



# Naval Fuels & Lubricants

## Cross Functional Team

### *Research Report*

## **Compatibility of Direct Sugar to Hydrocarbon (DSH-76) with Combined Contaminated Fuel Detector**

NF&LCFT REPORT 441/13-004

31 May 2013

Prepared By:

<p><b>Ryan Turgeon</b> <b>Fuels Chemist</b> <b>AIR-4.4.5.1</b></p>
<p><b>Kevin Bowes</b> <b>Chemical Engineer</b> <b>AIR-4.4.5.1</b></p>

NAVAIR Public Release 2013-504

Distribution Statement A - Approved for public release; distribution is unlimited

*Report prepared and released by:*



**Naval Air Systems Command  
Naval Fuels & Lubricants CFT  
22229 Elmer Road  
Patuxent River MD 20670-1534**

**Reviewed and Approved by:**

<p><b>RICHARD A. KAMIN Fuels Team Lead AIR-4.4.5</b></p>
--

**Released by:**

<p><b>DOUGLAS F. MEARNES Fuels &amp; Lubricants Systems Engineer AIR-4.4.1</b></p>

## TABLE OF CONTENTS

	Page
LIST OF TABLES.....	iv
LIST OF FIGURES .....	iv
EXECUTIVE SUMMARY .....	v
LIST OF ACRONYMS/ABBREVIATIONS.....	vi
1.0 BACKGROUND.....	1
2.0 OBJECTIVE.....	1
3.0 APPROACH.....	2
4.0 DISCUSSION.....	3
5.0 CONCLUSIONS .....	6
6.0 RECOMMENDATIONS.....	6
7.0 REFERENCES .....	6

## LIST OF TABLES

<b>Table</b>	<b>Title</b>	<b>Page</b>
Table 1:	Results of CCFD and ASTM testing on fuels with 10 mg/L test dust added.....	3
Table 2:	Comparison of CCFD instrument response by fuel type.....	4

## LIST OF FIGURES

<b>Figure</b>	<b>Title</b>	<b>Page</b>
Figure 1:	Plot of instrument response to particulate contamination of F-76, DSH-76, 50/50 F76/DSH76 and 70/30 F-76/DSH76. ....	4
Figure 2:	Comparison of CCFD free water measurement to free water added in F-76, 50/50 F76/DSH76, 70/30 F76/DSH and neat DSH-76. Free water was added at 0, 10 and 20 ppm. ....	5

## **EXECUTIVE SUMMARY**

Testing of the Navy's Combined Contaminated Fuel Detector with alternative sourced fuels was conducted as a part of the ongoing effort to certify the use of renewable fuels in all Navy aircraft and ships. Current certification efforts are focused on F-76 produced from the direct sugar to hydrocarbon (DSH) production process. Petroleum sourced F-76, alternative fuel source DSH-76 and 70/30 & 50/50 blends of F76/DSH76 fuel were contaminated with various levels of synthetic dirt or free water and tested in accordance with current CCFD operating guidelines. The particulate levels for all four samples were found to be measured much lower than the known amount of contaminant added or the results obtained by the ASTM referee gravimetric method. This discrepancy is a known issue with F-76 and the CCFD. To mitigate this issue, a comparison instrument response was plotted for each of particulate contaminants in various fuel types. This analysis showed that CCFD measurements are the same for all four of the fuels tested. The free water measurements were similar for neat DSH-76 and DSH-76 and petroleum F-76 blends within repeatability of the method. Based on the results of this study, use of F-76 produced by the DSH process will not adversely affect the contaminant measurements made by the CCFD.

## **LIST OF ACRONYMS/ABBREVIATIONS**

Combined Contaminated Fuel Detector.....	CCFD
Direct Sugar to Hydrocarbon Diesel Fuel.....	DSH-76
Naval Air Systems Command.....	NAVAIR
Naval Sea Systems Command .....	NAVSEA
Naval Ships Technical Manual Chapter .....	NSTM

# Compatibility of Direct Sugar to Hydrocarbon (DSH-76) with Combined Contaminated Fuel Detector

## 1.0 BACKGROUND

The Combined Contaminated Fuel Detector (CCFD) is the current shipboard quality surveillance instrument used to analyze the condition of periodically sampled fuel to ensure aviation and ship diesel fuel meets minimum acceptable limits of particulate and free water contamination prior to issuance.

NAVSEA maximum requirements for F-76 fuel are 2.5 mg/L sediment and 40 ppm free water (for gas turbine powered ships). The free water contamination (droplets) may appear as fine droplets or slugs of water in the fuel systems and may contain some dissolved hydrocarbons, fuel additives, or other water soluble/miscible materials. The particulate matter found in field fuel systems varies in shape and is commonly found in the 5 to 40 micron size range. Common particulate matter includes silica, rust, metal shavings, fibrous materials, coatings materials, elastomeric materials, hydrocarbon/oxidation materials, and other solid matter.

In the ongoing efforts to support the Navy's goals of utilizing renewable energy sources, aviation and marine diesel fuels derived from renewable sources are currently being tested and certified for fleet wide use. These fuels are intended to be used as "drop in" replacements without modification to existing engines, fuel systems or equipment. As a part of the certification effort, this test protocol will evaluate the use of the CCFD with the renewable aviation and marine diesel fuels.

The Direct Sugar to Hydrocarbon (DSH) production pathway produces fuels made from direct fermentation of sugar into olefinic hydrocarbons. The olefinic hydrocarbons are hydroprocessed to produce an iso-paraffinic hydrocarbon. The DSH fuel tested was a 98% pure branched paraffin with a fifteen carbon chain called 2,6,10 trimethyldodecane or farnesane. This fuel was unique because it is composed of a single molecule unlike petroleum fuels or Hydroprocessed Renewable Diesel (HRD-76) which are composed of a broad range of different normal and iso paraffins.

## 2.0 OBJECTIVE

The objectives of this test program are to:

- (1) Conduct testing with petroleum sourced F-76, a neat DSH-76, and 50/50 & 70/30 v/v blends of petroleum F-76 and DSH-76 to evaluate the impact of the DSH-76 and DSH-76 and petroleum blends on the CCFD's effectiveness at determining particulate contamination and free water
- (2) Examine material compatibility of CCFD instrument with DSH-76 fuels.

## **3.0 APPROACH**

### **3.1 Particulate Contaminant**

Prior to contamination, fuels were filtered to remove any existing particulates. Synthetic particulate contaminants were introduced by mass of 85% ISO Ultra Fine Test Dust (Silica) and 15% of Rio 9998 Test Dust (FeO<sub>3</sub>) per volume of fuel. A stock solution containing 10 mg/L was prepared. The IKA ULTRA TURRAX T25 Janke and Kunkel Type T25 S1 Homogenizer was used to mix the fuel with the dirt. This stock solution was tested via ASTM D5254 to independently verify the contamination level. Individual fuels of various particulate concentrations were prepared by diluting the stock solution with particulate free fuel until the desired contamination level was achieved. CCFD was calibrated using Wratten filters according to procedures in ship's maintenance procedure: MIP 6653/002 (A6SL). Testing of contaminants in neat DSH-76, blended DSH-76, and petroleum sourced F-76 was conducted following the Naval Ships Technical Manual Chapter (NSTM) Chapter 541 procedures for the CCFD. Each fuel was vacuum filtered through two 0.65 µm pore sized nitrocellulose filters. The light transmittance of each filter was measured using the CCFD instrument and recorded. The difference between the two filters was subtracted and plotted versus the fuel's particulate contamination level. Each concentration of particulate for each fuel type was measured three times. Instrument response to each fuel was defined as the slope of the linear least squares regression of the differential filter reading versus particulate concentration. The standard deviation of the slope was determined by using the "LINEST" function in Microsoft Excel 2007. The instruments response for renewable sourced fuels was compared to the response with petroleum F-76 using a Student's t-test.

### **3.2 Free Water Contaminant**

Free water contaminated samples were prepared by saturating the base fuel for at least 24 hours. Moist air was bubbled through fuel to ensure saturation. Free water at the desired concentration was then added to the water saturated fuel. Free water contaminated samples were tested immediately after water was added to ensure constant temperature and thus constant water solubility in the fuel. Samples were tested following the procedures in NSTM Chapter 541 (Diesel). Briefly, each fuel sample was vacuum filtered through a free water detection pad. Exposure to free water was revealed as fluorescent areas of the pad when exposed to ultraviolet light. For each test fuel and free water contamination level, at least three replicates were tested and averaged. Due to the relatively subjective rating method of the water detection pad, visual and photographic side by side comparisons of petroleum sourced fuels and alternative fuels were conducted, in addition to standard visual rating method described in NSTM manual.

### **3.3 Material/Instrument Compatibility**

In addition to verifying the contamination measurement results of DSH-76 and DSH-76/Petroleum based F-76 versus petroleum sourced F-76, the tests examined the material compatibility of the renewable sourced fuels with the CCFD instrument. During measurements and immediately following measurements of renewable sourced samples, the instrument was be

visually inspected for signs of incompatibility, including but not limited to: gums, dissolved or softened filters, fittings, stains, leaks or reduction in pumping suction.

## 4.0 DISCUSSION

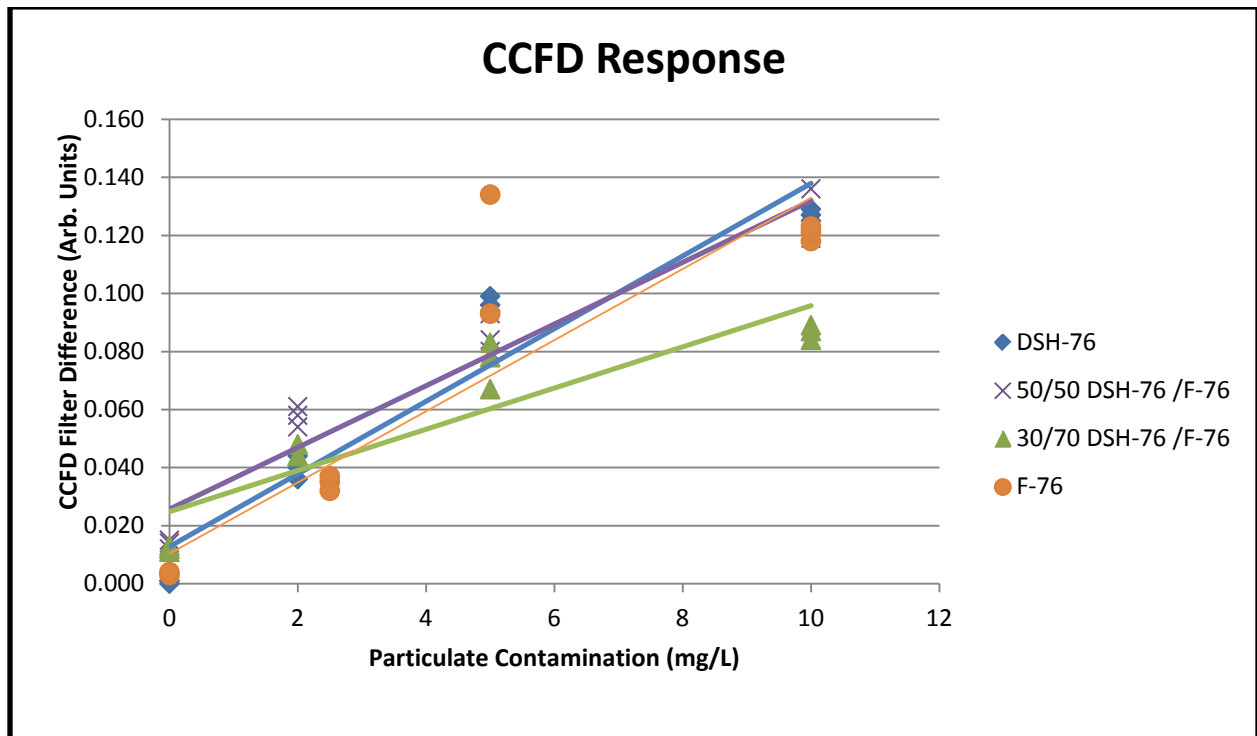
### 4.1 Particulate Contaminant

The initial test plan called for the concentration of particulate in the alternative fuel as determined by the CCFD to be compared with the concentration of particulate in the petroleum fuel. After calibration using Wratten Filter calibration standards, early testing of the CCFD showed a large discrepancy between the particulate concentration determined via CCFD and the known amount of particulate added to each fuel. Table 1 shows the results of fuels with 10 mg/L of 85% ISO Ultra Fine Test Dust (Silica) and 15% of Rio 9998 Test Dust (FeO<sub>3</sub>). In most cases the calibrated CCFD reading was lower than expected. The particulate level was verified using ASTM D5254. This was true for all four test fuels independent of their source. Further research revealed that this difference is a known issue with CCFD testing since it was originally developed for use with aviation turbine fuel JP-5 and was adapted for use with F-76.

**Table 1: Results of CCFD and ASTM testing on fuels with 10 mg/L test dirt added.**

Fuel Type	CCFD reading (mg/L) Instrument 1	CCFD reading (mg/L) Instrument 2	ASTM D 5254 (mg/L)
F-76	5.19	3.40	9.1
DSH-76	5.46	2.44	9.60
50/50 F76/DSH76	5.43	2.66	10.87
70/30 F76/DSH76	3.73	1.62	9.60

To account for the large variation between the CCFD results and the ASTM results, the test procedure was modified to compare the response of the CCFD to particulate contamination rather than numerical readings. This eliminated the systematic errors caused by the use of F-76 in the instrument. Figure 1 shows the instrument response plotted versus the particulate contaminant in each sample.



**Figure 1: Plot of instrument response to particulate contamination of F-76, DSH-76, 50/50 F76/DSH76 and 70/30 F-76/DSH76**

The plots show that instrument response to all fuel types is linear. This indicates that the CCFD will give a consistent reading with a known level of dirt. A statistical comparison of instrument response, see Table 2, shows that the instrument response for each petroleum fuel agrees with the instrument response of DSH-76/F-76 blends and neat DSH-76 within the accuracy of the test. Based upon these results, particulate concentration measurements by CCFD will be unaffected by the alternative fuels.

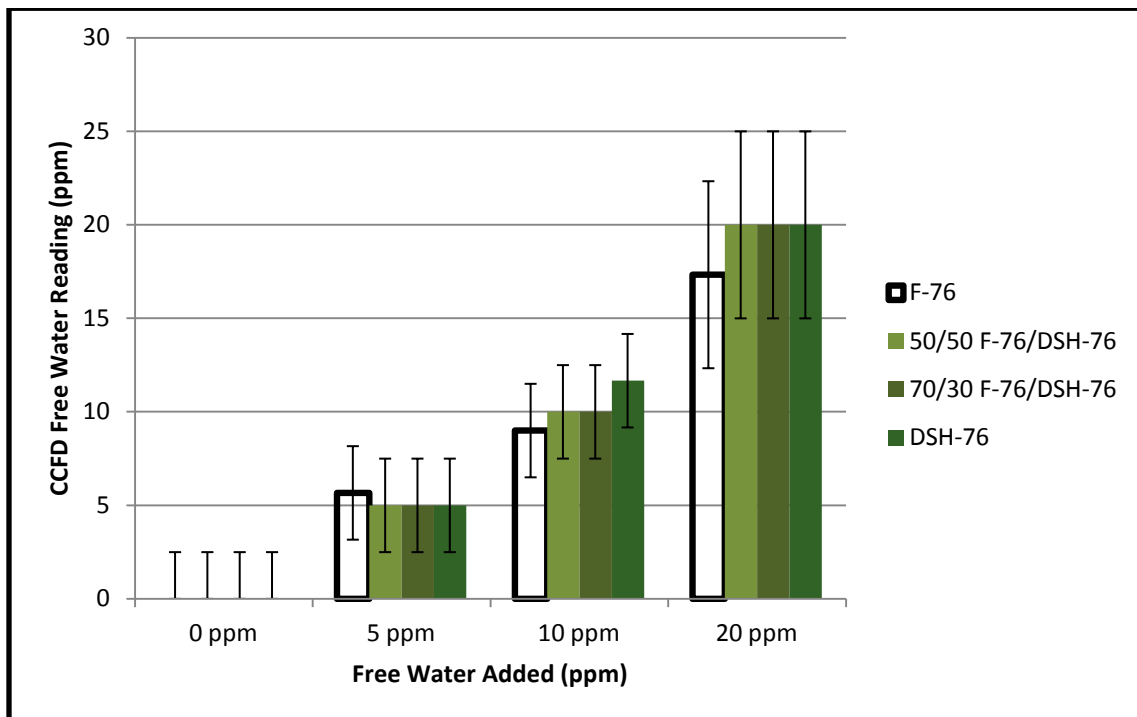
**Table 2: Comparison of CCFD instrument response by fuel type.**

Fuel Type	F-76	DSH-76	50/50 F76/DSH76	70/30 F76/DSH76
Instrument 1				
Response Factor	0.0123	0.0125	0.0106	0.0071
St Dev	0.0019	0.0011	0.0009	0.0010
Error (95% CI)	0.0042	0.0025	0.0019	0.0023
Agreement within Error	N/A	Yes	Yes	Yes
Instrument 2				
Response Factor	0.012	0.0084	0.0088	0.0055
St Dev	0.0011	0.0006	0.0005	0.0007
Error (95% CI)	0.0025	0.0013	0.0011	0.0016
Agreement within Error	N/A	Yes	Yes	No

## 4.2 Free Water Contaminant

Comparison between free water measurements was complicated due to the difficulty of preparing the samples and the subjective nature of the measurements. Preparation and handling of the samples was difficult due to the small volumes of free water needed to achieve the 0-20 ppm contamination levels. Free water loss due to dissolving or water trapped in the container after pouring the fuel into the CCFD resulted in lower than expected results for all fuel types. Measures were used to minimize the loss of free water including: saturating the fuel prior to the addition of free water, using “V” neck bottles to prevent low spots when poured, shaking the fuel/water mixture to suspend water prior to pouring into the CCFD and using new CCFD test pads. The free water contaminant pictures as displayed on the free water standard, did not match well to the actual free water test pads. This left the operator to use his/her best judgment to rate the test pad against the free water standard. To eliminate some of the variations caused by test pad reading, the same operator was used for all comparisons. Despite the difficulty in preparing free water samples and obtaining an accurate measurement, CCFD free water readings of DSH were reasonably accurate.

Figure 2 shows the comparison of F-76, DSH-76 blends and neat DSH-76. Each bar graph represented the average of 3 trials for each fuel type and free water level. Testing indicates the free water response in alternative fuels is as expected with petroleum fuels. Error bars represent the maximum uncertainty of each measurement, as the operator can only definitively report water levels to the nearest level printed on the free water standard.



**Figure 2: Comparison of CCFD free water measurement to free water added in F-76, 50/50 F76/DSH76, 70/30 F76/DSH76, and neat DSH-76. Free water was added at 0, 5, 10 and 20 ppm.**

### **4.3 Material/Instrument Compatibility**

The CCFD instrument was inspected during measurements and immediately following measurements of fuel samples. The instrument was visually inspected for signs of incompatibility, including but not limited to: gums, dissolved or softened filters, fittings, stains, leaks or reduction in pumping suction. No signs of incompatibility were found for DSH-76 and F76/DSH76 blends.

## **5.0 CONCLUSIONS**

The particulate levels were found to be measured much lower with the CCFD than the known amount of contaminant added or the ASTM referee method. This was true for the petroleum based F-76, the Neat DSH-76 and the blends of F-76 and DSH-76. Comparison of instrument response to particulate contaminants in various fuel types showed that CCFD measurements would be the same regardless of test fuel. Overall the free water measurements were the same for the Neat DSH-76 and the DSH-76 and F-76 blends within error of the method. Based on the results of this study, direct sugar to hydrocarbon process F-76 will not adversely affect the contaminant measurements made by the CCFD.

## **6.0 RECOMMENDATIONS**

It is recommended that DSH-76 continue qualification testing at 50/50 blends with F-76.

## **7.0 REFERENCES**

1. Naval Ships' Technical Manual Chapter 541Revision 5, August 2007
2. Laboratory Study of Mark I, Free water detector and Mark III Contaminated Fuel Detector. Reich, A.L. Report Ser #3109. 16 March 1977.

# REPORT DOCUMENTATION PAGE

Form Approved  
OMB No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing this collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. **PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.**

<b>1. REPORT DATE (DD-MM-YYYY)</b> 05-31-2013		<b>2. REPORT TYPE</b> Technical		<b>3. DATES COVERED (From - To)</b> 01-04-2012 to 05-23-2013	
<b>4. TITLE AND SUBTITLE</b> Compatibility of Direct Sugar to Hydrocarbon (DSH-76) with Combined Contaminated Fuel Detector				<b>5a. CONTRACT NUMBER</b> N/A	
				<b>5b. GRANT NUMBER</b> N/A	
				<b>5c. PROGRAM ELEMENT NUMBER</b> N/A	
<b>6. AUTHOR(S)</b> Turgeon, Ryan; Author Bowes, Kevin; Author Kamin, Richard ; Editor Mearns, Douglas ; Editor				<b>5d. PROJECT NUMBER</b> N/A	
				<b>5e. TASK NUMBER</b> N/A	
				<b>5f. WORK UNIT NUMBER</b> N/A	
<b>7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)</b> Naval Fuels & Lubricants Cross Functional Team 22229 Elmer Road Patuxent River, MD 20670				<b>8. PERFORMING ORGANIZATION REPORT NUMBER</b> NF&LCFT Report 441/13-004	
<b>9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)</b> Chief of Naval Operations N45 Rm 2E258 2000 Navy Pentagon Washington DC 20350-2000				<b>10. SPONSOR/MONITOR'S ACRONYM(S)</b> N/A	
				<b>11. SPONSOR/MONITOR'S REPORT NUMBER(S)</b> N/A	
<b>12. DISTRIBUTION / AVAILABILITY STATEMENT</b> A Approved for public release; distribution is unlimited.					
<b>13. SUPPLEMENTARY NOTES</b> N/A					
<b>14. ABSTRACT</b> Testing of the Navy's Combined Contaminated Fuel Detector with alternative sourced fuels was conducted as a part of the ongoing effort to certify the use of renewable fuels in all Navy aircraft and ships. Current certification efforts are focused on F-76 produced from the direct sugar to hydrocarbon (DSH) production process. Petroleum sourced F-76, alternative fuel source DSH-76 and 70/30 & 50/50 blends of F76/DSH76 fuel were contaminated with various levels of synthetic dirt or free water and tested in accordance with current CCFD operating guidelines. The particulate levels for all four samples were found to be measured much lower than the known amount of contaminant added or the results obtained by the ASTM referee gravimetric method. This discrepancy is a known issue with F-76 and the CCFD. To mitigate this issue, a comparison instrument response was plotted for each of particulate contaminants in various fuel types. This analysis showed that CCFD measurements are the same for all four of the fuels tested. The free water measurements were similar for neat DSH-76 and DSH-76 and petroleum F-76 blends within repeatability of the method. Based on the results of this study, use of F-76 produced by the DSH process will not adversely affect the contaminant measurements made by the CCFD. .					
<b>15. SUBJECT TERMS</b> Combined Contaminated Fuel Detector, CCFD, F-76, DSH-76					
<b>16. SECURITY CLASSIFICATION OF:</b>			<b>17. LIMITATION OF ABSTRACT</b> Unclassified Unlimited	<b>18. NUMBER OF PAGES</b> 13	<b>19a. NAME OF RESPONSIBLE PERSON</b> Douglas F. Mearns
<b>a. REPORT</b> UNCLASSIFIED	<b>b. ABSTRACT</b> UNCLASSIFIED	<b>c. THIS PAGE</b> UNCLASSIFIED			<b>19b. TELEPHONE NUMBER (include area code)</b> 301-757-3421