15 ppm, 100 ppm and 500 ppm respectively of Infineum R-690 additive (active ingredient *assumed* to be 100%) in clay treated BF-2 (Sample BM) were prepared at Pax River in late July 2005. A 400-mL aliquot of each sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of each sample was taken to NRL for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

40. Samples DN through DP (Samples #125 through #127) – Blends of 15 ppm, 100 ppm and 500 ppm respectively of Infineum R-650 additive (active ingredient *assumed* to be 100%) in new Diesel Composite (Sample DA) were prepared at Pax River in late August 2005. A 400-mL aliquot of each sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of each sample was taken to NRL for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

41. Samples DQ through DS (Samples #128 through #130) – Blends of 15 ppm, 100 ppm and 500 ppm respectively of Infineum R-650 additive (active ingredient *assumed* to be 100%) in Arctic Diesel #1 (Sample A(4)) were prepared at Pax River in late August 2005. A 400-mL aliquot of each sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of each sample was taken to NRL for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

42. Samples DT through DV (Samples #131 through #133) – Blends of 15 ppm, 100 ppm and 500 ppm respectively of Infineum R-650 additive (active ingredient *assumed* to be 100%) in clay treated Jet A (Sample BN) were prepared at Pax River in early September 2005. A 400-mL aliquot of each sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of each sample was taken to NRL for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

43. Samples DW through DY (Samples #134 through #136) – Blends of 15 ppm, 100 ppm and \ 500 ppm respectively of Infineum R-650 additive (active ingredient *assumed* to be 100%) in clay treated BF-2 (Sample BM) were prepared at Pax River in early September 2005. A 400-mL aliquot of each sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of each sample was taken to NRL for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

44. Samples DZ through EB (Samples #137 through #139) – Blends of 15 ppm, 100 ppm and 500 ppm respectively of DCI-4A additive (active ingredient measured to be 80%) in the new Diesel Composite (Sample DA) were prepared at Pax River in mid September 2005. A 400-mL aliquot of each sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of each sample was taken to NRL for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

45. Samples EC through EE (Samples #140 through #142) – Blends of 15 ppm, 100 ppm and 500 ppm respectively of DCI-4A additive (active ingredient measured to be 80%) in Arctic Diesel #1 (Sample A(4)) were prepared at Pax River in mid September 2005. A 400-mL aliquot of each sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of each sample was taken to NRL for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

46. Samples EF through EH (Samples #143 through #145) – Blends of 15 ppm, 100 ppm and 500 ppm respectively of DCI-4A additive (active ingredient measured to be 80%) in clay treated Jet A (Sample BN) were prepared at Pax River in mid September 2005. A 400-mL aliquot of each sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of each sample was taken to NRL for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

47. Sample EI through EK (Samples #146 through #148) – Blends of 15 ppm, 100 ppm and 500 ppm respectively of DCI-4A additive (active ingredient measured to be 80%) in clay treated BF-2 (Sample BM) were prepared at Pax River in mid September 2005. A 400-mL aliquot of each sample was submitted to the Pax River lab for BOCLE and SLBOCLE lubricity testing. A 400-mL aliquot of each sample was sent to SwRI for HFRR lubricity testing. A 40-mL aliquot of each sample was taken to NRL for FLHT testing. The samples for SwRI and NRL were stored in clear borosilicate glass. The SwRI samples were wrapped in aluminum foil, while the NRL samples were not.

48. Samples EL through EU *(sample identification EO not used)* (Sample #149 through #157) – Samples were prepared at NRL, by Dennis Hardy, on September 14, 2005 for the FY 05 Test Plan Task 10 blind comparison study of the FLHT. EL and EM were 25 ppm and 75 ppm respectively of Infineum R-690 additive (active ingredient *assumed* to be 100%) in Sample A(4) Arctic Diesel #1 (NRL #02-21). EN and EP (sample code EO not used) were 25 ppm and 75 ppm respectively of Infineum R-690 additive in Arco Hydrotreat Level 3 (HT3) from the 1998 diesel fuel upgrading study at SwRI. The neat Arco HT3 gave NTU of 10. Samples EQ through EU were mixtures of Haifa Hydrotreat Level 4 (HT4) (NRL #98-99) and Arco feedstock F-76 (EW, also NRL #99-3) from the 1998 diesel fuel upgrading study at SwRI, respectively (Haifa/Arco), 80/20 v/v, 88/12 v/v, 93/7 v/v, 97/3 v/v and 100/0 v/v. The neat Haifa HT4 gave NTU of 4 and the neat Arco feedstock gave "err 3" (>1100). All samples were in 125-mL brown borosilicate glass bottles and therefore did not have to be wrapped.

49. Sample BP(2) (Sample # 158) – This was a separate aliquot of sample BP (sample #75 -

50. Sample EV (Sample # 159) – This sample came from SwRI and was received at NRL and Pax in early 2006. This is an EPA diesel fuel of high lubricity as defined by SLBOCLE results (and other subsequent analysis). It is known in the SwRI hardware lubricity test program as Fuel 3000. See note #19 and note #49 for the other two fuels in this hardware study.

51. Sample EW (Sample # 160) – This is a very high lubricity diesel fuel sample was sent to NRL much earlier from a SwRI study on the effects of hydrotreating on diesel lubricity. This ARCO feedstock (NRL # 99-3) was used in the preparation of sample codes EL to EU above (see note #48 above).

52. Sample A(5) (Sample # 161) - NRL #02-21, Arctic Diesel #1. On a June 20, 2006, a five-gallon can was created out of three smaller samples of AD-1, most likely ranging from A to A(4). Used at NRL only for FY 06 Test Plan.

53. Sample EX (Sample # 162) - A 500-mL sample of 50% Arctic Diesel #1 (A(4)) and 50% BF-2 (B(4)) was prepared at NRL, by Rachel Hodges, on June 22, 2006. Of this volume, 20-mL remained from a 100-mL sample from May 16, 2006 by Rachel Hodges, 150-mL from a 500-mL sample from December 28, 2005 by Rachel Hodges, and 300-mL were added together on June 22, 2006. A 50-mL sample was kept at NRL for FLHT testing, a 300-mL sample was stored in a 600-mL brown borosilicate bottle and sent to Pax River (July 2006) for duplicate SLBOCLE and BOCLE testing, and a 150-mL sample was stored in a 150-mL brown borosilicate bottle and sent to SwRI for duplicate HFRR testing (July 2006).

54. Sample EY (Sample # 163) - A 500-mL sample of 55% BU and 45% Arctic Diesel #1 (A(5)) was created on June 22, 2006 by Rachel Hodges, at NRL. Of this volume, 20-mL remained from a 500-mL sample of the same blend from January 6, 2006 by Rachel Hodges. A 50-mL sample was kept at NRL for FLHT testing, a 300-mL sample was stored in a 600-mL brown borosilicate bottle and sent to Pax River (July 2006) for duplicate SLBOCLE and BOCLE

testing, and a 150-mL sample was stored in a 150-mL brown borosilicate bottle and sent to SwRI for duplicate HFRR testing (July 2006).

55. Sample EZ (Sample # 164) - A 500-mL sample of 13% ARCO (EW) and 87% Arctic Diesel #1 (A(5)) was created by Rachel Hodges at NRL on June 23, 2006. A 50-mL sample was kept at NRL for FLHT testing, a 300-mL sample was stored in a 600-mL brown borosilicate bottle and sent to Pax (July 2006) for duplicate SLBOCLE and BOCLE testing, and a 150-mL sample was stored in a 150-mL brown borosilicate bottle and sent to SwRI for duplicate HFRR testing (July 2006).

56. Samples FA-FB (Sample # 165 and # 166) - A 250-mL sample of 0.1% FSII in Jet A (BP(2)) was prepared at NRL, by Rachel Hodges, on June 19, 2006. A 50-mL sample was kept at NRL for analyses; a 200-mL aliquot was sent to Pax River (July 2006) for WSIM and D1401 testing (FA). A 20-mL sample of 0.1% FSII and 100 ppm Infineum R-650 additive in Jet A (BP(2)) was prepared at NRL, by Rachel Hodges, on June 19, 2006 (FB). A 5-mL sample was kept at NRL for FLHT analysis; a 150-mL aliquot was sent to Pax River (July 2006) for single WSIM and D 1401 testing. Both samples were stored in 600-mL brown borosilicate glass bottles.

57. Sample FC (Sample # 167) - A 150-mL sample of 100 ppm Infineum Additive R-650 in Jet A (BP(2)) was prepared at NRL, by Rachel Hodges, on June 21, 2006. A 50-mL sample was kept at NRL for FLHT analysis; an aliquot of approximately 100-mL was sent to Pax River (July 2006) for single WSIM and D 1401 testing. They were stored in a 150-mL brown borosilicate glass bottle.

58. Samples FD-FF (Samples # 168 through # 170) - Samples were created by Rachel Hodges, at NRL, on June 9, 2006. A 750-mL sample of Arctic Diesel #1 (A(4)) was created with 0.1% (1000 ppm) Middle Distillate Flow Improver (MDFI) Keroflux from BASF. Of this amount, a 250-mL sample was set aside as sample FD. In the 500-mL remaining, 0.2% Cetane Improver (CI) was added, and a 250-mL sample was set aside as sample FE, containing both CI and MDFI. Of the last 250-mL, 25 ppm Fuel Oil Additive 3 (FOA-3) was added to create sample FF. For all three samples, a 50-mL sample was kept at NRL for FLHT analysis and an aliquot of 200-mL was sent to Pax River (July 2006) for single WSIM and D 1401 testing. They were stored in 600-mL brown borosilicate glass bottles.

59. Samples FG-FJ (Samples # 171 through # 174) - Samples were created by Rachel Hodges, at NRL, on June 20, 2006. A 600-mL sample of Arctic Diesel #1 was created with 100 ppm Infineum R-650. Of this, a 150-mL sample was set aside as sample FG. In the 450-mL remaining, 0.1% MDFI (Keroflux from BASF) was added, and a 150-mL sample was set aside as sample FH. In the 300-mL remaining, 0.2% CI was added, and a 150-mL sample was set aside as sample FI. In the last 150-mL, 25 ppm FOA-3 was added as sample FJ. For each of the four samples, a 50-mL sample was kept at NRL for FLHT analysis and 100 mL were sent to Pax River (July 2006) for single WSIM and D 1401 testing. They were stored in 150-mL brown borosilicate glass bottles.

60. Sample FK (Sample # 175) - A 250-mL sample of 25 ppm FOA-3 in Arctic Diesel #1 (A(4)) was created by Rachel Hodges, at NRL, on June 19, 2006. The 250-mL was combined with approximately 30-mL that remained of the same blend created on May 18, 2006. A 50-mL aliquot was kept at NRL for FLHT analysis and the remaining 250-mL sample was stored in a 600-mL brown borosilicate glass bottle and sent to Pax River (July 2006) for single WSIM and D 1401 testing.

61. Sample FL (Sample # 176) - A 250-mL sample of 0.2% CI in Arctic Diesel #1 (A(4)) was created by Rachel Hodges, at NRL, on June 19, 2006. The 250-mL was combined with approximately 30-mL that remained of the same blend created on May 30, 2006. A 50-mL aliquot was kept at NRL for FLHT analysis and the remaining 200-mL sample was stored in a 600-mL brown borosilicate glass bottle and sent to Pax River (July 2006) for single WSIM and D 1401 testing.

62. Samples FM-FN (Samples # 177 and # 178) - A 400-mL sample of Infineum R-650 additive In Arctic Diesel #1 (A(4)) was created by Rachel Hodges, at NRL, on June 13, 2006. A 200-mL sample was removed and 25 ppm FOA-3 was added as sample FM. Of this amount, a 50-mL sample was kept for FLHT analysis at NRL, the remaining 150-mL sample was stored in 150-mL brown borosilicate glass bottle and sent to Pax River for single WSIM and D 1401 testing. To the remaining 200-mL, 0.2% CI was added as sample FN. For FN, a 50-mL sample was kept for FLHT analysis at NRL, the remaining 150-mL sample was stored in a 600-mL brown borosilicate glass bottle and sent to Pax River for single WSIM and D 1401 testing.

63. Samples FO-FP (Samples # 179 and # 180) - 500-mL of a 50:50 blend of Jet A (BP(2)) with 0.1% FSII, and Arctic Diesel #1 (A(4)) with 0.1% MDFI, 0.2% CI, and 25 ppm FOA-3 was created by Rachel Hodges on June 14, and 19, 2006. On June 14, a 250-mL sample was created, 33-mL from an existing FA sample, 92-mL from a new FA sample, and 125-mL from a new FF sample. On June 19, 2006, 250-mL more was added to the previous 250-mL: 125-mL as a new FA sample and 125-mL as a new FF sample. Thus, 500-mL of a 50:50 blend of FA and FF was created as sample FO. Of this sample, a 50-mL sample was kept for FLHT analysis at NRL and a 300-mL sample was bottled in a 600-mL brown borosilicate glass bottle and sent Pax River for single WSIM and D 1401 testing (July 2006). The last 150-mL sample was removed and 100 ppm Infineum R-650 was added as sample FP, of which a 50-mL sample was kept for FLHT analysis at NRL and 100-mL was sent to Pax River (July 2006) for single WSIM and D 1401 testing. The Pax River sample was stored in a 150-mL brown borosilicate bottle.

64. Samples FQ-FR (Samples # 181 and # 182) - A 400-mL sample of 50% Jet A (BP(2)) and 50% Arctic Diesel #1 (A(5)) was created by Rachel Hodges on June 22, 2006 at NRL. Of this amount, a 150-mL sample was set aside as sample FQ. In the remaining 250-mL, 100 ppm Infineum R-650 was added, as sample FR, of which a 50-mL sample was kept for FLHT analysis at NRL and the remaining 200-mL sample was sent to Pax River for single WSIM and D 1401 testing. The sample was stored in a 600-mL brown borosilicate bottle.

65. Samples FS-FT (Samples #183 and # 184) - 600-mL of a 178 ppm Betz 8Q405 additive in BP(2) was created by Rachel Hodges on June 1, 2006, at NRL. Of this amount, a 20-mL sample was removed and combined with 480-mL of neat BP(2) to create a 7.0 ppm solution as sample FS. On June 15, 2006, additional Betz 8Q405 additive was added to the too-dilute original solution to create 374 ppm 8Q405 in 500-mL Jet A (BP(2)) as sample FT. Of each sample, a 50-mL sample was kept at NRL for FLHT analysis, a 300-mL sample was sent to Pax River (July 2006) for single WSIM, and D 1401, and duplicate SLBOCLE, and BOCLE testing. The Pax River sample was stored in a 600-mL brown borosilicate bottle, and the remaining 150-mL was sent to SwRI (July 2006) for HFRR testing. The SwRI sample was stored in a 150-mL brown borosilicate bottle.

66. Sample FU (Sample # 185) - A 400-mL solution of 1.1 ppm SDA in Arctic Diesel #1 (A(4)) was created by Dennis Hardy and Kathy Lewis on May 19, 2006 as sample FU. On June 28, 2006, Rachel Hodges at NRL added 300-mL more of 1.1 ppm SDA in Arctic Diesel #1, of which 50-mL was A(4) and 250-mL was A(5). Of the overall sum, a 100-mL sample was kept at NRL for FLHT analysis, a 450-mL sample was sent to Pax River in a 600-mL brown borosilicate bottle for single WSIM and D 1401, and duplicate SLBOCLE and BOCLE testing, and a 150-mL sample was sent to SwRI for duplicate HFRR testing, which was stored in a 150-mL brown borosilicate bottle.

67. Samples FV-FZ (Samples # 186 through # 190) - A 1500-mL sample of 100 ppm Infineum R-650 in Jet A (BP(2)) was created by Rachel Hodges, at NRL, on May 31, 2006. Of this amount, a 75-mL sample was extracted and combined with 425-mL of neat Jet A (BP(2)) to form a 15 ppm R-650 sample called FV. A 125-mL sample of the original solution was combined with 375-mL of neat Jet A (BP(2)) to form a 25 ppm R-650 sample called FW. A 250-mL sample of the original solution plus 250-mL neat Jet A (BP(2)) were combined to form a 50 ppm R-650 sample called FX. 375-mL of the original solution was combined with 125-mL neat Jet A (BP(2)) to create a 75 ppm sample called FY. The remainder of the original 100 ppm R-650 solution, about 600-mL, was called sample FZ. Of this, a 50-mL sample was kept at NRL for FLHT analysis, a 300-mL sample was sent to Pax River (July 2006) for duplicate SLBOCLE and BOCLE testing, which was stored in a 600-mL brown borosilicate bottle, and a 125-mL sample was sent to SwRI (July 2006), for duplicate HFRR testing, and was also stored in a 150-mL brown borosilicate bottle.

68. Samples GA-GE (Samples # 191 through # 195) - A 1500-mL sample of 100 ppm Infineum R-650 in S-8 (BO) was created by Rachel Hodges, at NRL, on May 31, 2006. Of this amount, a 75-mL sample was extracted and combined with 425-mL neat S-8 (BO) to form a 15 ppm R-650 sample called GA. 125-mL of the original solution was combined with 375-mL of neat S-8 (BO) to form a 25 ppm R-650 sample called GB. 250-mL of the original solution plus 250-mL neat S-8 (BO) were combined to form a 50 ppm R-650 sampled called GC. 375-mL of the original solution was combined with 125-mL neat S-8 (BO) to create a 75 ppm sample called GD. The remainder of the original 100 ppm R-650 solution, about 600-mL, was called sample GE. Of these, a 50-mL sample was kept at NRL for FLHT analysis, a 300-mL sample was sent to Pax River (July 2006) for duplicate SLBOCLE and BOCLE testing, and was stored in a 600-mL brown borosilicate bottle, and a 125-mL sample was sent

to SwRI for duplicate HFRR testing, which was also stored in a 600-mL brown borosilicate bottle.

69. Samples GF-GM (Samples #196 through #203 – These are duplicate samples made by R. Hodges at NRL in August 2006 and tested for FLHT at NRL with a separate aliquot sent to Pax River for WSIM and D1401 testing in addition to SLBOCLE and BOCLE testing. The samples and aliquots sent to Pax River were made in brown borosilicate bottles.

Lubricity Sample Identification

N.B. NRL or Pax Sample ID's having numbers in parenthesis refer to replicate aliquots or mixtures. See previous "Notes" for details.

Number	SwRI Sample ID	NRL or Pax Sample ID	Actual Sample Identification
1	CL02-0421	A	Arctic Diesel #1 (NRL #02-21) Data from AET: BOTD = 760 μm Sulfur = 110 ppm
2	CL02-0422	B	NRL #02-19 (BF-2)
3	CL02-0423	C	1% BF-2 99% Arctic Diesel #1
4	CL02-0424	D	10% BF-2 90% Arctic Diesel #1
5	CL02-0425	E	50% BF-2 50% Arctic Diesel #1
6	CL02-0426	F	Arctic Diesel #2 (NRL #02-22) Data from AET: BOTD = 643 μm Sulfur = 360 ppm
7	CL02-0427	G	1% BF-2 99% Arctic Diesel #2
8	CL02-0428	H	10% BF-2 90% Arctic Diesel #2
9	CL02-0429	I	50% BF-2 50% Arctic Diesel #2
10	CL02-1042	J	50% BF-2 50% Isopar M
11	CL02-1043	K	10% BF-2 90% Isopar M
12	CL02-1044	L	1% BF-2 99% Isopar M

Number	SwRI Sample ID	NRL or Pax Sample ID	Actual Sample Identification
13	CL02-1045	M	100% Isopar M
14	CL03-0135	B(2)	100% BF-2 (NRL #02-19)
15	CL03-0139	K(2)	10% BF-2 90% Isopar M
16	CL03-0141	N	7% BF-2 93% Isopar M
17	CL03-0142	O	4% BF-2 96% Isopar M
18	CL03-0140	M(2)	100% Isopar M
19	CL03-0136	D(2)	10% BF-2 90% Arctic Diesel #1
20	CL03-0143	P	7% BF-2 93% Arctic Diesel #1
21	CL03-0144	Q	4% BF-2 96% Arctic Diesel #1
22	CL03-0134	A(2)	100% Arctic Diesel #1
23	CL03-0138	H(2)	10% BF-2 90% Arctic Diesel #2
24	CL03-0145	R	7% BF-2 93% Arctic Diesel #2
25	CL03-0146	S	4% BF-2 96% Arctic Diesel #2
26	CL03-0137	F(2)	100% Arctic Diesel #2

Number	SwRI Sample ID	NRL or Pax Sample ID	Actual Sample Identification
27	CL03-0398	A(3)	100% Arctic Diesel #1
28	CL03-0400	D(3)	10% BF-2 90% Arctic Diesel #1
29	CL03-0401	T	20% BF-2 80% Arctic Diesel #1
30	CL03-0402	U	30% BF-2 70% Arctic Diesel #1
31	CL03-0403	V	40% BF-2 60% Arctic Diesel #1
32	CL03-0462	E(2)	50% BF-2 50% Arctic Diesel #1
33	CL03-0466	W	60% BF-2 40% Arctic Diesel #1
34	CL03-0467	X	70% BF-2 30% Arctic Diesel #1
35	CL03-0468	Y	80% BF-2 20% Arctic Diesel #1
36	CL03-0469	Z	90% BF-2 10% Arctic Diesel #1
37	CL03-00399	B(3)	100% BF-2 (NRL #02-19)
38	CL03-0463	F(3)	100% Arctic Diesel #2
39	CL03-0464	H(3)	10% BF-2 90% Arctic Diesel #2
40	CL03-0458	AA	20% BF-2 80% Arctic Diesel #2

Number	SwRI Sample ID	NRL or Pax Sample ID	Actual Sample Identification
41	CL03-0459	AB	30% BF-2 70% Arctic Diesel #2
42	CL03-0460	AC	40% BF-2 60% Arctic Diesel #2
43	CL03-0465	I(2)	50% BF-2 50% Arctic Diesel #2
44	CL03-0461	AD	60% BF-2 40% Arctic Diesel #2
45	CL03-0763	AE	70% BF-2 30% Arctic Diesel #2
46	CL03-0764	AF	80% BF-2 20% Arctic Diesel #2
47	CL03-0765	AG	90% BF-2 10% Arctic Diesel #2
48	CL03-0775	M(3)	100% Isopar M (new)
49	CL03-0774	K(3)	10% BF-2 90% Isopar M
50	CL03-0766	AH	20% BF-2 80% Isopar M
51	CL03-0767	AI	30% BF-2 70% Isopar M
52	CL03-0768	AJ	40% BF-2 60% Isopar M
53	CL03-0773	J(2)	50% BF-2 50% Isopar M
54	CL03-0769	AK	60% BF-2 40% Isopar M

Number	SwRI Sample ID	NRL or Pax Sample ID	Actual Sample Identification
55	CL03-0770	AL	70% BF-2 30% Isopar M
56	CL03-0771	AM	80% BF-2 20% Isopar M
57	CL03-0772	AN	90% BF-2 10% Isopar M
58	CL03-1002	BA	99.5% Arctic Diesel #1 0.5% Biodiesel
59	CL03-1003	BB	99% Arctic Diesel #1 1% Biodiesel
60	CL03-1004	BC	98% Arctic Diesel #1 2% Biodiesel
61	CL03-1005	BD	95% Arctic Diesel #1 5% Biodiesel
62	CL03-1006	BE	80% Arctic Diesel #1 20% Biodiesel
63	CL03-1007	BF	100% Biodiesel
64	CL04-0025	BG	15 ppm DCI-4A in Arctic Diesel #1
65	CL04-0026	BH	50 ppm DCI-4A in Arctic Diesel #1
66	CL04-0027	BI	100 ppm DCI-4A in Arctic Diesel #1
67	CL04-0028	BJ	250 ppm DCI-4A in Arctic Diesel #1
68	CL04-0029	BK	500 ppm DCI-4A in Arctic Diesel #1

Number	SwRI Sample ID	NRL or Pax Sample ID	Actual Sample Identification
69	CL04-0030	BL	1000 ppm DCI-4A in Arctic Diesel #1
70	CL05-0238	A(4)	100% Arctic Diesel #1
71	CL05-0239	B(4)	100% BF-2 (NRL #02-19)
72	CL05-0240	BM	100% Clay Treated BF-2
73	CL05-0241	BN	100% Clay Treated Jet-A
74	CL05-0242	BO	100% Syntroleum S-8 (SwRI AL-27074)
75	CL05-0243	BP	100% Jet-A (non-additized) (SwRI AL-27069)
76	CL05-0244	BQ	100% ULSD Good Lubricity (SwRI AL-26927)
77	CL05-0245	BR	Composite of ULSD low lubricity, from SwRI hydrotreated samples stored at NRL.
78	CL05-0246	BS	50% Jet-A 50% Clay Treated Jet-A
79	CL05-0247	BT	50% BF-2 50% Arctic Diesel #1
80	CL05-0248	BU	EPA Emissions Certification Fuel; Grade No. 2 Diesel w/ Good Lubricity (SwRI AL-27070)
81	CL05-0249	BV	10% S-8 90% BF-2 (B(4))
82	CL05-0250	BW	20% S-8 80% BF-2 (B(4))

Number	SwRI Sample ID	NRL or Pax Sample ID	Actual Sample Identification
83	CL05-0251	BX	30% S-8 70% BF-2 (B(4))
84	CL05-0252	BY	40% S-8 60% BF-2 (B(4))
85	CL05-0253	BZ	50% S-8 50% BF-2 (B(4))
86	CL05-0254	CA	60% S-8 40% BF-2 (B(4))
87	CL05-0255	CB	70% S-8 30% BF-2 (B(4))
88	CL05-0256	CC	80% S-8 20% BF-2 (B(4))
89	CL05-0257	CD	90% S-8 10% BF-2 (B(4))
90	CL05-0358	CE	15 ppm Infineum additive R-690 in S-8
91	CL05-0359	CF	100 ppm Infineum additive R-690 in S-8
92	CL05-0360	CG	200 ppm Infineum additive R-690 in S-8
93	CL05-0361	CH	500 ppm Infineum additive R-690 in S-8
94	CL05-0362	CI	15 ppm Infineum additive R-690 in Jet A
95	CL05-0363	CJ	100 ppm Infineum additive R-690 in Jet A
96	CL05-0364	CK	500 ppm Infineum additive R-690 in Jet A

Number	SwRI Sample ID	NRL or Pax Sample ID	Actual Sample Identification
97	CL05-0365	CL	15 ppm Infineum additive R-690 in Composite Sample
98	CL05-0366	CM	100 ppm Infineum additive R-690 in Composite sample
99	CL05-0367	CN	500 ppm Infineum additive R-690 in Composite sample
100	CL05-0389	CO	15 ppm Infineum additive R-690 in Arctic Diesel #1
101	CL05-0390	CP	100 ppm Infineum additive R-690 in Arctic Diesel #1
102	CL05-0391	CQ	500 ppm Infineum additive R-690 in Arctic Diesel #1
103	CL05-0392	CR	15 ppm Infineum additive R-650 in S-8
104	CL05-0393	CS	100 ppm Infineum additive R-650 in S-8
105	CL05-0394	CT	500 ppm Infineum additive R-650 in S-8
106	CL05-0395	CU	15 ppm Infineum additive R-650 in Jet-A
107	CL05-0396	CV	100 ppm Infineum additive R-650 in Jet-A
108	CL05-0397	CW	500 ppm Infineum additive R-650 in Jet-A
109	CL05-0398	CX	50% S-8 50% Jet-A
110	CL05-0399	CY	50% S-8 50% Clay treated Jet-A

Number	SwRI Sample ID	NRL or Pax Sample ID	Actual Sample Identification
111	CL05-0400	CZ	50% Clay Treated Jet-A 50% ULSD low lubricity Composite H-treated from NRL (Sample BR)
112	CL05-0488	DA	10% ULSD Composite fuel from SwRI hydrotreatment study (Sample BR) 90% Arctic Diesel #2
113	CL05-0489	DB	15 ppm additive DCI-4A in S-8
114	CL05-0490	DC	100 ppm additive DCI-4A in S-8
115	CL05-0491	DD	500 ppm additive DCI-4A in S-8
116	CL05-0492	DE	15 ppm additive DCI-4A in Jet-A
117	CL05-0493	DF	100 ppm additive DCI-4A in Jet-A
118	CL05-0494	DG	500 ppm additive DCI-4A in Jet-A
119	CL05-0495	DH	15 ppm Infineum additive R-690 in Clay Treated Jet-A
120	CL05-0496	DI	100 ppm Infineum additive R-690 in Clay Treated Jet-A
121	CL05-0497	DJ	500 ppm Infineum additive R-690 in Clay Treated Jet-A
122	CL05-0498	DK	15 ppm Infineum additive R-690 in Clay Treated BF-2
123	CL05-0499	DL	100 ppm Infineum additive R-690 in Clay Treated BF-2
124	CL05-0500	DM	500 ppm Infineum additive R-690 in Clay Treated BF-2

Number	SwRI Sample ID	NRL or Pax Sample ID	Actual Sample Identification
125	CL05-0501	DN	15 ppm Infineum additive R-650 in new Diesel Composite (Sample DA)
126	CL05-0502	DO	100 ppm Infineum additive R-650 in new Diesel Composite (Sample DA)
127	CL05-0503	DP	500 ppm Infineum additive R-650 in new Diesel Composite (Sample DA)
128	CL05-0504	DQ	15ppm Infineum additive R-650 in Arctic Diesel #1
129	CL05-0505	DR	100 ppm Infineum additive R-650 in Arctic Diesel #1
130	CL05-0506	DS	500 ppm Infineum additive R-650 in Arctic Diesel #1
131	CL05-0507	DT	15 ppm Infineum additive R-650 in Clay Treated Jet-A
132	CL05-0508	DU	100 ppm Infineum additive R-650 in Clay Treated Jet-A
133	CL05-0509	DV	500 ppm Infineum additive R-650 in Clay Treated Jet-A
134	CL05-0510	DW	15 ppm Infineum additive R-650 in Clay Treated BF-2
135	CL05-0511	DX	100 ppm Infineum additive R-650 in Clay Treated BF-2
136	CL05-0512	DY	500 ppm Infineum additive R-650 in Clay Treated BF-2
137	CL05-0529	DZ	15 ppm DCI-4A (18 mg of 80% active ingredient) in ULSD Composite fuel Sample DA
138	CL05-0518	EA	100 ppm DCI-4A (120 mg of 80% active ingredient) in ULSD Composite fuel Sample DA

Number	SwRI Sample ID	NRL or Pax Sample ID	Actual Sample Identification
139	CL05-0519	EB	500 ppm DCI-4A (600 mg of 80% active ingredient) in ULSD Composite fuel Sample DA
140	CL05-0520	EC	15 ppm DCI-4A (18 mg of 80% active ingredient) in Arctic Diesel #1
141	CL05-0521	ED	100 ppm DCI-4A (120 mg of 80% active ingredient) in Arctic Diesel #1
142	CL05-0522	EE	500 ppm DCI-4A (600 mg of 80% active ingredient) in Arctic Diesel #1
143	CL05-0523	EF	15 ppm DCI-4A (18 mg of 80% active ingredient) in Clay Treated Jet-A
144	CL05-0524	EG	100 ppm DCI-4A (120 mg of 80% active ingredient) in Clay Treated Jet-A
145	CL05-0525	EH	500 ppm DCI-4A (600 ppm of 80% active ingredient) in Clay Treated Jet-A
146	CL05-0526	EI	15 ppm DCI-4A (18 mg of 80% active ingredient) in Clay Treated BF-2
147	CL05-0527	EJ	100 ppm DCI-4A (120 mg of 80% active ingredient) in Clay Treated BF-2
148	CL05-0528	EK	500 ppm DCI-4A (600 mg of 80% active ingredient) in Clay Treated BF-2
149	*	EL	25 ppm Infineum additive R-690 in Arctic Diesel #1 (Sample A(4))
150	*	EM	75 ppm Infineum additive R-690 in Arctic Diesel #1 (Sample A(4))
151	*	EN	25 ppm Infineum additive R-690 in Arco HT3
152	*	EP	75 ppm Infineum additive R-690 in Arco HT3

Number	SwRI Sample ID	NRL or Pax Sample ID	Actual Sample Identification
153	*	EQ	80% Haifa HT4 (NRL #98-99) 20% Arco (NRL #99-3)
154	*	ER	88% Haifa HT4 (NRL #98-99) 12% Arco (NRL #99-3)
155	*	ES	93% Haifa HT4 (NRL #98-99) 7% Arco (NRL #99-3)
156	*	ET	97% Haifa HT4 (NRL #98-99) 3% Arco (NRL #99-3)
157	*	EU	100% Haifa HT4 (NRL #98-99)
158	CL05-0243	BP(2)	Separate aliquot of100% Jet-A (non-additized)(SwRI AL-27069)
159	Known as Fuel 3000 at SwRI for hardware testing	EV	New 5000g good lubricity fuel for hardware testing at SwRI
160	*	EW	ARCO feedstock at NRL (not HT)
161	*	A(5)	Composite of Arctic Diesel #1: mixture of remaining A, A(2), A(3) and A(4)
162	*	EX	50% BF-2 (B(4)) 50% Arctic Diesel #1 (A(5))
163	*	EY	55% BU (Good lubricity diesel No. 2) 45% Arctic Diesel #1 (A(5))
164	*	EZ	13% Arco (EW) 87% Arctic Diesel #1 (A(5))
165	*	FA	0.1% FSII in Jet A (BP(2))
166	*	FB	0.1% FSII, 100 ppm Infineum R-650 in Jet A (BP(2))

Number	SwRI Sample ID	NRL or Pax Sample ID	Actual Sample Identification
167	*	FC	100 ppm Infineum R-650 additive in Jet A (BP(2))
168	*	FD	0.1% MDFI in Arctic Diesel #1 (A(4))
169	*	FE	0.1% MDFI, 0.2% CI in Arctic Diesel #1 (A(4))
170	*	FF	0.1% MDFI, 0.2% CI, 25 ppm FOA-3 in Arctic Diesel #1 (A(4))
171	*	FG	100 ppm Infineum R-650 in Arctic Diesel #1 (A(4))
172	*	FH	100 ppm Infineum R-650, 0.1% MDFI in Arctic Diesel #1 (A(4))
173	*	FI	100 ppm Infineum R-650, 0.1% MDFI, 0.2% CI in Arctic Diesel #1 (A(4))
174	*	FJ	100 ppm Infineum R-650, 0.1% MDFI, 0.2% CI, 25 ppm FOA-3 in Arctic Diesel #1 (A(4))
175	*	FK	25 ppm FOA-3 in Arctic Diesel #1 (A(4))
176	*	FL	0.2% CI in Arctic Diesel #1 (A(4))
177	*	FM	100 ppm Infineum R-650, 25 ppm FOA-3, in Arctic Diesel #1 (A(4))
178	*	FN	100 ppm Infineum R-650, 0.2% CI, in Arctic Diesel #1 (A(4))
179	*	FO	50% Jet A (BP(2)) with 0.1% FSII 50% Arctic Diesel #1 (A(4)) with 0.1% MDFI, 0.2% CI, and 25 ppm FOA-3

Number	SwRI Sample ID	NRL or Pax Sample ID	Actual Sample Identification
180	*	FP	50% Jet A (BP(2)) with 0.1% FSII 50% Arctic Diesel #1 (A(4)) with 0.1% MDFI, 0.2% CI, and 25 ppm FOA-3 plus 100 ppm Infineum R-650
181	*	FQ	50% Jet A (BP(2)) 50% Arctic Diesel #1 (A(5))
182	*	FR	50% Jet A (BP(2)) 50% Arctic Diesel #1 (A(5)) plus 100 ppm Infineum R-650
183	*	FS	7.0 ppm Betz 8Q405 in Jet A (BP(2))
184	*	FT	374 ppm Betz 8Q405 in Jet A (BP(2))
185	*	FU	1.1 ppm SDA in Arctic Diesel #1 (A(4))
186	*	FV	15 ppm Infineum R-650 in Jet A (BP(2))
187	*	FW	25 ppm Infineum R-650 in Jet A (BP(2))
188	*	FW	50 ppm Infineum R-650 in Jet A (BP(2))
189	*	FY	75 ppm Infineum R-650 in Jet A (BP(2))
190	*	FZ	100 ppm Infineum R-650 in Jet A (BP(2))
191	*	GA	15 ppm Infineum R-650 in S-8 (BO)

Number	SwRI Sample ID	NRL or Pax Sample ID	Actual Sample Identification
192	*	GB	25 ppm Infineum R-650 in S-8 (BO)
193	*	GC	50 ppm Infineum R-650 in S-8 (BO)
194	*	GD	75 ppm Infineum R-650 in S-8 (BO)
195	*	GE	100 ppm Infineum R-650 in S-8 (BO)
196	*	GF	Duplicate of sample FH
197	*	GG	Duplicate of sample FV
198	*	GH	Duplicate of sample FX
199	*	GI	Duplicate of sample FY
200	*	GJ	Duplicate of sample FZ
201	*	GK	Duplicate of sample GB
202	*	GL	Duplicate of sample GD
203	*	GM	Duplicate of sample GE

* No SwRI Sample ID number assigned.

APPENDIX C

Specification Results for 9 petroleum diesel fuels used in the program.

Characteristic	F-76 Req.	Fuel B	Fuel BM	Fuel BN
Acid Number	0.3 mg KOH/g max	0.019	0.000	0.001
Appearance @ 25 C	C&B	C&B	C&B	C&B
Aromatics	Record %	16.7		16.4
Ash	0.005 wt% max	0.000	0.000	
Carbon Residue	0.20 wt% max	0.08	0.08	
Cetane Index	43 min	54		
Cloud Point	-1 C max	-4	-6.9	
Color	3 max	0.9	<0.5	
Corrosion @ 100 C	No. 1 max	1a	1a	1a
Demulsification	10 minutes max	2	2	
Density @15 C	876 kg/m^3 max	837	837	790
Distillation				
IBP		212	216	148
10%	Record	250	250	161
50%	Record	282	282	188
90%	357 C max	317	314	235
EP	385 C max	334	331	257
Res and Loss	3.0% vol max	1.6		1.5
Flash Point	60 C min	85	92.5	
Hydrogen Content	12.5 wt% min	13.9	13.7	14.08
Particulates	10 mg/L max	0.4	0.000	
Pour Point	-6 C max	-12	-12	
Storage Stability	3.0 mg/100 mL max	0.2	0.2	
Sulfur	1.0 wt% max	0.09	0.078	0.002
Calcium	1.0 ppm max	<0.1	<0.1	
Lead	0.5 ppm max	<0.1	<0.1	
Na + K	1.0 ppm max	<0.1	<0.1	
Vanadium	0.5 ppm max	<0.1	<0.1	
Viscosity @40 C	1.7-4.3 cSt	3.33	3.32	

Characteristic	F-76 Req.	Fuel BO	Fuel BP	Fuel BQ
Acid Number	0.3 mg KOH/g max	<0.015	0.01	0.006
Appearance @ 25 C	C&B			C&B
Aromatics	Record %	0.5	17	
Ash	0.005 wt% max		<0.01	0.000
Carbon Residue	0.20 wt% max		0.07	0.14
Cetane Index	43 min	64	41	48
Cloud Point	-1 C max			-18
Color	3 max			2
Corrosion @ 100 C	No. 1 max	1a	1a	1a
Demulsification	10 minutes max			5
Density @15 C	876 kg/m^3 max	751	789	842
Distillation				
IBP		159	133	185
10%	Record	171	154	214
50%	Record	201	181	259
90%	357 C max	248	232	306
EP	385 C max	272	251	338
Res and Loss	3.0% vol max	1.0	1.4	1.5
Flash Point	60 C min	46	38	69
Hydrogen Content	12.5 wt% min	15.2	14.7	13.17
Particulates	10 mg/L max			0.5
Pour Point	-6 C max			-27
Storage Stability	3.0 mg/100 mL max			0.9
Sulfur	1.0 wt% max	0.000	0.004	0.03
Calcium	1.0 ppm max			<0.1
Lead	0.5 ppm max			<0.1
Na + K	1.0 ppm max			0.1
Vanadium	0.5 ppm max			<0.1
Viscosity @40 C	1.7-4.3 cSt		1.09	2.308

Characteristic	**F-76 Req.**	**Fuel BR**	**Fuel BU**	**Fuel DA**
Acid Number	0.3 mg KOH/g max		0.02	0.006
Appearance @ 25 C	C&B	C&B	C&B	
Aromatics	Record %			
Ash	0.005 wt% max	0.000	<0.01	0.001
Carbon Residue	0.20 wt% max	0.05	0.16	
Cetane Index	43 min	53.9	48	
Cloud Point	-1 C max	-4.4	-18	-37.4
Color	3 max	<0.5	<2.0	<0.5
Corrosion @ 100 C	No. 1 max	1a	1a	1a
Demulsification	10 minutes max	2	5	1
Density @15 C	876 kg/m^3 max	836	842	801
Distillation				
IBP		205	171	
10%	Record	236	208	
50%	Record	278	257	
90%	357 C max	329	306	
EP	385 C max	355	334	
Res and Loss	3.0% vol max	1.9	0.7	
Flash Point	60 C min	85	69	52
Hydrogen Content	12.5 wt% min	13.75	13.04	13.99
Particulates	10 mg/L max	0.2		0.0
Pour Point	-6 C max	-12		-51
Storage Stability	3.0 mg/100 mL max	0.9		
Sulfur	1.0 wt% max	0.007	0.035	0.033
Calcium	1.0 ppm max	<0.1	<1	0.1
Lead	0.5 ppm max	0.1	<1	<0.1
Na + K	1.0 ppm max	<0.1	<1	0.6
Vanadium	0.5 ppm max	<0.1	<1	<0.1
Viscosity @40 C	1.7-4.3 cSt	3.244	2.36	