CRC Report No. 670

The Effect of Biodiesel Impurities on Wax Settling in Low Temperature Light-Duty Diesel Vehicles

CRC Project: DP-05-12

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COORDINATING RESEARCH COUNCIL, INC.
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Executive Summary of the Project

The objective of this study was to determine if settling of wax, biodiesel or biodiesel impurities impact light duty diesel vehicle low temperature operability (LTO) performance during extended periods of non-operation that coincides with low temperatures. No vehicle operation over a weekend is a good example.

A further objective of the study was to evaluate the correlation between common (LTO) bench test results; namely, cloud point (CP), cold filter plugging point (CFPP), low temperature flow test (LTFT) and actual light duty diesel (LDD) vehicle performance at low temperatures for petroleum diesel and biodiesel blends.

To that end, a petroleum diesel and four B100 materials were tested as B5 blends in three light duty diesel vehicles. Biodiesel fuels included those with both high and low saturated fatty acid methyl ester content, as well as high and low saturated monoglyceride (SMG) content. Blends contained 5 vol% biodiesel with No. 1 and No. 2 diesel blended to achieve target cloud points. Upper and lower fuel tank samples were taken immediately before the operability test cycle began. These samples were tested by several laboratories. Fuel filters were recovered immediately after the cycle and material collected from the filters was analyzed by one laboratory. The mineral diesel fuels components were characterized in detail by multiple laboratories. The test fuels were evaluated by LTO bench tests by multiple laboratories.

The scope of the test program was very limited. Each fuel was to be tested in each vehicle as a pass fail criteria at a single temperature. Vehicle operability would be measured after the vehicles were exposed to diurnal cooling cycle simulating a “weekend” cold soak. The results were compared to the predicted operability limit based on CP.

Vehicle failure was determined by a failure to start at a prescribed temperature or a significant rise in filter pressure over the time of the test cycle.

Conclusions:

No conclusions relative to the original project objective can be drawn because the tests were not conducted with good fidelity relative to the original test parameters. All of the diurnal temperature cycle targets for the fuel failed to meet the program targets. Start-Up test temperature failed to meet the program targets in more than 50% of the tests. More detail is found in Appendix E. In addition, data collection from the vehicle instrumentation was in many cases considered suspect and some data was not collected.

There was some useful information developed from the project. Laboratory analysis of upper and lower samples showed minimal wax settling or biodiesel component settling but no evidence of issues related to saturated monoglyceride (SMG) crystals persisting above CP.

CFPP D6371 data, for the majority of the fuels, was below or near the lowest fuel tank temperature. LTFT D4539 data ranged from slightly above to well below the lowest fuel tank temperature. The tests showed no evidence that the significantly higher D5773 CP relative to the D2500 (another test method) CP is predictive of operability.

Fuel filter pressure drop measurements may hint at potential issues with high SMG fuels that have large difference between D5773 and D2500 CP, but results were not consistent and conclusions cannot be drawn.

Failures occurred only for TF8 (which was a blend of No. 1 diesel with a low CP, high MG B100) after a constant temperature cooling cycle at the CP. Thus, the unique factors that caused failure are very low test temperature (-40°, i.e. a very low CP blend) and long constant temperature soak.

It is important that the reader understand that the full test program was not properly executed. Therefore the results should not be taken to imply that the settling of wax, biodiesel or biodiesel impurities will not impact vehicle operability during extended cold soaks.

No additional conclusions can be drawn relative to the applicability of the LTO bench test methods used in this project.

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BP
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Acronyms and Abbreviations

ASTM	ASTM International, a standards setting organization
AWCD	All-weather chassis dynamometer
Bxx	Biodiesel blend containing xx volume percent biodiesel
BP	BP Corporation
CFI	Cold Flow Improvers
CFPP	Cold filter plugging point
CP	Cloud point
CRC	Coordinating Research Council
CSFBT	Cold Soak Filter Blocking Tendency
CSFT	Cold soak filtration time
FAME	Fatty acid methyl ester
FMT	Final melting temperature
GM	General Motors Corporation
HD	Heavy-duty
kph	kilometer per hour
LD	Light-duty
LDD	Light-duty diesel
LTFT	Low-temperature flow test
LTO	Low temperature operability
SFPP	Simulated Filter Plugging Point
PPD	Pour Point Depressant
SMG	Saturated monoglyceride
USDOE	United States Department of Energy
VW	Volkswagen

1. Introduction

The objective of this study is to test vehicle operability for a range of biodiesel containing fuels over a "weekend" diurnal cooling cycle. This cooling might reveal paraffin wax, saturated FAME, or saturated monoglyceride (SMG) settling. It may also reveal insolubility of SMG that may not be observable on shorter timescales.[1,2] Phase 1 involved running a series of laboratory tests simulating weekend diurnal temperature conditions on stored fuel while acquiring visual and analytical evidence for wax settling in ULSD/Biodiesel/Jet No. 1 fuel blend combinations. In Phase 2 the study evaluated the predictive ability of bench test results for low-temperature performance (CP, CFPP, etc.) of B5 blends by comparing with actual light-duty diesel (LDD) vehicle performance on an all-weather chassis dynamometer. The study followed protocols similar to those used in the CRC Diesel Performance Group low-temperature performance study conducted previously,[3,4,5] and also similar to those described by Chandler.[6,7]

2. Background

Diesel fuels must operate over a wide range of climatic conditions and are therefore formulated to have a lower CP during winter months. Significantly lower CP fuels are required in northern states and colder areas. Tenth percentile minimum ambient air temperatures for all regions of the United States are shown in Appendix X5 to the ASTM D975 Standard Specification for Diesel Fuel Oils[8] and are used to estimate low-temperature operability requirements. Low-temperature operability is commonly ensured by using fuels with CP below the tenth percentile minimum ambient air temperature. For petroleum-derived diesel fuels flow improver additives can allow operability at temperatures as much as 10°C below CP.

Early published studies by Chandler examined low-temperature operability of both light-duty diesel (LDD) and heavy-duty diesel (HDD) vehicles. The goal of this work was to understand how well

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[1] Chupka, G.M., Yanowitz, J., Chiu, G., Alleman, T.A., McCormick, R.L. "Effect of Saturated Monoglyceride Polymorphism on Low-Temperature Performance of Biodiesel" *Energy Fuels* 25 (1) 398–405 (2011).
[2] Chupka, G.M., L., Fouts, L., McCormick, R.L. "Effect of Low Level Impurities on Low-Temperature Performance of Biodiesel" *Energy Environ. Sci.*, 5 8734-8742 (2012).
[3] CRC Report No. 649. Evaluation of Low Temperature Operability Performance of Light-Duty Diesel Vehicles for North America - Vehicle Test Report. CRC Project No. DP-2-04-1 and Evaluation of Low Temperature Operability for Light-Duty Diesel Vehicles for North America - Data Analysis Report. CRC Project No. DP-2-04-2. November 2007. www.crcao.org.
[4] CRC Report 650: Biodiesel Blend Low Temperature Performance Validation, CRC Project DP-20-07, June 2008. www.crcao.org.
[5] CRC Report 656: Biodiesel Blend Low-Temperature Performance Validation, CRC Project: DP-2a-07-2, February 2010. www.crcao.org.
[6] Chandler, J.E. "Comparison of All Weather Chassis Dynamometer Low-Temperature Operability Limits for Heavy and Light Duty Trucks with Standard Laboratory Test Methods" *SAE Technical. Paper No*. 962197 (1996).
[7] Chandler, J.E., Zechman, J.A. "Low-Temperature Operability Limits of Late Model Heavy-Duty Diesel Trucks and the Effect Operability Additives and Changes to the Fuel Delivery System Have on Low-Temperature Performance" *SAE Technical. Paper No*. 2000-01-2883 (2000).
[8] ASTM D975 Standard Specification for Diesel Fuel Oils. Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

laboratory bench test such as CP and LTFT predicted low-temperature vehicle performance.6 The study found that LTFT was developed to predict operability for the most severe engine fuel-system from a low-temperature operability standpoint (associated with the predominant engines in the HD market at that time). Chandler found that the LD vehicles in the 1990s were not as severe as the HD vehicles. Using either CP or LTFT as a predictor of low temperature operability protected all of the vehicles tested in that study; CP was overly conservative when additives were used. A subsequent study examined newer fuel system designs.7 Differences in filter location, porosity, and fuel circulation rate significantly affected low-temperature operability. LTFT continued to be a good predictor of the operability limit, particularly when additives were used.

The more recent study reported in CRC Report No. 649 examined both LDD and heavy-duty diesel (HDD) vehicles using petroleum derived fuels.3 The study found large differences in performance depending upon the vehicle and fuel. CP was the most conservative estimator of performance and protected all vehicles; however, considerable operability was observed below CP. LTFT was the next best test for protecting all vehicles. Biodiesel blend operability may be complicated by the observation that some biodiesel blendstock can form precipitates in blends at temperatures above CP. Biodiesel does not consist of 100% fatty acid methyl esters (FAME), but contains impurities such as mono- and di-glycerides (partly converted feedstock) as well as non-lipid materials that are mainly plant sterols, tocopherols, sterol glucosides, and waxy hydrocarbons that can become insoluble at low-temperatures. Furthermore, saturated monoglycerides (SMG) have been shown in bench tests to undergo a polymorphic phase transition, where SMG initially crystallized on cooling can convert to a different crystalline form with even lower solubility.[1,2,9] Two recent CRC studies have examined low-temperature operability for biodiesel blends, and had as their goal to validate that fuels blended from B100 having a cold soak filtration time below 200 seconds did not cause fuel filter clogging above CP.[4,5] The efficacy of the cold soak filtration test was demonstrated in these studies.

However recent laboratory bench studies (Phase 1 of this project) have shown that there can be a significant difference in LTO bench test results when diesel and biodiesel fuels are subjected to a diurnal cooling cycle similar to a weekend cold soak. It was this difference that inspired the Phase 2 vehicle testing program.

3. Phase 1 Study Methods and Results

The purpose of Phase 1 was to test at bench scale if wax settling could be observed to occur in several different diesel fuels. Fuels were held in tall graduated cylinders over a weekend diurnal cooling and warming cycle that ranged from 2°C above CP to 5°C below CP. Visual determination and standard tests were employed to see if significant differences in estimated vehicle performance would be predicted for the top and bottom samples from the cylinders as measured by CP, Pour Point, CFPP, SFPP and LTFT. Research at NREL has shown that measurement of a final melting temperature (FMT) can be useful in

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[9] Chupka, G.M., Fouts, L., Lennon, J.A., Alleman, T.L., Daniels, D.A., McCormick, R.L. “Saturated Monoglyceride Effects on Low-Temperature Performance of Biodiesel Blends” *Fuel Processing Technology* 118 302-309 (2014).

identification of polymorphic phase transformation of SMG.[1,2] A significant difference between the CP and FMT indicates that a polymorphic phase transformation of saturated monoglycerides may be occurring. FMT were measured with a Phase Technology 70X Analyzer (Phase 70X), the instrument used to measure CP by D5773. The Phase 70X utilizes diffusive light scattering to identify the onset of crystallization (CP per ASTM D5773) or the disappearance of solids (FMT). During the FMT test, the sample is cooled at several ^{o}C/min. until the instrument detects the formation of particles, which is seen as a rapid and large increase in the light scattering signal. The cooling continues to partial solidification, followed by controlled warming. The temperature at which all of the crystals dissolve or re-melt into solution (indicated by the signal returning to baseline where it will remain flat and constant) is recorded as the Final Melting Temperature (FMT) of the system. The FMT is very similar to ASTM D5972 *Standard Test Method for Freezing Point of Aviation Fuels (Automatic Phase Transition Method)*. The cooling and heating rate can be varied from 0.1 to 30^{o}C/min. For this study the cooling rate was as fast as possible and the heating rate was 1.5^{o}C/min. Additional information can found at; http://www.phase-technology.com/pdf/Study-of-Solid-Liquid-Phase-Equilibria-With-Phase-Technology-Analyzers.pdf

The FMT method has no established precision or bias and has not been correlated to vehicle failures.

Free and Total Glycerin of blend samples was measured by Ion Chromatography using a Metrohm 871 Advanced Bioscan and a Metrohm 819 IC Detector.

Fuels with and without conventional cold flow improver and wax anti-settling performance were included. Fuels were selected to cover the -10°C to -35°C operating range in case CP or wax content/type is a factor in wax settling issues. An example diurnal cooling cycle is shown in Figure 1 for a fuel having a CP of -10°C. A biodiesel having a nominally 0°C CP was tested at 0, 5, and 10 vol% in various blends of No. 2 diesel with other hydrocarbon diesel blendstocks (such as kerosene) and with different flow improver and wax anti-settling additives.

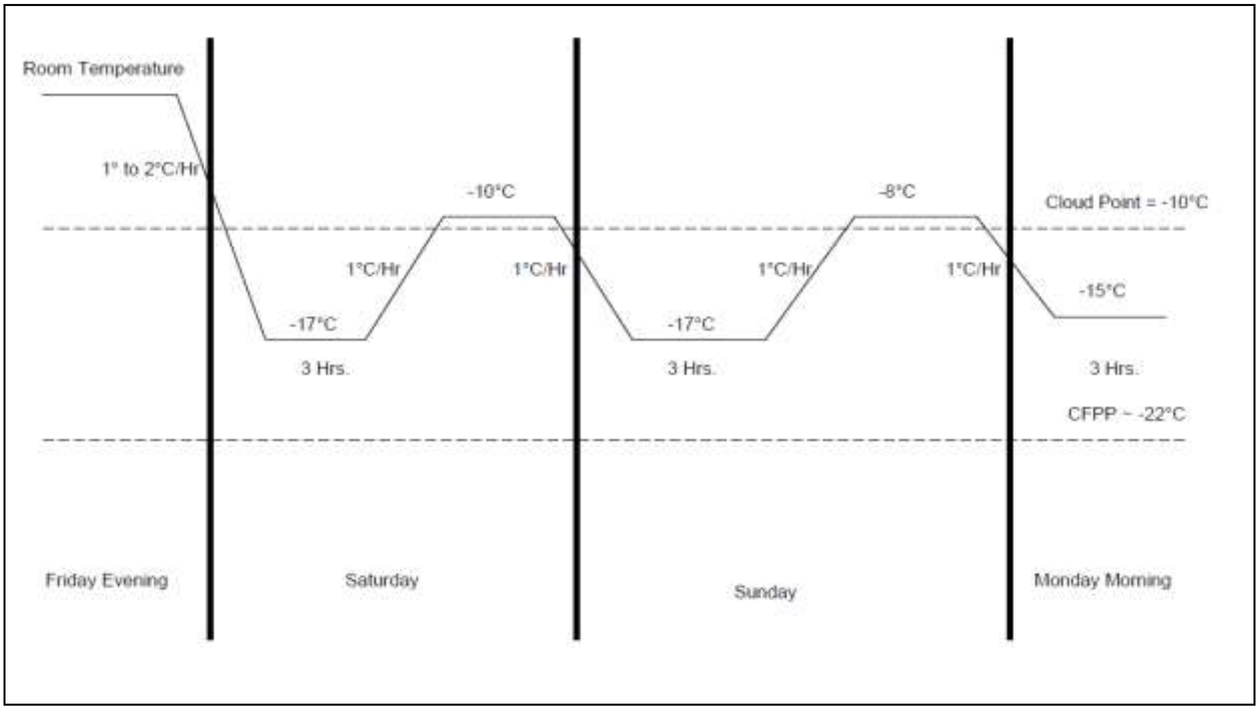


Figure 1. Example diurnal cooling cycle used in Phase 1.

Table 1 reports test results for the B100 used in Phase 1. Notably this sample fails the oxidation stability requirement (3 h minimum) of the ASTM D6751 standard but based on acid value does not appear to have oxidized extensively. The hydrocarbon portion of the blends was prepared using several hydrocarbon blendstocks: a 140 ppm sulfur No. 2 diesel fuel, a 100% paraffinic renewable jet fuel (camelina jet), kerosene, and a heavily hydrotreated blendstock. These were combined to achieve the desired CP. Appendix A presents additional data from the Phase 1 study, including a detailed property table for these hydrocarbon blend stocks.

Table 1 uses a modified version of ASTM D6584 to determine saturated monoglycerides in additional to total monoglycerides. This modified method has no established precision or bias and has not been correlated to vehicle failures. The modified method will be referred to as Modified D6584 from this point forward. Details of the modification are found after Table 2 below.

Table 1. Properties of biodiesel used in Phase 1.

Property	Test Method	Result
Oxidation stability	EN15751	2.13 h
Acid value	D664	0.32 mg KOH/g
Karl Fisher water	D6304	662 ppm
Free glycerin	Modified D6584	0.007 wt%
Total glycerin	Modified D6584	0.059 wt%
Saturated monoglycerides	Modified D6584	0.033 wt%
Cloud point	D5773	-0.2°C

To illustrate the impact of wax/impurity settling results are shown for a blend of 38% No. 2 Diesel/57% Kero/5% B100 having a CP of -20°C. Figure 2 shows photographs of graduated cylinders on the third day of diurnal cycling. While all fuels passed LTFT at the final temperature of -26.2°C, CP for the bottom sample of the untreated fuel is dramatically higher than for the top sample (-13.2°C versus -26°C), indicating that settling of components poorly soluble at cold temperatures has occurred. The use of both Additive A and Additive B significantly reduced this difference. Table 2 shows additional results for the top and bottom, unadditized samples. The results show significant accumulation of total glycerin (free glycerin plus mono, di, and triglycerides) in the bottom sample, along with a high FMT indicative of SMG polymorphic phase transformation. Details about the FMT are found in the next paragraph.

Table 2. Analysis of Phase 1 blend of 38% No. 2 Diesel/57% Kero/5% B100 CP of -20°C. FMT by NREL in-house method.

Analysis	**Top**	**Bottom**
Cloud point, °C	-24.4	-11.8
Final melting temperature, °C	-20.8	-4.8
Free glycerin, ppm	BD	1.62
Total glycerin, ppm	18.07	48.76

Table 2 indicates the standards that will be prepared per Sec 9.1. The modified method adds an additional stock solution using monostearin, monopalmitin, and monomyristin. This additional stock solution is added to the standard solutions (Table 3) using volumes of 4, 20, 50, 100, and 150 µL for solutions number 1 through 5, respectively.

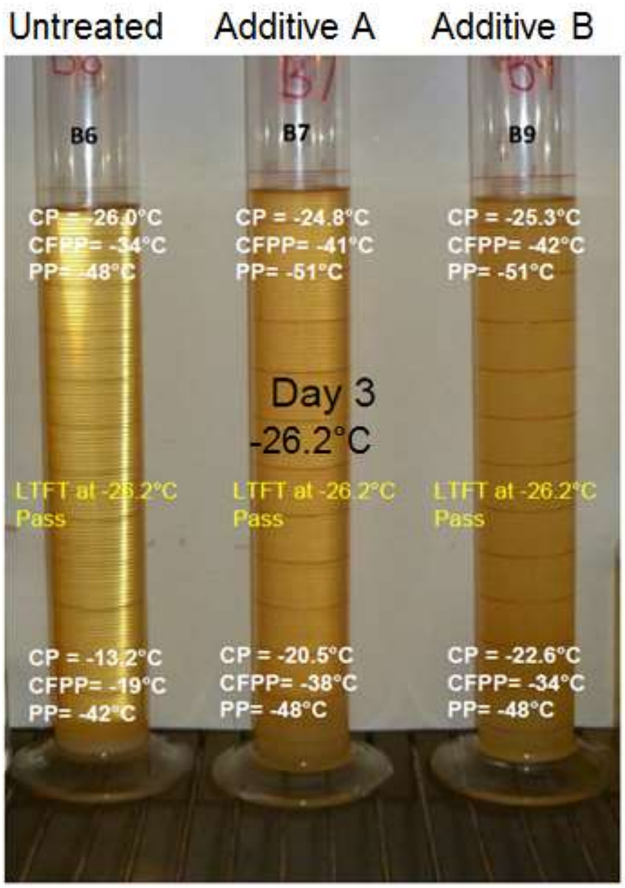


Figure 2. Example results for Phase 1 wax settling. Blend of 38% No. 2 Diesel/57% Kero/5% B100 CP of -20°C, photograph taken at -26.2°C.

4. Phase 2 Approach, Methods and Results

The testing program general concept is as follows:

- Testing of eight fuels in three LDD vehicles. The test eight fuels were blended at various ratios from a number of blend streams. The blend streams included a No. 2 S15 petroleum diesel fuel, a No. 1 S15 petroleum diesel fuel, and four B100s. The four B100s cover a range of CP and saturated monoglycerides.
- The mineral diesel portion of the B5 fuels was blended to give a similar cloud point among six of the B5s.
- Biodiesel was characterized for quality by multiple labs based on the ASTM D6751 standard. Base mineral diesel fuels were characterized using ASTM D975, CP, CFPP, PP and other low temperature operability parameters by multiple labs.
- The test fuel blends were characterized for CP, CFPP, PP, LTFT by multiple labs.
- The vehicles were cooled to test temperature (initial test temperature was the CP) using a diurnal cycle that was similar in time and temperatue to a weekend cold soak, started and driven on an all-

weather dyno. Temperature cycles of the all weather chassis dynamometer (AWCD) were predetermined by the CP of the fuel.

- The vehicle testing protocol was very close to that used in previous studies except for the cooling cycle[1,3].

Laboratory and Test Vehicles

The test program was conducted in an AWCD. The Mahle AWCD is located in Troy Michigan and consists of a climatic wind tunnel cell and a static soak room. The climatic wind tunnel can be conditioned and controlled to a temperature from 55^{o}C to -30^{o}C. Wind speeds of up to 200kph that can be matched to vehicle dynamometer speed. The dynamometer can accommodate front-wheel-drive, rear-wheel-drive and all-wheel-drive vehicles and can create a road-load simulation for loading of the drivetrain and engine. The static soak room can accommodate one vehicle at a time. Air temperature can be controlled from 50^{o}C to -40^{o}C. Both cells can be controlled to ramp temperature with respect to time and can be controlled independent of each other. Photographs of the vehicle test cell are shown in Figure 3.

The panel desired modern engine types that would be considered relatively new, representative of the market and have differences in fuel system design. Vehicles used in testing were loaned by individual companies. Basic information for the vehicles is shown in Table 3.

Figure 3. Vehicle Setup at Mahle

Table 3. Light duty diesel vehicles procured for the test program

Make	Model	Year	Engine Type
Jeep (Chrysler)	Grand Cherokee	2014	Eco Diesel – 3.0 Liter turbocharged V6
Chevrolet (GM)	Silverado 3500HD	2012	Duramax LML – 6.6 Liter turbocharged V8
Volkswagen (VW)	Beetle	2013	2.0 Liter turbocharged I-4

The vehicle fuel system consists of all components between the fuel tank and the fuel injectors including the lift pump, high pressure pump, fuel filters, pressure controls, and accumulators. The fuel filters used in each vehicle are described in Table 4. All three vehicles are based on common rail high pressure system configuration which is preferred systems in modern diesel vehicles. The high pressure fuel pump is a positive displacement pump to pressurize fuel and is generally driven by the engine to compress the fuel to the desired pressure. The pump supplies constant high pressure fuel to the rail accumulators. The injector on each of the engine cylinders meters the fuel as needed. Some of the system differences are in the pressure rating of the common rail systems, fuel flow demands based on engine operation, pump capacity, strategies for fuel return, and fuel heating. The Silverado (Figure 4), the excess fuel return path is available from the high pressure pump and from the injectors and fuel can be returned to the tank or the fuel filter based on the strategy for heating the fuel in cold weather conditions. The Jeep (Figure 5) has no additional fuel return line from the main fuel filter to the tank. For the Beetle (Figure 6) the configuration is significantly different with the fuel return lines from the injectors and high pressure pump routed to the main fuel filter, and a fuel return line from the main fuel filter to the tank.

Table 4. Fuel Filters to be used in Tests

	Micron	Inlet fuel heated by
Silverado 3500HD	4	Heated fuel return
Grand Cherokee	3	Electric Heating Element
Beetle	3	Electric Heating Element

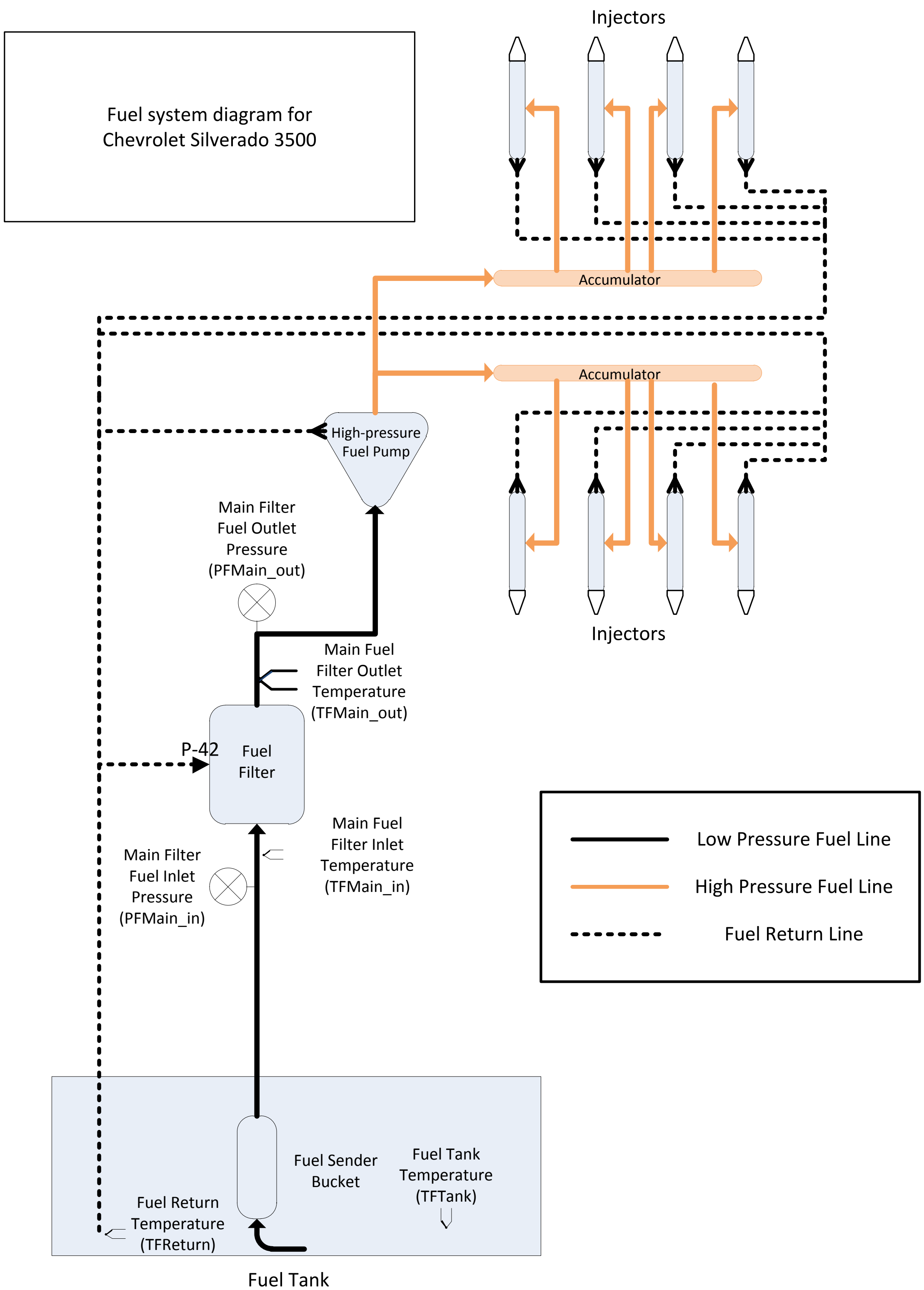


Figure 4. Fuel System for Silverado 3500HD

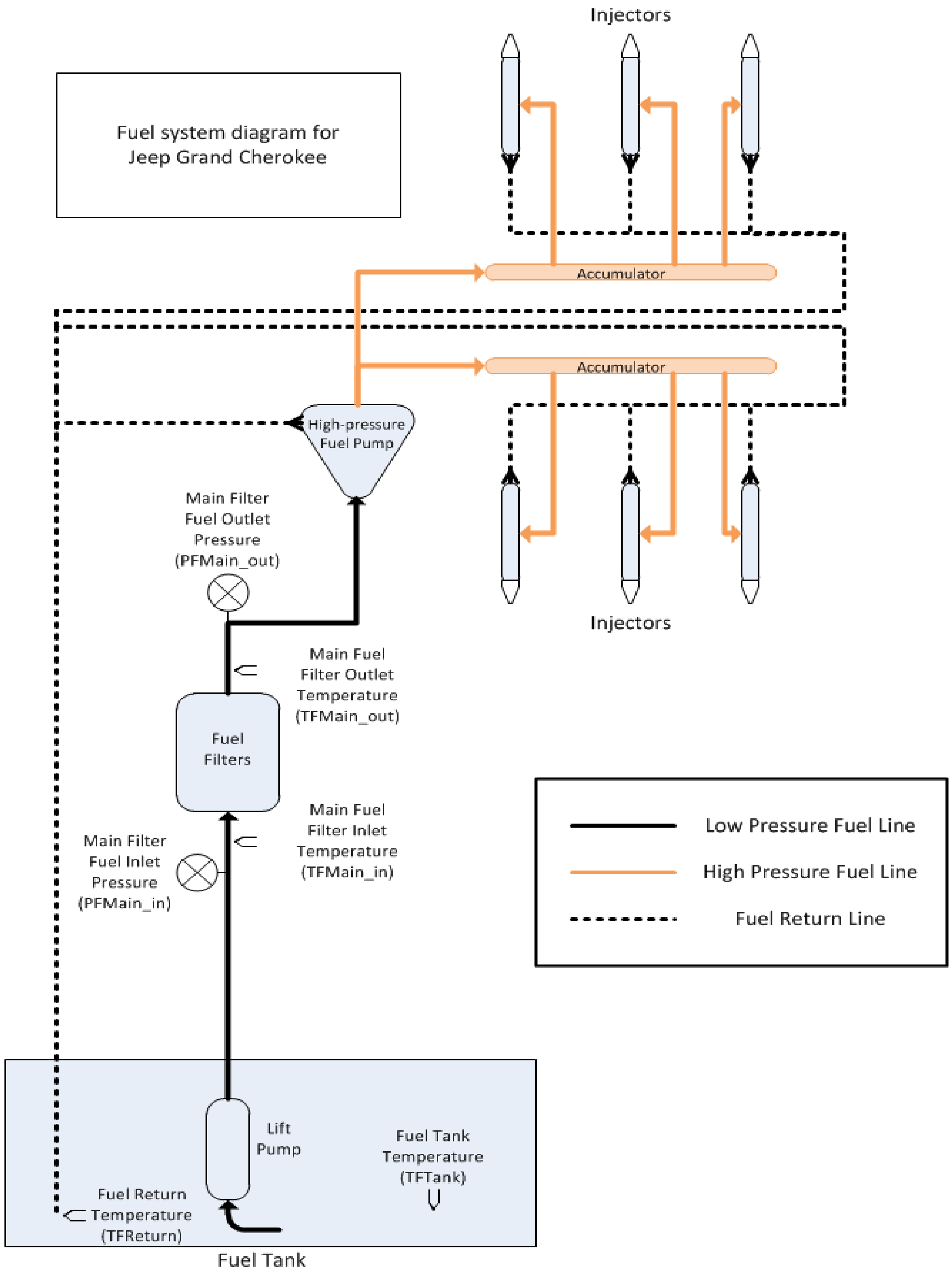


Figure 5. Fuel System for Grand Cherokee

Fuel system diagram for Volkswagen Beetle

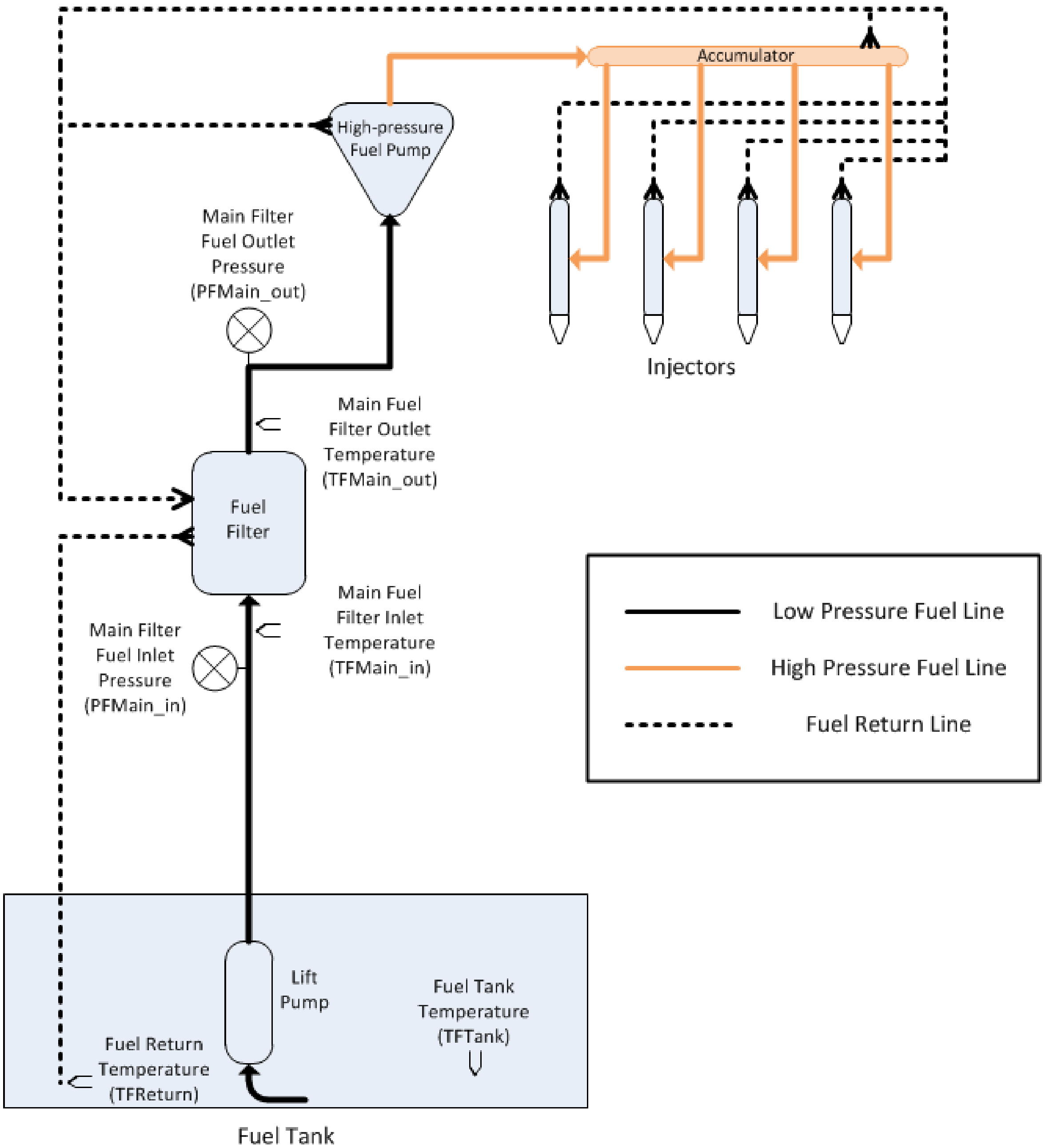


Figure 6. Fuel System For VW Beetle

From this point forward the vehicles are randomly coded as vehicle 1, vehicle 2 and vehicle 3.

Test Vehicle Preparation

Vehicles were prepared for testing at MAHLE. Vehicles were instrumented according to the list of channels shown in Table 5. Additionally the vehicle fuel tanks were fitted with two sample ports in the bottom of the fuel tank. One sample port was configured to draw fuel from the bottom of the tank and the other was equipped with an extension tube to allow it to draw fuel approximately one inch below the maximum fill level of the fuel tank (Figure 7). Vehicles also had the oil changed to accommodate the cold weather testing according to the OEMs recommendation. Campbell data acquisition systems were used in the test program.

Table 5: Instrumentation list

Description	Campbell Name	Unit	Sensor Type	Comment
Tunnel temp	T_tunnel	˚C		Tunnel output
Fuel tank temp	TFTank	˚C	TC (K-Type)	bottom
Main filter Fuel inlet Temperature	TFMain_in	˚C	TC (K-Type)	Inlet to fuel filter housing
Main filter Fuel outlet Temperature	TFMain_out	˚C	TC (K-Type)	Outlet of fuel filter house
Fuel return temp	TFReturn	˚C	TC (K-Type)	Return port in fuel sending unit on tank
Engine oil temp	T_oil	˚C	TC (K-Type)	Dipstick tip
Coolant temp	T_coolant	˚C	TC (K-Type)	Radiator inlet, Under hose fitting
Ambient Air temp	T air	˚C	TC (K-Type)	Tip of vehicle antenna
Main filter Fuel inlet Pressure	PFMain_in	kPa	Prs. TransD	Inlet to fuel filter housing
Main filter Fuel outlet Pressure	PFMain_out	kPa	Prs. TransD	Outlet of fuel filter house
Calculated filter pressure drop	PfMain_dp	kPA		Calculated difference
Battery voltage	Volt_bat	V		Measured across battery terminals
Engine RPM	E_RPM	RPM	Banner	Optical pick up on cramp pulley, 4 pulse per revolution
Vehicle speed	Speed_Veh	KPH		Tunnel output
Wind speed	Speed_wind	KPH		Tunnel output

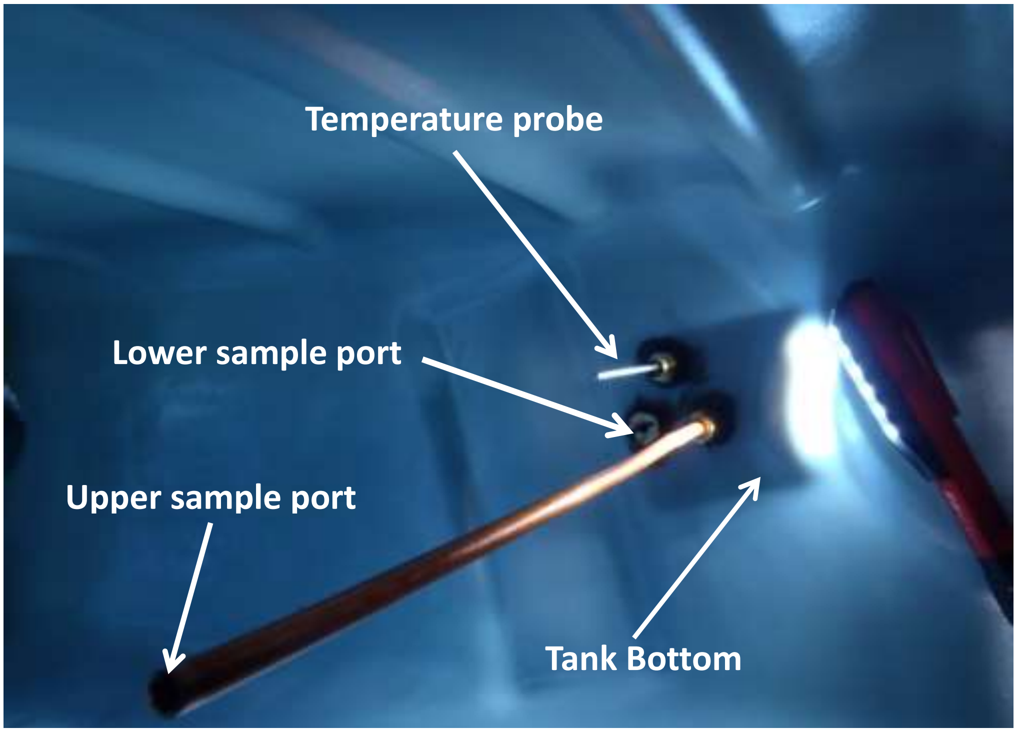


Figure 7: Example of In-Tank Fittings

Test Fuels

Two petroleum diesel fuels were procured a No. 1 S15 diesel and a No. 2 S15 diesel. Four biodiesel (B100) samples were procured and were shipped to IOSP for blending. They were supplied by four different biofuel manufacturers and designated B100 A, B, C, and D. The proposed operability targets of the fuel blends used in this study are listed in Table 6. Detailed characterization results for the actual fuels and blending components are presented in the Results section.

Table 6: Proposed test fuel matrix

Fuel	No. 2	No. 1	Vol% Bio	Blend CP, °C	Additive	Operability Target, °C	Purpose
TF 1	Yes	Yes	0	-28 (~-18°F)	no	CP	Control
TF 2	Yes	Yes	5*	<-20 (<-4°F)	no	CP	Low CP B100/ high MG
TF 3	Yes	Yes	5*	<-20 (<-4°F)	yes	10°C below CP	Low CP B100/ high MG+additive
TF 4	Yes	Yes	5‡	<-20 (<-4°F)	no	CP	Low CP B100/ low MG
TF 5	Yes	Yes	5‡	<-20 (<-4°F)	yes	10°C below CP	Low CP B100/ low MG+additive
TF 6	Yes	Yes	5*	<-20 (<-4°F)	no	CP	High CP B100 with high MG
TF 7	Yes	Yes	5‡	<-20 (<-4°F)	no	CP	High CP B100/ low MG
TF 8	No	Yes	5*	-40 (-40°F)	no	CP	Low CP B100/ high MG

*B100 total monoglyceride level >0.4 wt% (target 0.5 to 0.6 wt%). ‡B100 total monoglyceride level<0.4 wt% (target 0.3 wt%).

Test Procedures

Below is a detailed explanation or the preparation of the vehicle for each test fuel and the procedure used for the testing of each fuel.

Vehicle Preparation for Testing of Each Fuel:

1. Drain current fuel.
2. Use lower sample port in bottom of fuel tank to drain contents of tank into waste drum. Vehicle to be elevated on vehicle lift to facilitate draining.
3. Change filter and flush.
4. Change fuel filter using manufacture's procedure. (this filter will be for flushing)
5. Filter from pervious fuel testing will be placed in metal can. Label can with vehicle and fuel used in test.
6. Filter from initial setup will not be saved.
7. Add approximately 5 gallons of the next fuel to be tested into the fuel tank.
8. Run vehicle for 10 minutes.
9. Drain and discard fuel via lower tank sample port.
10. Add approximately 5 gallons of the next fuel to be tested into the fuel tank.
11. Run vehicle for 10 minutes.
12. Drain and discard fuel via lower tank sample port.
13. Prepare vehicle for testing.
14. Change fuel filter using manufactures procedure.
15. Discard flush filter.
16. Fill vehicle fuel tank fully with next fuel to be tested.
17. Start vehicle and run for 10 minutes.
18. Record in test log any irregularities as well as time, date and fuel number added to vehicle.

Testing Procedure for Each Fuel:

1. Cold soak the vehicle using a diurnal cycle for approximately 60 hours and an example cycle is shown in Figure 8. In general, the cold soak cycle for fuels the fuels was to be as follows:
    a. Cool chamber to 5°C above cloud point at 30°C/hour.
    b. Maintain temperature in the AWCD either for two hours or until the temperature of the fuel was 5±1°C above the cloud point of the fuel.
    c. Cool AWCD to 10°C below the cloud point of the fuel at a rate of 2°C/hour.
    d. Maintain temperature in the chamber at 10°C below the cloud point of the fuel for 6 hours.
    e. Raise the temperature of the chamber to the cloud point of the fuel at a rate of 2.0°C/hour.
    f. Maintain temperature in the chamber at the cloud point of the fuel for 3 hours.
    g. Cool chamber to 10°C below the cloud point of the fuel at a rate of 2°C/hour.
    h. Maintain temperature in the chamber at 10°C below the cloud point of the fuel for 6 hours.
    i. Raise the temperature of the chamber to 2°C above the cloud point of the fuel at a rate of 2.0°C/hour.

j. Maintain temperature in the chamber at 2°C above the cloud point of the fuel for 3 hours.
k. Cool chamber to the cloud point of the fuel at a rate of 2°C/hour.
l. Maintain temperature in the chamber at the cloud point of the fuel for 12.5 hours.
m. Due to limitations of the climate chamber, the cold soak cycle for fuel 8 was as follows:
    i. Cool chamber to the cloud point at a rate of 30°C/hour.
    ii. Maintain temperature in the chamber at the cloud point of the fuel for 55.5 hours.

2. Vehicles were soaked with batteries. An external power supply was used to assist in starting of the vehicles.
3. Vehicles were soaked in either the static soak room or the AWCD tunnel.
4. Original testing was conducted with low wind speed of approximately seven miles per hour around vehicles. Fans were used to increase air flow under the vehicles for the purpose of increasing the cooling rate of the fuel tank beginning on 5/12/14 and continuing through the end of the testing.
5. Install Vehicle in tunnel.
6. Roll/push vehicle into position on Dynos for testing.
7. Install power supply to vehicle for starting assistance.
8. Ensure that data collection of 1hz has been started.
9. Start vehicle according to following:
    a. Use the starting procedure recommended by the vehicle or engine constructor. Use all recommended starting aids. E.g. block heaters at very low temperatures, glow plugs, ether sprays, etc.
    b. If a recommendation does not exist, the following procedure is suggested:
    c. Place the gear-lever in neutral. (In position "N" for vehicles equipped with automatic transmission).
    d. Operate the starting aid(s).
    e. Declutch if the transmission is manual.
    f. Start a timing device and turn the key to operate the starter motor in sequences of 30 seconds cranking, unless otherwise stated by the manufacturer. If possible record cranking speed as this may explain a poor result if too low.
    g. In case of non-start, release the key, stop the stopwatch and wait 1minute between each sequence. (If vehicle manufacturer advises longer than 30 seconds cranking the waiting period between successive attempts should be extended; e.g. 1 minute cranking, 2 minutes wait, etc).
    h. Repeat the above action 1. to 3. until a start has been achieved with a maximum of 3 attempts.
10. Keep the starter motor operating after the initial firings until the engine can auto-rotate. Release the accelerator pedal when the engine speed reaches a suitable level for that vehicle. For vehicles equipped with automatic fast idle, release the accelerator completely and note idle speed. For other vehicles, regulate the idle speed as appropriate for that vehicle. If the engine stalls during the first ten seconds of idling speed in auto-rotation, restart the engine, as from section 2. If engine again stalls, terminate the cold start test. Report the result as "Stall at start".

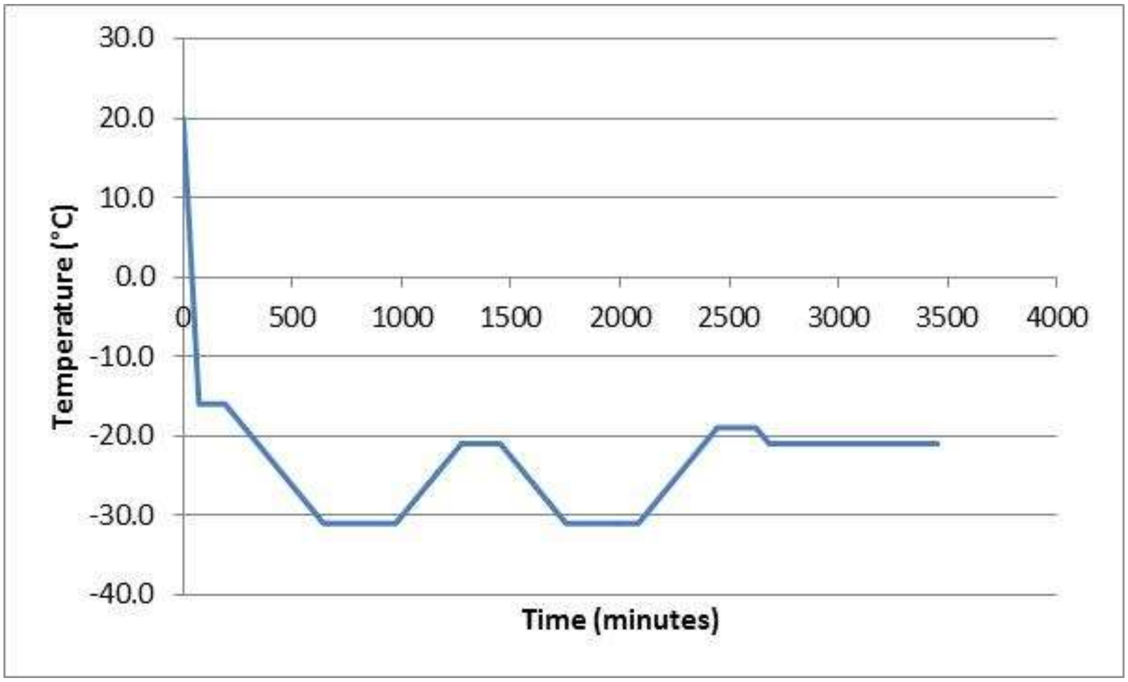


Figure 8: Example of AWCD Cooling Cycle for Fuel with -21°C Cloud Point

Vehicle drive plan

1. Switch on all electric accessories at their maximum power (head lights, lights, de-icing, vent, …), and keep, or leave, the engine at fast idling for 1min and 30 seconds after cranking.
2. Start the timer for the beginning of the operability test, and accelerate through the gears so as to reach 60 km/h (37.5 mph) in the appropriate gear within approximately 35 seconds. For each vehicle determine a suitable engine speed for gear change. Thereafter always change at this engine speed in order to ensure consistency from test to test. For vehicles with automatic transmission, allow the transmission, to dictate the shifts, with gear selector in "D". If a stall occurs, restart the engine immediately and perform the acceleration again. Note the occurrence of the stall and record the length of time when engine was not operating.
3. Drive at 60 km/h in the appropriate gear.
4. At 3 min 35 sec, accelerate at full load up to 110 km/h (68.75 mph) within approximately 25 sec.
5. Drive at 110 km/h in the highest gear and maintain, if possible, for 30 min. If vehicle has automatic trasmission allow controler to select gear.
6. The total test time is 34 min. including the acceleration phase and a graphic of this drive pattern is shown in Figure 9.

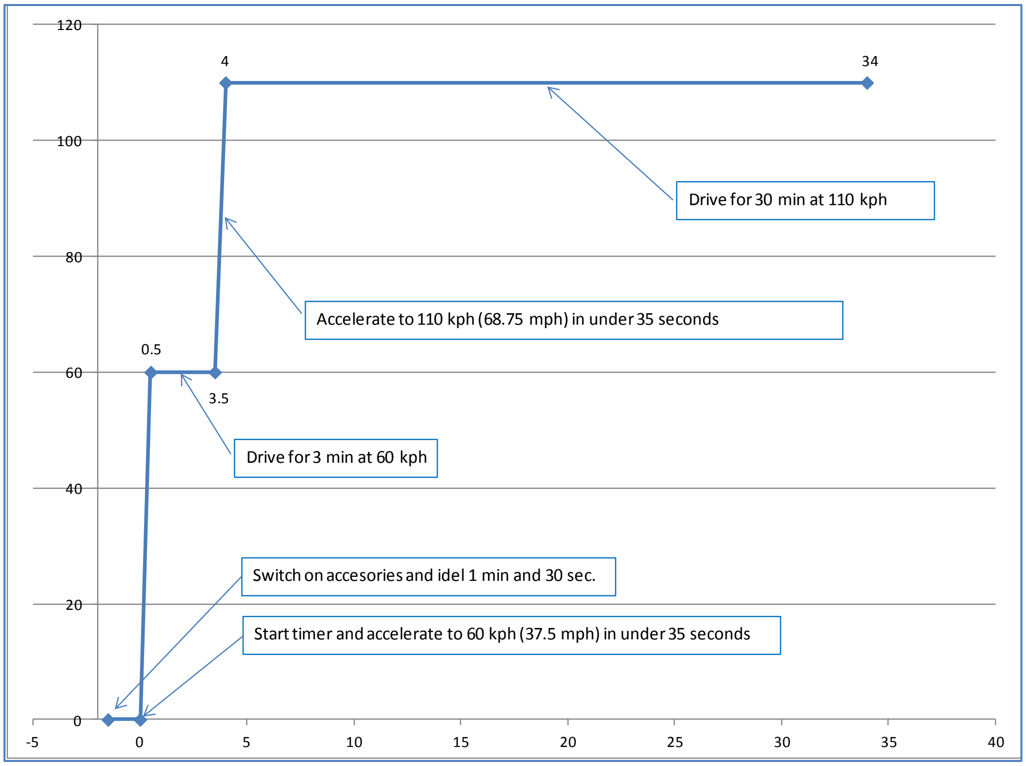


Figure 9: Graphic of the vehicle drive pattern

5. Fuel and Blend Component Characterization Results

Base Diesel Characterization

Two petroleum diesel fuels were procured representing ASTM D975 No. 1 S15 diesel and a No. 2 S15 diesel. Fuel properties are found in Table 7 and Table 8.

The fuels met all ASTM D975 specification limits except viscosity and cetane number for the No. 1 diesel fuel. Average viscosity as measured by three independent laboratories was 1.19 cSt @ 40°C, which is slightly less than the D975 limit of 1.3 cSt. Average cetane number (CN) was 34.9, which is less than the D975 limit of 40. In addition, the lubricity of the No. 1 diesel may be questionable since one lab test result showed it met specification, but the other did not meet specification. One lab measured HFRR lubricity of 414 microns wear scar diameter, while the other lab measured 780 microns, compared to the ASTM D975 maximum limit of 520 microns.

The No. 1 diesel fuel used in these tests was procured with the understanding that it must have very low cloud point and will be used as a diesel fuel blend stock to improve cold flow properties of No. 2 diesel. Therefore, the No. 1 diesel manufactured for cold flow blending purposes has properties more like jet fuel in order to make it more effective at improving cold flow upon blending with No. 2 diesel.

It is expected that all test fuels blended from No. 1 and No. 2 diesel, as shown in Table 11, meet all D975 specifications, including viscosity and cetane number.

Notice that there is a significant difference between cetane number (CN) measured by ASTM D613 engine method and derived cetane number (DCN) measured by ASTM D6890 Ignition Quality Tester (IQT) method. This difference is about 11 numbers for the No. 1 diesel (34.9 CN vs. 45.9 DCN) and about 5 numbers (54.8 CN vs. 49.8 DCN) for the No. 2 diesel fuel. The reason for the poor correlation between CN and DCN for these fuels is unknown.

Table 7. Properties of No.1 diesel fuel used for fuel blending.

Properties No. 1 Diesel	Lab B	Lab D	Lab F	Method
API Gravity, 60°F	46.25	45.38	46.4	D4052
Density, 15°C	0.7953	0.7992	0.7947	
Viscosity @ 40°C, cSt	1.178	1.223	1.162	D445
Sulfur, ppmw (XRF)			<5	D2622
Sulfur, ppmw	0	<0.3		D7039
PMCC Flash Point, °C	53	50 (122oF)	54	D93
Cloud Point, °C		-62.9	-62.3	D5773
Cloud Point, °C	-63.3			D7689
CFPP, °C	-64	-	-	D6371
LTFT, °C	-	-	-	D4539

Table continued on next page

Properties No. 1 Diesel Continued	Lab B	Lab D	Lab F	Method
Calculated Cetane Index, Proc. A	45.3	42.7	44.3	D4737
Derived Cetane Number by IQT			45.9	D6890
Cetane number	35.7	34.16		D613
Distillation, °C IBP	150.1	162.2	152.7	D86
5%	171.2	173.9	173.3	
10%	175.8	176.3	174.4	
20%	180.5	179.8	178.7	
30%	184.2	182.9	182.3	
40%	188.8	186.6	185.8	
50%	193.5	190.7	189.8	
60%	197.1	195.3	194.1	
70%	201.1	200.6	199.3	
80%	206.5	206.7	205.7	
90%	214.3	214.5	214.1	
95%	223.5	220.3	220.5	
EPT	235.1	229.5	231.1	
Recovery (%)	98.7	98.8		
Residue (%)	1.1	1.1		
Loss (%)	0.2	0.1		
IBP	115.0	46.1		D2887 % Off
5%	150.1	65.6		
10%	159.5	70.8		
20%	169.9	76.6		
30%	177.9	81.1		
40%	186.6	85.9		
50%	195.9	91.1		
60%	201.1	93.9		
70%	209.9	98.8		
80%	218.5	103.6		
90%	229.4	109.7		
95%	236.9	113.8		
EPT	256.9	124.9		
Lubricity, HFRR,@ 60°C, WSD, micron	414		780	D6079
Conductivity, pS/m	451 @ 22.0°C		-	D2624
Ash % mass, max	0.002		0.003	D482

Table continued on next page

Properties No. 1 Diesel Continued	Lab B	Lab D	Lab F	Method
Carbon residue, Ramsbottom, % mass, max	0.05		0.06	D524
ASTM D130 Copper Strip Corrosion, 3 hrs at 50°C	1b		1b	
Water and Sediment, % vol, max	0.00	0.00		D2709
Aromaticity %Vol, max: aromatics (vol%)	15.9	15.4		D1319
olefins (vol%)	0.9	1.1		
saturates (vol%)	83.2	83.5		
SFC Aromatics, wt %			18.1	
SFC PNA'S, wt %			<0.5	

Table 8. Properties of No. 2 diesel fuel used for fuel blending.

Properties No. 2 Diesel	Lab B	Lab D	Lab F	Method
API Gravity, 60°F	34.14	33.89	34.0	D4052
Density, 15°C	0.8542	0.8547	0.8540	
Viscosity @ 40°C, cSt	3.429	3.409	3.130	D445
Sulfur, ppmw (XRF)			<5	D2622
Sulfur, ppmw	4.7	4		D7039
PMCC Flash Point, °C	71	71.1 (160oF)	72	D93
Cloud Point, °C		-14.3	-13.9	D5773
Cloud Point, °C	-15.1	-14.3	-13.9	D7689
CFPP, °C	-16	-15	-13.9	D6371
LTFT, °C	-13 Pass -15 Fail	-13 Pass -15 Fail	-14 Pass -15, -16:Fail	D4539
Pour Point, °C		-20.6	-21	D5949
Pour Point, °C	-24			D7346
SFC Aromatics, wt %			29.2	
SFC PNA'S, wt %			3.9	
Calculated Cetane Index, Proc. A	46.8	46.4	45.9	D4737
Derived Cetane Number by IQT			49.8	D6890
Cetane number	54	55.69		D613

Table continued on next page

Properties No. 2 Diesel	Lab B	Lab D	Lab F	Method
Distillation, °C IBP	182.5	176.7	163.9	D86
5%	212.5	213.2	205.4	
10%	224.6	224.2	219.8	
20%	240.4	239.8	235.3	
30%	251.5	248.9	246.8	
40%	260.7	259.7	257.4	
50%	270.8	270.1	268.1	
60%	282.8	281.0	279.3	
70%	295.9	293.8	291.9	
80%	310.1	308.1	306.8	
90%	329.8	327.2	326.2	
95%	344.2	341.6	341.1	
EPT	354.7	352.0	350.6	
Recovery (%)	98.0	98.2		
Residue (%)	1.4	1.4		
Loss (%)	0.6	0.4		
IBP	110.4	116.6		D2887 % Off
5%	185.6	184.7		
10%	204.9	204.1		
20%	229.4	228.1		
30%	246.6	244.9		
40%	260.8	259.4		
50%	275.1	273.9		
60%	291	289.9		
70%	306.9	305.7		
80%	325.4	324.2		
90%	349.6	348.5		
95%	367.3	366.2		
EPT	403.3	402.9		
Lubricity, HFRR,@ 60°C, WSD, micron	406		430	D6079
Conductivity, pS/m	701 @ 22.0°C		1012 @ 23.7°C	D2624
Ash % mass, max	0.007		0.004	D482
Carbon residue, Ramsbottom, % mass, max	0.06		0.07	D524
ASTM D130 Copper Strip Corrosion, 3 hrs at 50°C	1b		1b	
Water and Sediment, % vol, max	0.00	0.00		D2709
Aromaticity %Vol, max: aromatics (vol%)	27.8	27.5		D1319
olefins (vol%)	4.2	4.4		
saturates (vol%)	68	68.1		

B100 Characterization

Four biodiesel (B100) samples procured directly from biodiesel producers. Properties are listed in Table 9. These are intended to represent the following general categories of biodiesel:

- B100 A: High cloud point, high monoglyceride
- B100 B: Low cloud point, high monoglyceride
- B100 C: Low cloud point, low monoglyceride
- B100 D: High cloud point, low monoglyceride

All properties are on specification for D6751 with the exception of the acid value for B100 B (exceeds limit of 0.50 mg KOH/g). The certificate of analysis from the manufacturer lists a value of 0.48 mg KOH/g, very near to the limit, and it is possible that this sample slightly oxidized to have a higher acid value during storage prior to this study or that our slightly high value is due to analytical variability.

Table 9. Properties of B100 used for preparing B5 blends (BD=below detection).

Property	Method	B100 A	B100 B	B100 C	B100 D
Oxidation Stability, h	EN14112	36	8.0	6.7	35
Karl Fischer Water, ppm	D6304	82	260	244	54
Acid Value, mg KOH/g	D664	0.42	0.52	0.36	0.44
Cold Soak Filterability, s	D7501	103	104	89	94
Cloud Point, °C	D5773	14.8	-3.0	1.4	15.3
C18:2, wt%	EN14102	2.21	38.63	41.83	2.07
Free Glycerin, wt%	Modified D6584	0.011	BD	0.019	0.011
Total Glycerin, wt%	Modified D6584	0.153	0.135	0.032	0.089
Monoglycerides, wt%	Modified D6584	0.467	0.392	0.052	0.260
Saturated Monoglycerides, wt%	Modified D6584	0.263	0.080	0.011	0.149

Fuel Blend Characterization Results

Preliminary Cloud Point Blend Study

A cloud point study of the No. 1 and No. 2 diesel fuels was performed to determine the impact on CP as the ratio of No. 1 to No. 2 was increased (Figure 10). It can be seen that increasing amounts of No. 1 diesel into the No. 2 fuel caused the CP to decrease at an almost linear rate until the blend was more than 80% volume No. 1. Table 12 shows CP results for preliminary hand blends prepared to ensure that blends at the target CP level could be prepared. Blend CP values are within 2°C of target with the exception of test fuel (TF) 6 with a CP that is 3°C above the target value.

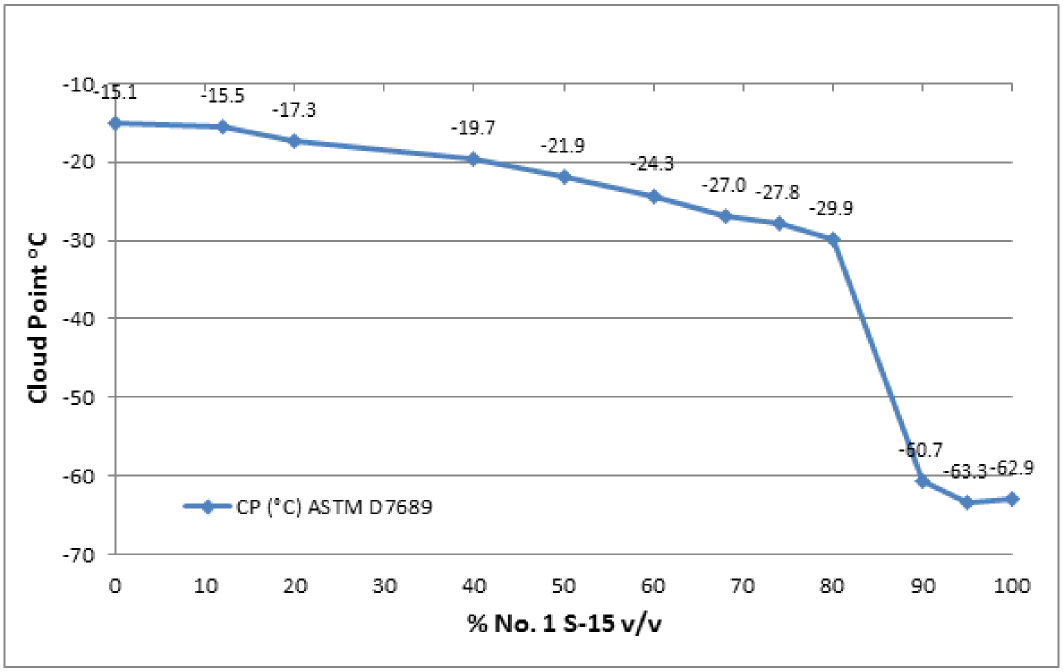


Figure 10: Cloud Point Blend Study for Petroleum Based Blend Components

Table 10: Cloud Point Data for Hand Blends of Test Fuels (*includes additive)

	77	**Lab B**			
Fuel	**B100**	**No. 2 ULSD (% v/v)**	**No. 1 ULSD (% v/v)**	**B100 (% v/v)**	**Avg CP °C D5771**
TF 1	--	32	68	0	-27.0
TF 2	B	40	55	5	-21.5
TF 3*	B	40	55	5	-21.5
TF 4	C	45	50	5	-21.5
TF 5*	C	45	50	5	-21.5
TF 6	A	40	55	5	-23.0
TF 7	D	40	55	5	-20.5
TF 8	B	0	95	5	-40.5

Low-Temperature Operability Properties for Vehicle Test Fuels

Cloud point and FMT results from six laboratories using several different test methods are reported in Table 11. TF 1 is the hydrocarbon control fuel and there is good agreement between the various test methods on the CP of this fuel. In particular, D5773, the most commonly applied method, gives an average result of -27.7°C, essentially the same as the -28°C provided by D2500 (the referee method). There is also good agreement for TF 4 and TF 6, however many of the B5 blends show significantly higher CP by D5773 than by D2500 or other methods, in agreement with previous reports.9 This is believed to be caused by the crystallization of SMG that is not detected as easily by other methods. Fuel TF 8 CP measurements by D5773 showed a much higher standard deviation than for any of the other fuels. The biodiesel blends showed FMT significantly above CP in almost all cases (Figure 11).

Table 11: Cloud Point and FMT Data for Test Fuels in °C

		Lab A	Lab B			Lab C				Lab D	Lab E		Lab F
Fuel	**B100**	**D5773**	**D5771**	**D5773**	**D7689**	**D2500**	**D5772**	**D5773**	**D5773***	**D5773**	**D5773**	**FMT****	**D5773**
TF 1	--	-27	-27	-29.3	-26.8	-28	-30	-29, -27	-27.1	-27.6	-27.6	-23.7	-27.1
TF 2	B	-13	--	-12.7	-22.4	-22	-21	-5, -6	-9.2	-9.2	-8.9	8.2	-9.2
TF 3	B‡	-12	-21	-21.9 -12.5***	-22.7	-23	-22	-9, -7	-10.2	-7.5	-10.8	-4.7	-9.8
TF 4	C	-21	-21	-22.7	-23	-22	-23	-22,-21	-21.8	--	-22.9	-18.3	-21.1
TF 5†	C‡	--	--	-23.5 -13***	--	--	--	--	--	--	-22.4	-12.3	-21.7
TF 6	A	-22	-23	-23.5	-24.3	-25	-25	-23, -21	-22.8	-22.2	-23.2	-17.7, -8.4	-22.0
TF 7	D	-10	-21	-14.7	-22.1	-21	-21	-12, -10	-11.5	-22.9	-11.6	5.6	-10.1
TF 8	B	-36	-41	-42.1 -25***	-41.3	-42	--	-39, -29	-19.5	-10.8	-25.3	-24.5	-27.9

†When two values are given, these are replicate determinations unless otherwise noted. ‡Includes additive. *Re-run after conditioning. **Final melting temperature, °C. ***Early cloud point. †TF 5 was not tested in the AWCD and therefore not widely distributed.

Table 12: Cloud Point Data °C; Average, Minimum and Maximum using all CP methods

Fuel	Avg	Min	Max	D2500
TF 1	-27.8	-30.0	-26.8	-28.0
TF 2	-12.6	-22.4	-5.0	-22.0
TF 3	-14.6	-23.0	-7.0	-23.0
TF 4	-22.0	-23.0	-21.0	-22.0
TF 6	-23.1	-25.0	-21.0	-25.0
TF 7	-15.7	-22.9	-10.0	-21.0
TF 8	-31.6	-42.1	-10.8	-42.0

D2500 Referee test is supplied for comparison

Table 13: Cloud Point Data °C excluding All D5773 Average, Minimum and Maximum for all fuels °C

Fuel	Avg	Min	Max	D2500
TF 1	-28.0	-30.0	-26.8	-28.0
TF 2	-21.8	-22.4	-21.0	-22.0
TF 3	-22.2	-23.0	-21.0	-23.0
TF 4	-22.3	-23.0	-21.0	-22.0
TF 6	-24.3	-25.0	-23.0	-25.0
TF 7	-21.3	-22.1	-21.0	-21.0
TF 8	-41.4	-42.0	-41.0	-42.0

D2500 Referee method is provided for comparison

CFPP results for the test fuels are presented in Table 14, LTFT results in Tables 15-16, and pour point results in Table 17. Figure 12 compares average D5773 CP results to results for CFPP and LTFT. Clearly D5773 CP is more conservative (predicts higher operability limit temperature) than CFPP or LTFT for many fuels. Figure 13 shows the same plot using the average CP from the other (non-D5773) methods.

Table 14: Cold Filter Plugging Point D6371 (CFPP in °C) Data for Test Fuels

Fuel	Lab A	Lab B	Lab C		Lab D	Lab F	Average	Min	Max
TF 1	-28	-29	-27	-28	-31	-27.1	-28	-31	-27
TF 2	-28	-27	-26		-28	-26.2	-27	-28	-26
TF 3	-30	-27	-28		-30	-27.8	-29	-30	-27
TF 4	-27	-27	-25		-26	-25.1	-26	-27	-25
TF 6	-28	-28	-27		-29	-26.0	-28	-29	-26
TF 7	-25	-27	-26	-25	-25	-24.1	-25	-27	-24
TF 8	-48	-48	-45	-41	-48	-45.9	-46	-48	-41

Table 15: Low Temperature Flow Test D4539 (LTFT in °C) Data for Test Fuels

	Lab B		Lab C		Lab D		Lab F	
Fuel	Pass	Fail	Pass	Fail	Pass	Fail	Pass	Fail
TF 1	-27	-28	-27	-29	-25	-27	-26	-27
TF 2	-22	-23	-21	-23	-14	-16	-22	23
TF 3	-24	-25	-21	-23	-4	-6	-25	-26
TF 4	-21	-22	-21	-23	-20	-22	-21	-22
TF 6	-24	-25	-25	-27	-23	-25	-21	-22
TF 7	-23	-24	-21	-23	0	-2	-21	-22
TF 8	-41	-42	-39	-41			-27	-28

Table 16: LTFT in °C Average, Minimum and Maximum Data for Test Fuels

	Avg	Min	Max	Avg	Min	Max
Fuel	Pass			Fail		
TF 1	-26	-27	-25	-28	-29	-27
TF 2	-20	-22	-14	-10	-23	23
TF 3	-23	-25	-21	-25	-26	-23
TF 4	-21	-21	-20	-22	-23	-22
TF 6	-23	-25	-21	-25	-27	-22
TF 7	-22	-23	-21	-23	-24	-22
TF 8	-36	-41	-27	-37	-42	-28

Table 17: Pour Point (PP in °C) Data for Test Fuels

	Lab A	Lab B	Lab C		Lab D	Lab F			
Fuel	D5949	D7346	D6892	D6892	D5949	D5949	Avg	Min	Max
TF 1	-51	-57	-54		-51	-48	-52	-57	-48
TF 2	-33	-30	-30		-29	-33	-31	-33	-29
TF 3	-39	-30	-48		-40	-36	-39	-48	-30
TF 4	-33	-27	-36		-32	-30	-32	-36	-27
TF 6	-45	-38	-39		-48	-45	-43	-48	-38
TF 7	-21	-27	-30	-30	-26	-36	-28	-36	-21
TF 8	-66	-48	-57	-54	-65	-69	-60	-69	-48

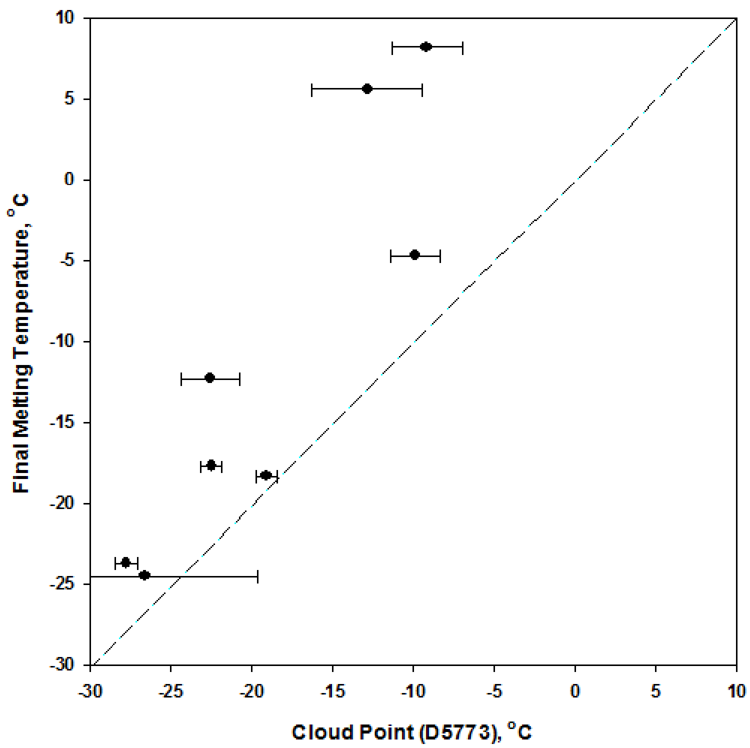


Figure 11. Comparison of final melting temperature with D5773 cloud point.

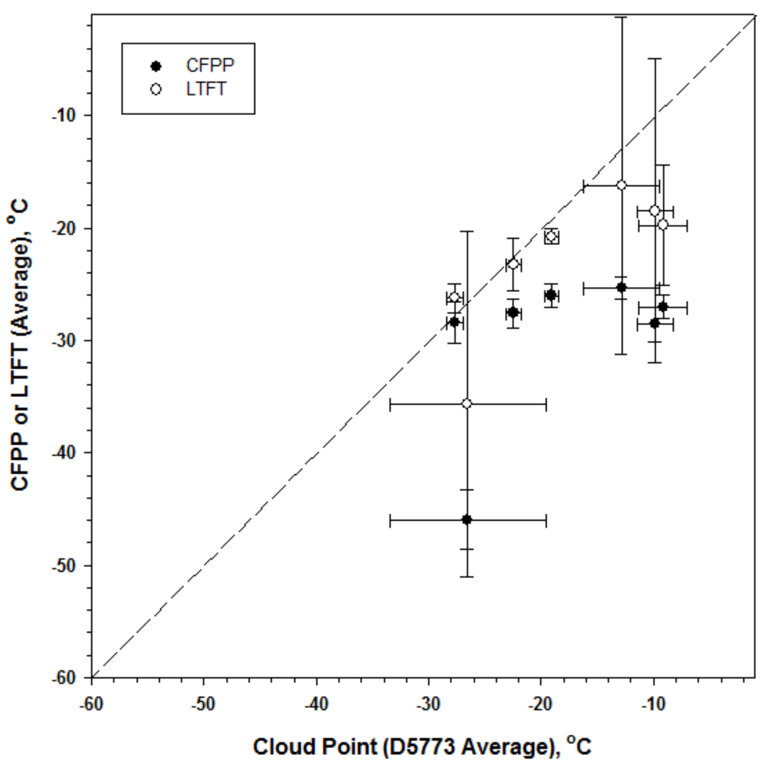


Figure 12. Relationship between D5773 CP and CFPP or LTFT (error bars are 95% confidence intervals based on replicate determinations at several laboratories).

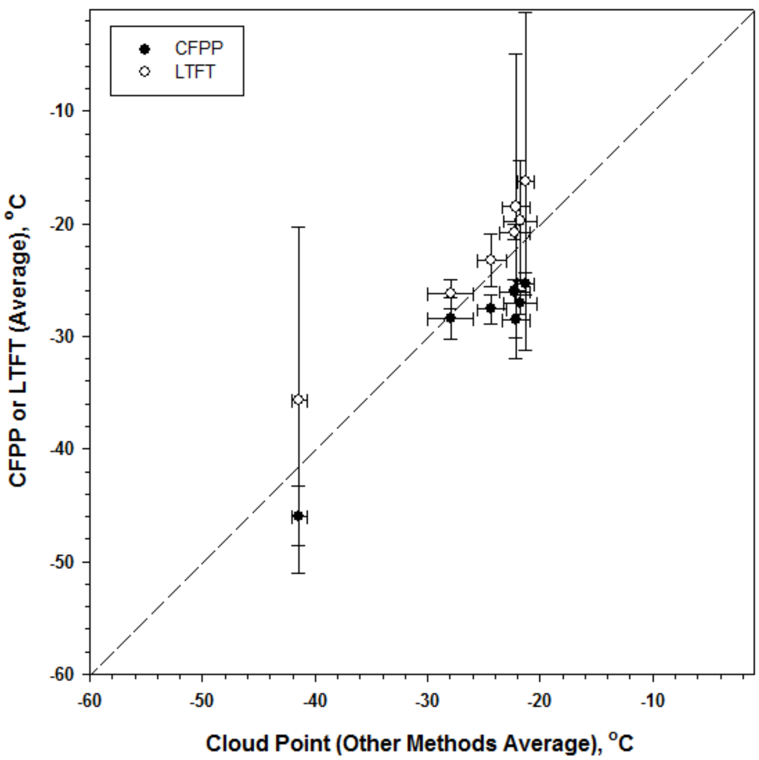


Figure 13. Relationship between average CP by methods other than D5773 and CFPP or LTFT (error bars are 95% confidence intervals based on replicate determinations at several laboratories).

Upper and Lower Fuel Tank Sample Data

Table 18 shows fuel CP and final test cell temperature for each vehicle test. In three cases the test lab failed to perform diurnal cycling and simply cooled the cars to test temperature and held them there for the prescribed period of time. There were also three tests where the test temperature was more than 1 to 1.5°C above CP as measured by the non-D5773 tests as noted above, D5773 CP results were in many cases significantly higher than results from other tests.

Observations of the samples were recorded as the samples were being collected for each test and these are shown in Table 18. In many cases upper samples were clear while lower samples were visually cloudy. Notably the bottom sample for TF3, which included a wax anti-settling additive, was visibly cloudy. Table 23 presents results of results of CP and FMT testing for these samples. CFPP and CP by D7689 was measured by Lab B and the CP and FMT using D5773 was measured by Lab E. In general, Lab B and Lab E agreed on CP measurements except for TF2 and TF8. This could be due to the better sensitivity of D5773 versus D7689.9 CFPP did not seem to indicate any difference between the top and bottom samples for any of the vehciles or fuels tested.

Table 18. Comparison of fuel CP and final test cell temperatures.

	D5773 Mean CP	Other Mean CP	Final Test Cell Temperature, C
TF 1 Vehicle 1	-27.7	-28	-27
TF 1 Vehicle 2	-27.7	-28	-27
TF 1 Vehicle 3	-27.7	-28	-27
TF 2 Vehicle 1	-9.2	-21.8	-21
TF 2 Vehicle 2	-9.2	-21.8	-21
TF 2† Vehicle 3	-9.2	-21.8	Data collection failure
TF 3# Vehicle 1	-11	-22.2	-25
TF 4 Vehicle 1	-19.1	-22.3	-21
TF 4 Vehicle 2	-19.1	-22.3	-21
TF 4 Vehicle 3	-19.1	-22.3	-19
TF 6 Vehicle 1	-22.5	-24.3	-25
TF 6 Vehicle 3	-22.5	-24.3	-21*
TF 7 Vehicle 1	-12.9	-21.3	-20
TF 7 Vehicle 2	-12.9	-21.3	-20
TF 7 Vehicle 3	-12.9	-21.3	-18
TF 8 Vehicle 1	-28.7	-41.4	-41*
TF 8 Vehicle 3	-28.7	-41.4	-40*

†Data recorder failed to collect any data from this test.
#Fuel with wax antisettling additive.
*No diurnal cycle.

TF1 was the control fuel and it did not contain any B100. It does not appear that wax settling took place for the three vehicles run on this fuel as neither the CP and FMT for the top and bottom samples show major differences between them. Both Lab B and E CP measurements are in close agreement. There was a descrepancy for vehicle 1 however where it appears that the top and bottom samples may have been switched or mislabeled as the CP for the bottom samples was actually lower than that from the top. For this fuel, the vehicle was cooled below CP in all cases, and the test temperature was about 2-5°C above the CP of the fuel.

TF2 contained 5% biodiesel that had high MG content. For this fuel, Lab B did not see any issues with CFPP or an increase in the CP between top and bottom samples. However, Lab E, saw a large increase in the CP using D5773 which could be due to the better sensitivity of this method versus D7689. Also there was a large temperature variation (~30°C) between CP and FMT indicating that SMGs were present in the bottom samples suggesting that SMGs and/or wax settling occurred for this fuel in all three vehicles tested. This fuel was cooled below the CP in each case as well. For vehicles 1 and 2, the test temperature was 5-7°C above the CP, but for vehicle 3, the test temperature was below the CP of the fuel by 5°C. This did not seem to affect the results for vehicle 3 versus vehicles 1 and 2 in terms of the CP reults.

TF3 also contained 5% biodiesel with high MG content, but an additive was used in this fuel. Lab B did not see a change in the CP from the top to the bottom samples. Lab E did see an increase in CP from the top to the bottom and also a jump in the FMT, but not as large as for TF2. This would indicate that the additive helped with the wax settling for this vehicle test run. This fuel was only run in one vehicle. The vehicle was held below the CP and was run 2°C above the CP during testing.

TF4 contained 5% biodiesel with low amounts of MGs. Neither Lab B or Lab E found large differences in the CP between top versus bottom samples indicating that no wax settling occurred for this fuel in any of the three vehicles run. For this fuel, cooling and run test temperatures were very inconsistent. Vehicles 1 and 2 were held below the CP, but vehicle 3 was not. Also vehicles 2 and 3 were run well above the CP of the fuel while vehicle 1 was run 2°C above CP.

TF5 No vehicles were run with TF5 due to time constraints.

TF6 contained 5% biodiesel with a high MG and a high initial CP. For this fuel, Lab E again saw a slight increase in the top versus the bottom samples indicating that some wax settling did occur. This increase was not noted by Lab B. This is surprising in that a high MG B100 would be expected to cause some issues with wax settling as was seen with TF2. The difference between TF2 and TF 6 is that TF2 was a low CP B100 and TF6 was a high CP B100. reason issues may not have been identified with TF6 is because vehicle 1 was cooled to just below the CP, where as with TF2, the vehicles were cooled about 5°C below the CP. Vehicle 3 was never cooled below the CP and was also tested 6°C above the CP which is higher than with most of the other test runs.

TF7 contained 5% biodiesel and had low MG content in a high CP B100. Lab E once again saw an increase in CP between the top and bottom samples that Lab B did not. There was also an increase in the FMT noted by Lab E. This would indicate that SMGs were present and/or wax settling was occuring. This was not as significant as with TF2 and was most significant in vehicle 1 versus any of the other vehicles. Vehicles 1 and 3 were cooled to below the CP, but Vehicle 2 was only cooled to within 6°C of the CP. Both vehicles 2 and 3 were run above the CP with Vehicle 2 run almost 15°C above CP leading to large inconsistencies in the way these vehicles were tested with this fuel.

TF8 contained 5% biodiesel blended with a low CP disel fuel. While a large difference in the top and bottom was not noted by Lab E, the CPs measured by Lab E were much higher (20°C) than those measured by Lab B. It does not appear that wax settling occurred for this fuel, but the large difference in CP measurements between labs may indicate that there was material coming out of solution that was not detected by Lab B. It was noted that this fuel was cloudy and separated prior to the start of testing. Because the CP of this fuel was so low, it appears that the test chamber was cooled as close to -40°C as possible, but neither vehicle was held below CP. Vehicle 3 was run well above CP and Vehicle 1 failed to start. Photographs of fuel samples placed in the chamber are found in Appendix F.

Table 19: Test Facility Upper and Lower Fuel Sample Observations

Vehicle	Fuel #	Upper sample observations	Upper Sample Temp.	Lower Sample Observations	Lower Sample Temp.
Vehicle 1	1	Clear	N/A	Cloudy/ thicker Flowed slow	N/A
Vehicle 2	1	Clear	N/A	Cloudy Flowed well	N/A
Vehicle 3	1	Clear	N/A	Cloudy, Slow to flow	N/A
Vehicle 1	2	Cloudy, still thin	N/A	Cloudy, film on surface	N/A
Vehicle 2	2	Cloudy, still thin	N/A	Cloudy, Slow to flow	N/A
Vehicle 3	2	Cloudy, still thin	N/A	Cloudy, Thicker, slow to flow	N/A
Vehicle 1	3	Clear	-13.6	Very Cloudy	-18.5
Vehicle 1	4	Slighty cloudy	N/A	Cloudy wax/film on surface	N/A
Vehicle 2	4	Clear	N/A	Slightly Cloudy	N/A
Vehicle 3	4	Clear	N/A	Cloudy/waxy	N/A
Vehicle 1	6	Clear	-15	Slightly Coudy/ Slight seperation	-17.6
Vehicle 3	6	Clear / slight cloudy	-13.7	Cloudy slight gelling	-14.8
Vehicle 1	7	Clear / slight cloudy	N/A	Cloudy with film on surface	N/A
Vehicle 2	7	Clear	N/A	Clear	N/A
Vehicle 3	7	Clear	N/A	Clear	N/A
Vehicle 1	8	Cloudy	-31.8	Cloudy, Separated	-28.8
Vehicle 3	8	Slighty cloudy	-30.2	Slighty cloudy, runs well	-34.8

Table 20: Upper and Lower Fuel Tank Sample Low Temperature Property Data

	Lab B				Lab E				Notes
	CFPP D6371		CP D7689		CP D5773		FMT D5773		
Fuel	Upper	Lower	Upper	Lower	Upper	Lower	Upper	Lower	
TF 1 Vehicle 1	-22	-29	-25.5, -9.0	-30.0, -29.3	-23.4	-27.5	-19.8	-23.36	Top and bottom samples ID appears to be switched.
TF 1 Vehicle 2	-32	-25	-32.8, -31.1	-27.4, -14.1	-29.2	-24.7	-26.6	-23.1	~5°C difference between top and bottom CP-wax settling?
TF 1 Vehicle 3	-32	-31	-31.8, -15.2	-30.9, -30.0	-29.1	-29.4	-27.4	-26.7	No variation in top and bottom samples, no increase in FMT
TF 2 Vehicle 1	-30	-31	-23.4	-21.8	-23.2	-4.5	-20	10.5	Large CP difference between top and bottom samples. Large increase in FMT indicates monos and polymorphism
TF 2 Vehicle 2	-29	-30	-23.0	-22.1, -21.6	-21.8	-6.7	-18.3	9.4	Large variation in top vs. bottom samples. High FMT indicative of monos and polymorphism
TF 2 Vehicle 3	-33	-27	-28.2	-21.5	-27.7	-8.3	-24.3	6	Large variation in top vs. bottom CP Increase in FMT indicative of monos and polymorphism
TF 3 Vehicle 1	-30	-29	-26.4, -21.5	-22.0, -23.2	-25.9	-16.4	-21.4	-5.7	Increase in FMT indicates monos and polymorphism. See early and late rise in CP on Phase Tech-also indicates SMGs .This fuel was additized-low CP B100/high MG
TF 4 Vehicle 1	-23	-25	-23.5, -11.7	-21.5, -21.1	-22	-22	-19.6	-19.3	No variance top vs. bottom CP or FMT
TF 4 Vehicle 2	-28	-25	-22.8, -22.8	-22.4, -22.6	-21.6	-22.4	-18.3	-19	No variation in top vs. bottom samples. No high FMT
TF 4 Vehicle 3	-24	-25	-21.5, -22.9	-23.0, -22.8	-22.4	-22.5	-18.6	-19.2	No variation in top vs. bottom samples. No increase in FMT
TF 6 Vehicle 1	-27	-27	-25.1, -24.8	-23.4	-23.7	-23.6	-18.9	-17.9	~5°C difference in CP and FMT
TF 6 Vehicle 3	-27	-28	-23.2, -24.2	-22.6, -24.3	-23.5	-23.1	-18	-18.2	~5°C difference in top vs. bottom for CP and FMT
TF 7 Vehicle 1	-31	-27	-24.6, -24.6	-20.1, -13.2	-24.1	-5.8	-21.1	11.4	Large increase in FMT indicative of polymorphism or wax settling in tank?
TF 7 Vehicle 2	-29	-28	-20.6, -21.8	-20.6, -21.0	-20.5	-20.2	-17.4	-13	Slight rise in FMT for bottom sample
TF 7 Vehicle 3	-23	-26	-21.4, -22.2	-20.7, 20.5	-20.7	-20.5	-18.7	-18	No high FMT or large variance in top vs. bottom
TF 8 Vehicle 1	-46	-47	-42.3, -41.6	-41.9, -39.6	No data	No data	No data	No data	
TF 8 Vehicle 3	-46	-46	-42.2, -41.9	-42.1, -41.6	-20.9	-22.1	-25.6	-24.4	See late rise in CP method at -38°C-can indicate monos present

Table 21 provides a comparison of upper vs. lower fuel tanks samples. The top and bottom samples for Vehicle 1/TF 1 appear to have been switched. The "Total n-paraffin content" represents the total of the n-paraffins of the fuel. The n-paraffin range was within C_7 through C_{24} for all fuels. This method does not show the presence of FAME.

A higher n-paraffin result does necessarily indicate wax precipitation. Figure 14 compares the n-paraffin content of the upper and lower samples of TF 2 Vehicle 1 by carbon number. The total n-paraffin content was virtually identical. Figure 15 is from the same data set but shows that the lower sample was weighted with higher carbon number n-paraffins.

Table 21 also provides differential scanning calorimetric (DSC) data comparison for the upper and lower samples. The heat flow data is measured from the onset to 10°C below the onset. A higher value usually represents a relative increased level or combination of; n-paraffin content, SMGs, and saturated FAME.

Table 21: Upper and Lower Fuel Tank Sample Property Data

	Total n-paraffin content %w/w			DSC			
Fuel	Upper	Lower	Delta Lower - Upper	Upper J/g	Lower J/g	Delta Lower Upper	Notes
TF 1 Vehicle 1	15.19	15.32	0.13	4.94	5.17	4.68%	Top and bottom switched?
TF 1 Vehicle 2	14.93	14.95	0.02	4.90	5.00	2.01%	~5°C difference between top and bottom CP-wax settling?
TF 1 Vehicle 3	14.90	14.92	0.02	4.94	4.99	1.05%	No variation in top and bottom samples, no FMT increase
TF 2 Vehicle 1	13.14	13.10	-0.04	2.20	2.38	8.39%	Large CP difference between top and bottom samples. Large increase in FMT indicates monos and polymorphism
TF 2 Vehicle 2	12.91	13.19	0.28	2.21	2.32	4.98%	Large variation in top vs. bottom samples High FMT indicative of monos and polymorphism
TF 2 Vehicle 3	12.95	13.09	0.15	2.19	2.37	8.06%	Large variation in top vs. bottom CP Increase in FMT indicative of monos and polymorphism
TF 3 Vehicle 1	13.01	13.23	0.22	2.18	2.34	7.34%	Increase in FMT indicates monos and polymorphism. See early and late rise in CP on Phase Tech-also indicates SMGs. This fuel was additized-low CP B100/high MG
TF 4 Vehicle 1	12.94	12.49	-0.46	1.23	1.29	4.86%	No variance in top vs. bottom CP or FMT
TF 4 Vehicle 2	13.03	12.87	-0.16	1.27	1.29	1.49%	No variation in top vs. bottom samples. No high FMT
TF 4 Vehicle 3	13.02	12.74	-0.28	1.27	1.31	3.07%	No variation in top vs. bottom samples. No FMT increase
TF 6 Vehicle 1	13.14	13.18	0.04	3.05	3.08	0.98%	~5°C difference in CP and FMT
TF 6 Vehicle 3	13.13	13.14	0.02	2.85	3.08	8.03%	~5°C difference in top vs. bottom for CP and FMT
TF 7 Vehicle 1	13.59	13.30	-0.29	2.30	3.22	40.00%	Large increase in FMT indicative of polymorphism or wax settling in tank?
TF 7 Vehicle 2	13.49	13.35	-0.14	2.69	2.75	2.23%	Slight rise in FMT for bottom sample
TF 7 Vehicle 3	13.39	13.31	-0.08	2.75	2.96	7.63%	No high FMT or large variance in top vs. bottom
TF 8 Vehicle 1	17.42	14.15	-3.27	6.25	6.32	1.10%	
TF 8 Vehicle 3	16.36	No data	No data	6.08	6.05	-0.49%	Late rise in CP method at -38°C-can indicate monos present

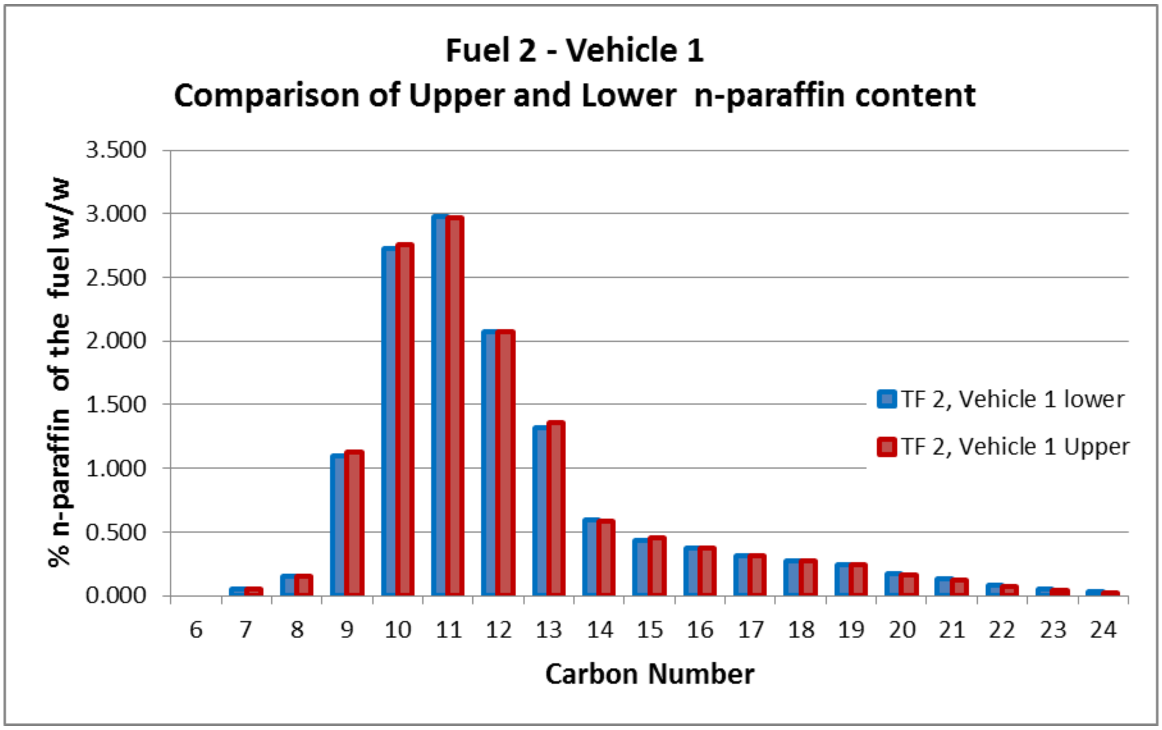


Figure 14: Example of the comparison of upper sample and lower sample n-paraffin content taken just before vehicle test cycle.

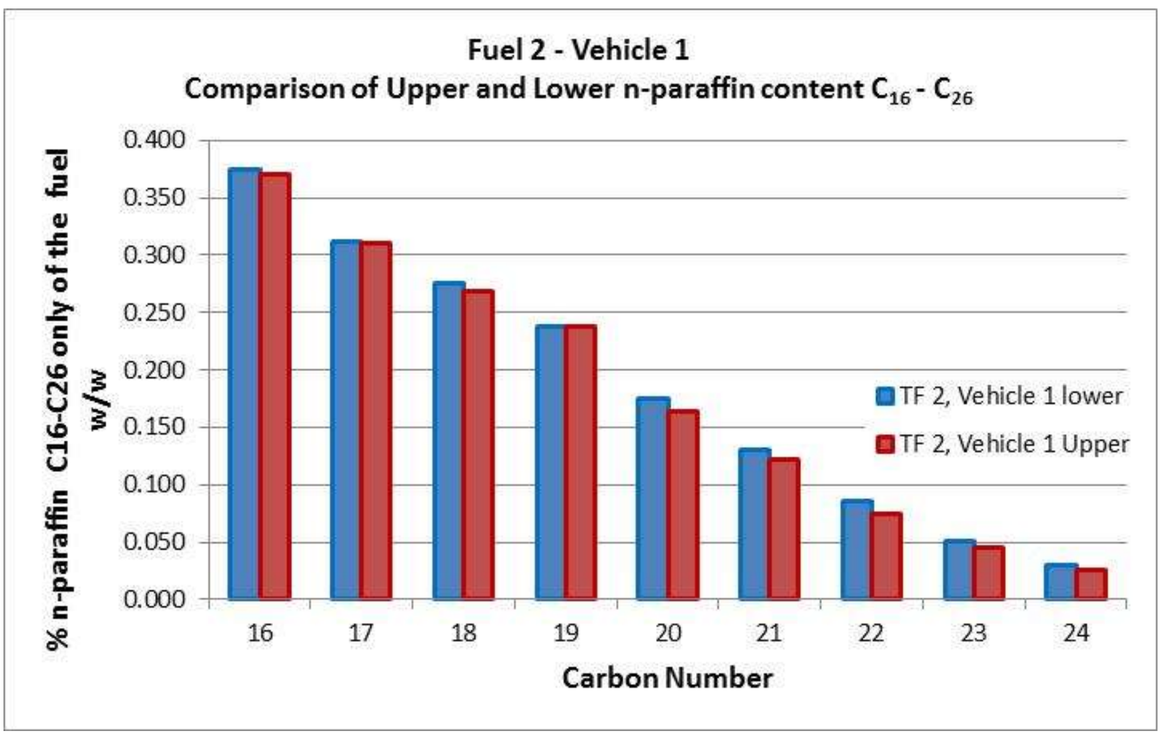


Figure 15: Example of the comparison of upper sample and lower sample n-paraffin content taken just before vehicle test cycle.

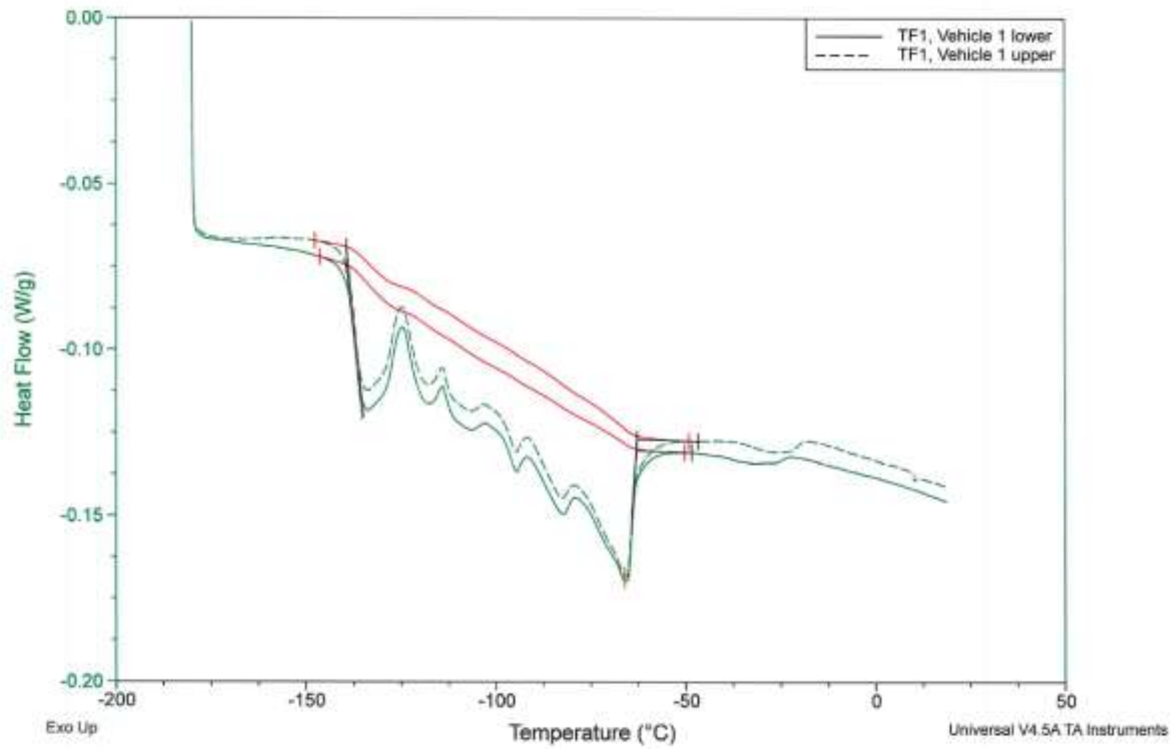


Figure 16: Example of the comparison of upper sample and lower sample of DSC curves taken just before vehicle test cycle. Fuel1 Vehicle 1

Table 22: FTIR - Key Peaks

Fuel	Vehicle	Peak area lower	Peak area upper	Delta	Notes
1	1	0.000	0.000	0	Top and bottom switched?
1	2	0.000	0.000	0	~5°C difference between top and bottom CP-wax settling?
1	3	0.000	0.000	0	No variation in top and bottom samples, no increase in FMT
2	1	0.334	0.326	2.45%	Large CP difference between top and bottom samples. Large increase in FMT indicates monos and polymorphism
2	2	0.326	0.281	16.01%	Large variation in top vs. bottom CP Increase in FMT indicative of monos and polymorphism
2	3	0.357	0.326	9.51%	Large variation in top vs. bottom samples High FMT indicative of monos and polymorphism
3	1	0.350	0.311	12.54%	Increase in FMT indicates monos and polymorphism. See early and late rise in CP on Phase Tech-also indicates SMGs This fuel was additized-low CP B100/high MG
4	1	0.353	0.331	6.65%	No variance in top vs. bottom CP or FMT
4	2	0.292	0.290	0.69%	No variation in top vs. bottom samples No high FMT
4	3	0.282	0.229	23.14%	No variation in top vs. bottom samples No increase in FMT
6	1	0.346	0.255	35.69%	~5°C difference in CP and FMT
6	3	0.288	0.275	4.73%	~5°C difference in top vs. bottom for CP and FMT
7	1	0.299	0.297	0.67%	Large increase in FMT indicative of polymorphism or wax settling in tank?
7	2	0.270	0.200	35.00%	Slight rise in FMT for bottom sample
7	3	0.291	0.274	6.20%	No high FMT or large variance in top vs. bottom
8	1	0.357	0.331	7.85%	
8	3	0.357	0.331	7.85%	See late rise in CP method at -38°C-can indicate monos present

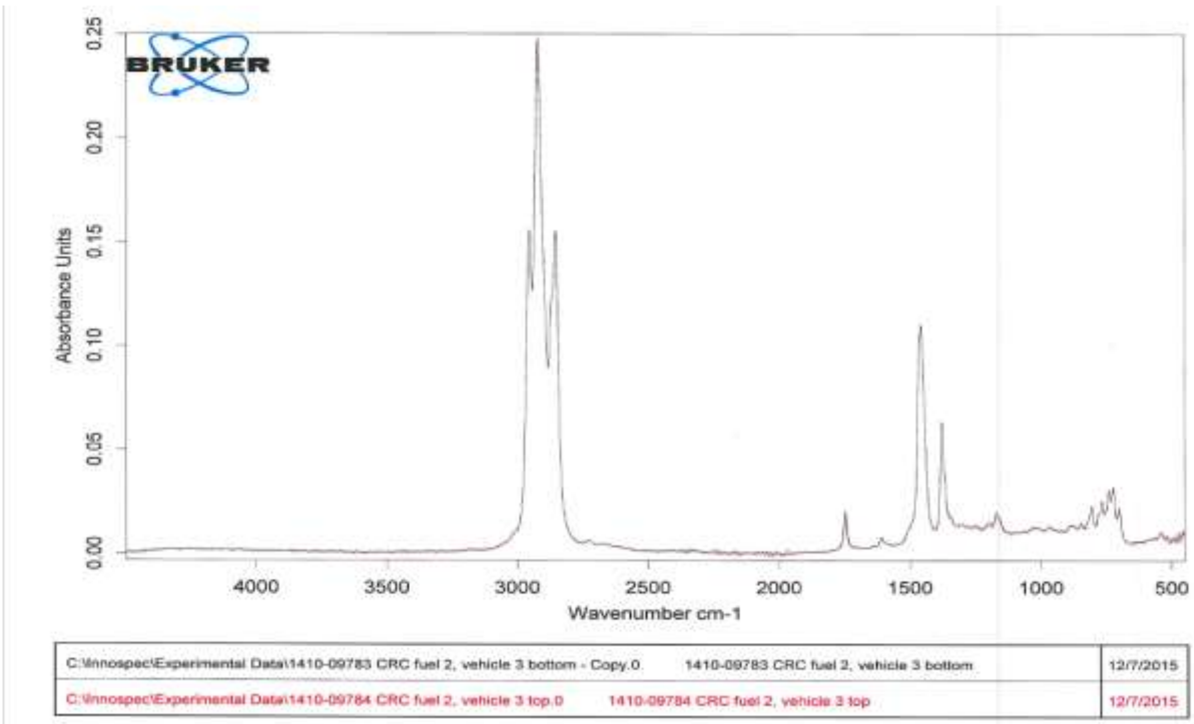


Figure 17: FTIR comparison of Upper and lower samples from Fuel 2 Vehicle 3

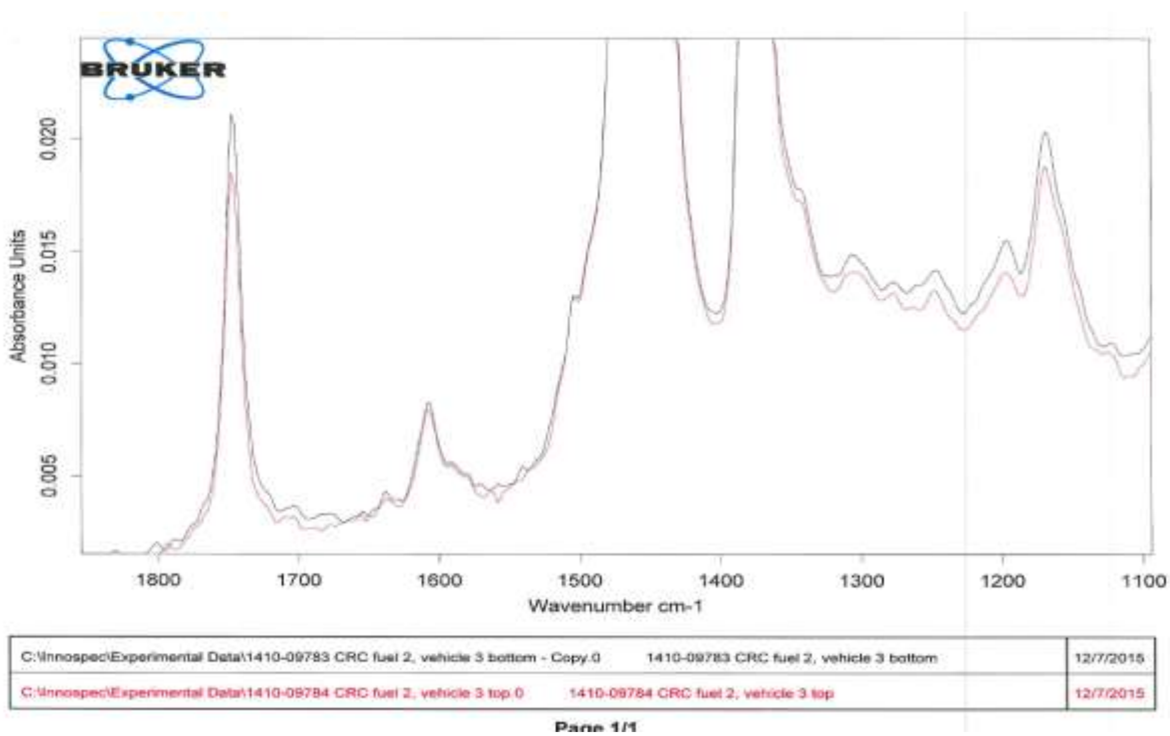


Figure 18: FTIR comparison of Upper and lower samples from Fuel 2 Vehicle 3

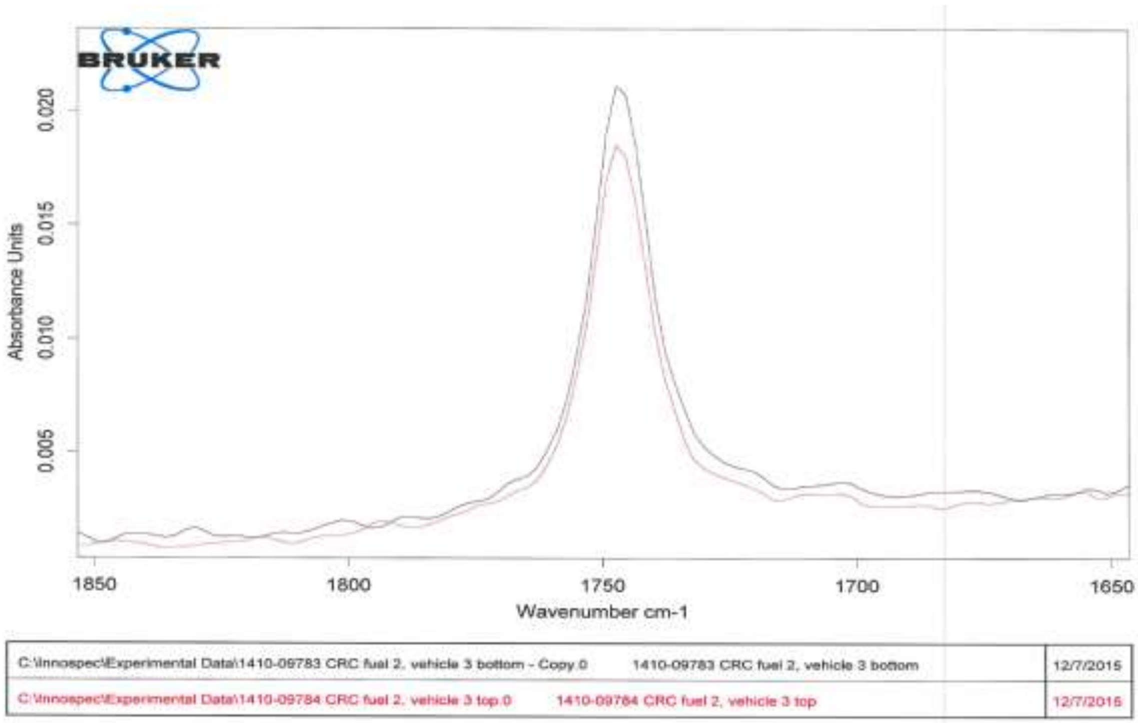


Figure 19: FTIR comparison of Upper and lower samples from Fuel 2 Vehicle 3

Filter Sample Analysis Data

Fuel filters were obtained post-test for many fuel-vehicle combinations. In cases where fuel remained in the vehicle filters and could be drained off, a sample was taken and tested for CP and FMT and results are shown in Table 23.

For TF1, there was no increase in CP or FMT from the drained fuel when compared to the base fuel prior to being run in the vehicle. This is as expected because this was the control fuel and there was no B100 blended in. The exception was Vehicle 1 which could be due to a labeling error or an error during the experimental set up where a fuel swap was not done properly (SMG (monostearin) was found in this filter sample which should have contained no B100: See filter analysis section).

For TF 2, 4, and 7, there was an increase in the CP indicating that some of the MG present in the B100 was removed by the filter as expected.

Table 23: CP and FMT Results for Fuel Drained from Filters

ID	Sample	CP (loop)	CP (base oil)	FMT	Notes
1412-14398	Fuel 2 Vehicle 2				
1412-14399	Fuel 7 Vehicle 1	-7.6	-7.7	5.2	High FMT-varying CP results by lab
1412-14400	Fuel 7 Vehicle 3	-6.6	-9.8	-4.5	Lower FMT than vehicle 1
1412-14401	Fuel 1 Vehicle 3	-20.3	-25.6	- 23.2	baseoil method matches with CP reported by other labs
1412-14402	Fuel 4 Vehicle 1	-5.5	-5.7	6.8	Higher CP than other labs. High FMT
1412-14403	Fuel 2 Vehicle 1				
1412-14404	Fuel 4 Vehicle 2				
1412-14405	Fuel 8 Vehicle 1				
1412-14406	Fuel 8 Vehicle 3				
1412-14407	Fuel 2 Vehicle 3	-11.9	-8.9/-19.8 (late)	-4.0	Varying CP results by lab. Matches with Lab A
1412-14408	Fuel 6 Vehicle 3				
1412-14409	Fuel 1 Vehicle 2	-25.8	-28.1	- 22.5	Matches with other labs
1412-14410	Fuel 6 Vehicle 1				
1412-14411	Fuel 1 Vehicle 1	-10	-8/-11 (late)	6.2	Much higher CP and FMT Sample label issue?
1412-14412	Fuel 4 Vehicle 3				
1412-14413	Fuel 7 Vehicle 2	-4.4	-8.4	0.7	Varying CP results by lab. Matches labs A and C

Fuel filters from vehicles were sent to Lab E for analysis of SMGs present by Modified D6584. The filters were cut open and a portion of the filter material was removed. The filter material was then washed consecutively in three solvents of increasing polarity, heptane, 1:1 chloroform:methanol, and pyridine. After extraction in each solvent, the solvent was removed under a stream of nitrogen, internal standards were added, and the residue was derivatized using N-Methyl-N-(trimethylsilyl) trifluoroacetamide (MSTFA). The samples were allowed to react for 15 minutes before being diluted with 8 mL of heptane. The samples were run on the GC following Modified D6584 and the % of each component was calculated. For the most part, there was nothing found in the heptane fractions except residual fuel. In the case of TF8, there were SMGs found in all solvent fractions. Filters exposed to TF 2 appeared to contain the most SMGs. In general, MGs and SMGs were found on all of the filters from all three vehicles with all of the TFs except TF1 indicating that the filters removed some of the components that crystallized out of solution. Because the entire filter was not analyzed, an accurate quantitation of how much MG and SMG present was difficult, but a general ratio of components removed is shown in Table 24.

Table 24: ASTM Modified D6584 Results for Filters Removed from Vehicles

ID	Sample	Heptane	Chloroform:methanol	Pyridine
1412-14403	Fuel 2 Vehicle 1	N/A	0.14% sat/0.15% unsat	0.6% sat/0.4%unsat/0.4% glycerin
1412-14398 A	Fuel 2 Vehicle 2	1.7% sat/ 0.14% unat	5.6% sat/0.44% unsat	6.9% sat/0.63% unsat
1412-14398B	Fuel 2 Vehicle 2	0.4% sat	N/A	5%sat/0.4% unsat
1412-14407	Fuel 2 Vehicle 3	N/A	0.6% sat/0.16% unsat	N/A
1412-14402 E	Fuel 4 Vehicle 1	N/A	N/A	0.8% sat/0.9% unsat
1412-14402 I	Fuel 4 Vehicle 1	N/A	0.15% sat/0.11% unsat	0.5% sat/0.3% unsat/1.4% glycerin
1412-14410 E	Fuel 6 Vehicle 1	N/A	N/A	0.08% sat/0.13% unsat
1412-14410 I	Fuel 6 Vehicle 1	N/A	0.16%sat/0.24% unsat	0.4%sat/0.29% unsat
1412-14408	Fuel 6 Vehicle 3	N/A	0.25%sat/0.36% unsat	0.17%sat/0.15% unsat
1412-14399 E	Fuel 7 Vehicle 1	N/A	0.5% sat/0.06% unsat	7.4% sat/1.9% unsat
1412-14399 I	Fuel 7 Vehicle 1	N/A	0.3% sat/0.19% unsat	2% sat/0.9% unsat/1.84% glycerin
1412-14413	Fuel 7 Vehicle 2	N/A	0.25% sat/0.02% unsat	0.33% sat/0.02% unsat
1412-14405 E	Fuel 8 Vehicle 1	0.13% sat/ 0.26% unsat	0.06% sat/0.1% unsat	0.14% sat/0.14% unsat
1412-14405 I	Fuel 8 Vehicle 1	0.15% sat/ 0.14% unsat	0.5% sat/0.4% unsat	0.16% glycerin
1412-14406	Fuel 8 Vehicle 3	0.1% sat/ 0.14% unsat	0.5% sat/0.5% unsat	1.1% sat/0.5% unsat/0.16% glycerin

6. Vehicle LTO Results

Driver Recordings and Test Log

Depending upon the severity of wax plugging in the fuel system, the performance of a diesel vehicle will be affected in different ways. These are, in order of increasing severity:

1. No observable effect upon performance.
2. Slight fluctuation of speed (surge), engine misfire, or the need for significant pedal adjustment to maintain speed.
3. Inability to maintain speed even with pedal fully depressed.
4. Stalling of engine.

Record occurrence at the end of each minute of testing.

Below is a log of the fuels that were tested in each vehicle, the date of the test and operator comments.

Table 25. Driver comments test log for each vehicle test.

Vehicle	Fuel #	Date	Comment
Vehicle 1	1	4/14/2014	Soak control error. Testing completed despite soak error. Test complete with no issues
Vehicle 2	1	4/21/2014	Vehicle ran with no issues.
Vehicle 3	1	4/28/2014	Vehicle ran with no issues. Stall on take off due to opperator error. Restart and run.
Vehicle 1	2	4/28/2014	Vehicle ran with no issues.
Vehicle 2	2	4/28/2014	Vehicle ran with no issues.
Vehicle 3	2	5/1/2014	Traction control error with vehicle. No warning lights but throttle limited.
Vehicle 1	3	6/9/2014	Vehicle ran with no issues.
Vehicle 1	4	5/12/2014	Underbody fans used in soak for this test on. Vehicle ran with no issues.
Vehicle 2	4	5/12/2014	Vehicle ran with no issues.
Vehicle 3	4	5/12/2014	Removed traction control fuse and ABS fuse. Vehicle ran fine
Vehicle 1	6	6/2/2014	Vehicle ran with no issues.
Vehicle 3	6	5/26/2014	Difficult to start. Once started ran with no issues.
Vehicle 1	7	5/5/2014	Vehicle ran with no issues.
Vehicle 2	7	5/5/2014	Vehicle ran with no issues.
Vehicle 3	7	5/5/2014	Second error with traction control. Restart engine ran test to completion.
Vehicle 1	8	5/26/2014	Failed to start. Constant cold soak.
Vehicle 3	8	6/2/2014	Tecnically fail to start. Allowed engine compartment to warm slightly started and ran.

Vehicle 1

Vehicle started well for all tests other than final test of fuel 8. Vehicle would not start following a constant -40C soak. Issue was not with fuel system but with electrical. Vehicle would not turn over sufficiently to induce a engine start. Block heater was not used for testing.

Vehicle 2

Vehicle could not be shifted in and out of neutral at cold temperatures. The manual override for the transmission would freeze up at low temperatures. Electronic key resignation was inconsistent at cold temperatures. Battery was replaced in key fob and fob was maintained in a warm area despite these steps the vehicle would not recognize key consistently causing starting difficulties.

Vehicle 3
Vehicle exhibited inconsistent issues with traction control at low temps with no indication of traction control engagement. Vehicle would cause issue for a short time then run properly. Traction control and ABS fuses were removed. System operated better following removal of fuses.

Testing struggled to achieve desired fuel temperatures in vehicles. It was anticipated that the fuel would soak down to temperatures close to ambient air in the allotted time during the temperature cycle. It was discovered that the fuel did not reach anticipated temperatures. Investigations revealed sensitivity to the underbody airflow and large influence on the temperature of the fuel. If underbody air flow was increased above the recommended 7 mph level fuel would reach a closer temperature. Vehicle 1 showed the greatest impact on this and also was the vehicle with the most exposed fuel tank. Vehicle 2 and Vehicle 3 showed the highest resistance to fuel cooling possible caused by the close to floor board fuel tank mounting. This close mounting would only allow air to freely circulate under the fuel tank of these two vehicle while the more exposed fuel tank of Vehicle 1 would allow circulation or air around much more of the fuel tank.

Fidelity of Diurnal Cooling Cycles

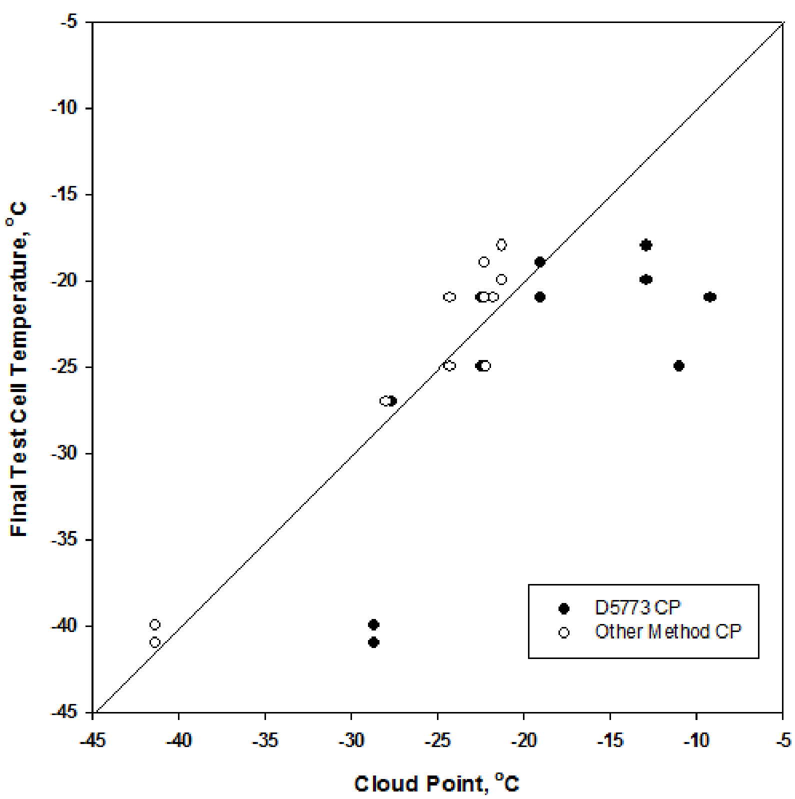


Figure 20. Comparison of test temperature and fuel cloud point for vehicle tests.

Operability Results for Each Vehicle

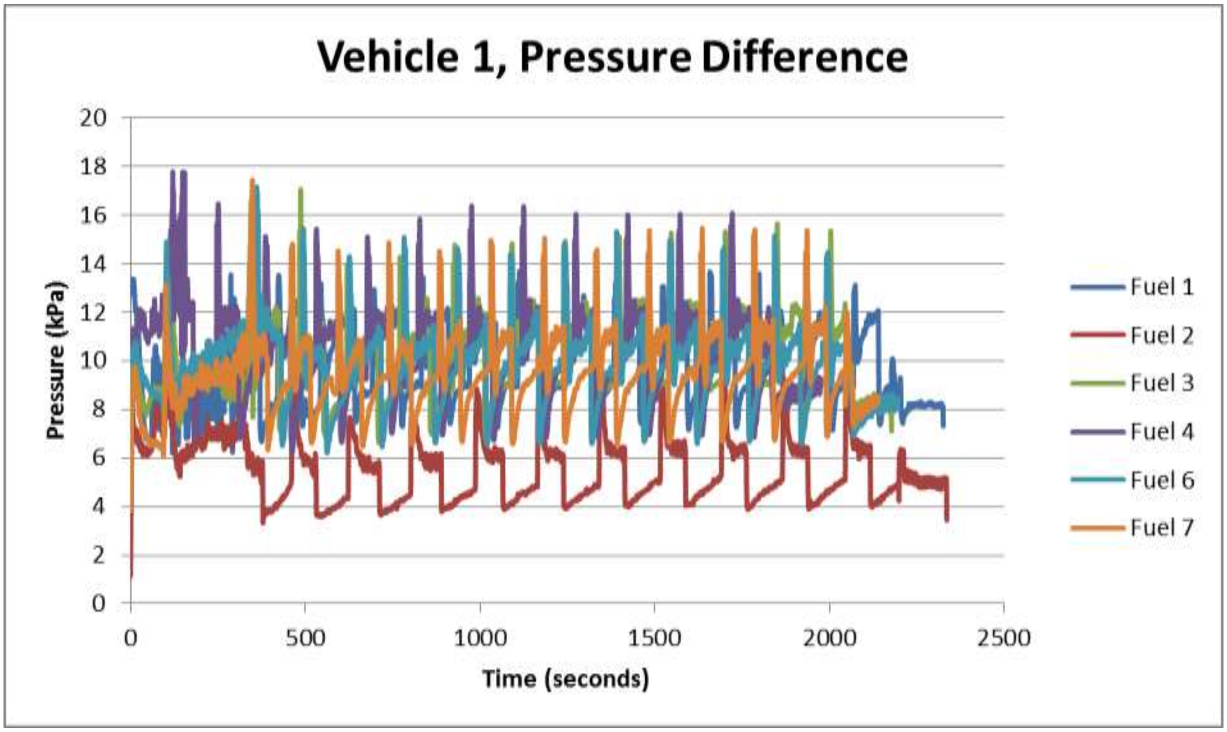


Figure 21: Pressure Differences for fuels in vehicle 1

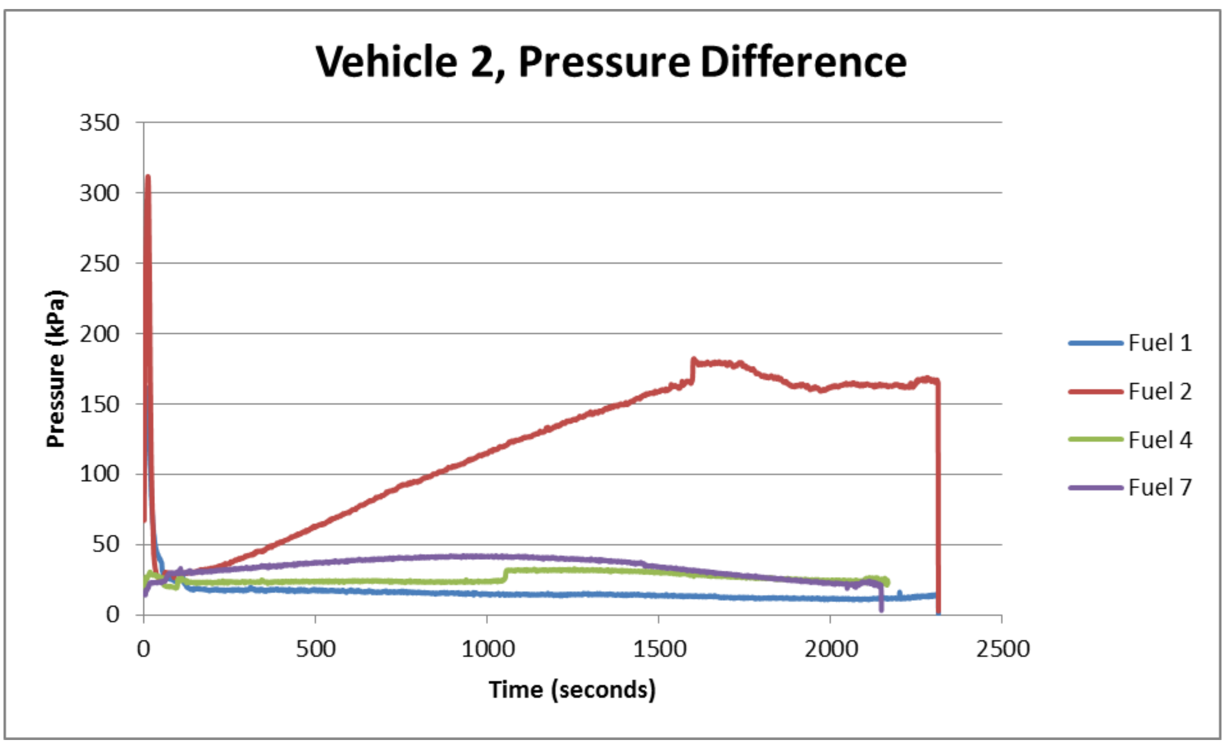


Figure 22: Pressure Differences for fuels in vehicle 2

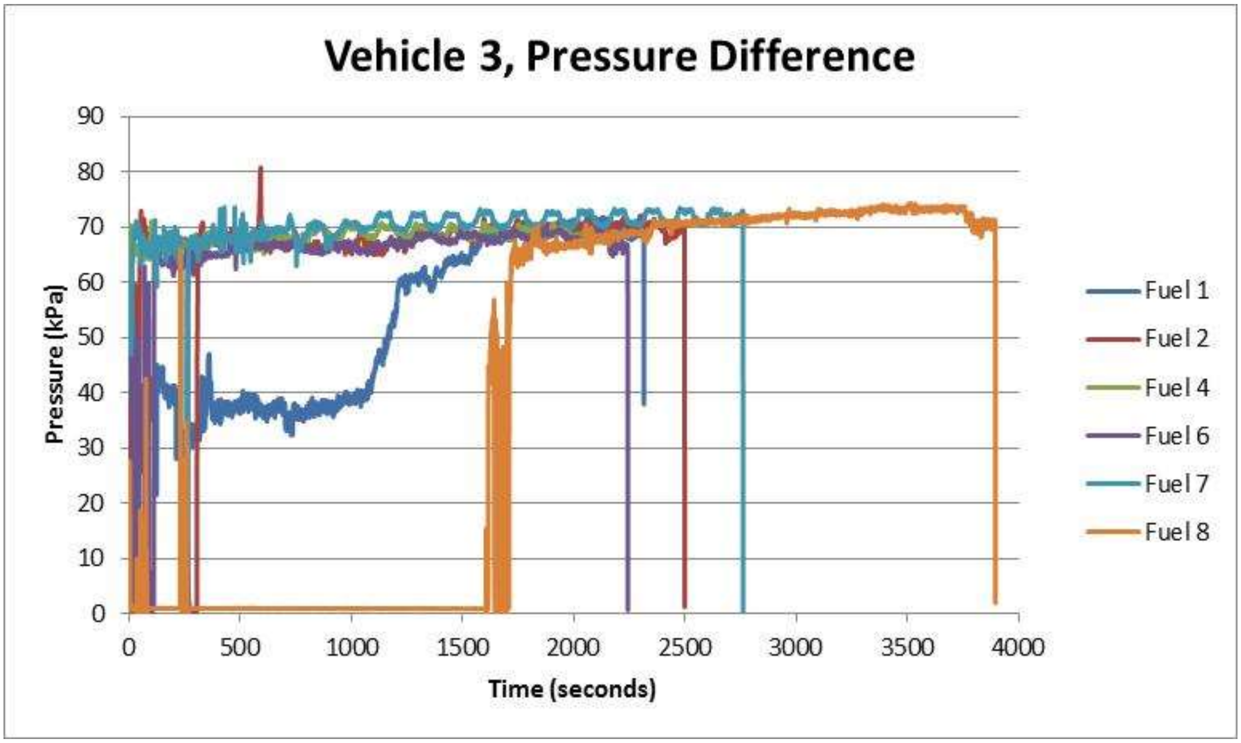


Figure 23: Pressure Differences for fuels in vehicle 3

Appendix A: Phase 1 Results Details

Analysis of Phase 1 Samples:

51 samples were received from Infineum in two batches. 48 samples were blends with one batch being labeled B and the other C. Also received were: one B100 (REG) and two diesel fuels (kerosene and low stream aromatic). Each sample was blended in different combinations of biodiesel (B100) blend level with different diesel fuels by Infineum. Some of the blends also contained an additive. The blends were sampled at Infineum and shipped to NREL for analysis. These samples were to be tested for Cloud Point (CP), final melting temperature (FMT), glycerin, and gas chromatography (GC) analysis for the diesel profile. These tests will be used to assess the amount of wax settling that occurred between the top and the bottom samples and what effect blend level, diesel fuel type, and additive had on the amount of wax settling.

Experimental:

CP and FMT were measured with a Phase Technology 70X Analyzer. CP was performed using D5773. FMT is an experiment that was designed specifically for NREL by Phase Technology. This test cools the sample rapidly until there is sufficient scattered light to indicate that crystals are present. At this point, the instrument will then heat the sample at 1.5°C/minute until all of the crystals have been re-dissolved into solution. This is indicated by the return of the signal to baseline where it will remain flat and constant. It has been noted[1] that there can be a significant difference between the CP and FMT indicating that a polymorphic phase transformation of saturated monoglycerides may be occurring.
Free and Total Glycerin were measured by Ion Chromatography using a Metrohm 871 Advanced Bioscan and a Metrohm 819 IC Detector. Total glycerin of REG B100 was performed by GC analysis using Modified D6584.
Diesel profiles were obtained using an Agilent 7890 GC/FID with a Supelco Equity 1 (30mx0.25mmx0.25um film thickness) column. The injection was performed in split mode with a 1uL injection and the inlet at 250°C with a 100:1 split ratio. Initial oven conditions were: 50°C, hold for 0 min, then ramp at 10°C to 325°C and hold for 5 min. The average velocity in the column is 29 cm/sec and the FID is set to 350°C.

Results:

Cloud Point and Final Melting Temperature Results:

Table 1 contains the data for the fuels used to blend the samples. Tables 2 and 3 contain all of the CP and FMT measurements for the blends received for B and C groups.

Table A1: CP and FMT Measurements for Fuels Used or Blending

Fuels Used for Blending				
Fuel	**CP**	**FMT**	**Total Glycerin**	**Free Glycerin**
Low Aromatic Stream	-5.6	-0.9	NA	NA
Kerosene	-49.6	-43.2	NA	NA
REG	1.0	4.1	0.059%	0.007%

Table A2: CP and FMT measurements for B Group Blends:

Sample		CP	FMT	CP(bottom-top)	FMT(bottom-top)	B100 %	Diesel	Additive
B1	Top	-26.2	-23.7	-10.3	-10.6	0	40% ULSD:60% Kero	None
	Bottom	-15.9	-13.1					
B2	Top	-26.8	-23.5	-9.6	-13.4	0	40% ULSD:60% Kero	A
	Bottom	-17.2	-10.1					
B4	Top	-23.4	-15.3	-2.1	-0.8	0	40% ULSD:60% Kero	B
	Bottom	-21.3	-14.5					
B6	Top	-24.4	-20.8	-12.6	-16.0	5	38% ULSD:57% Kero	None
	Bottom	-11.8	-4.8					
B7	Top	-24.4	-17.2	-8.3	-10.4	5	38% ULSD:57% Kero	A
	Bottom	-16.1	-6.8					
B9	Top	-24.1	-19.8	-6.1	-12.4	5	38% ULSD:57% Kero	B
	Bottom	-18.0	-7.4					
B11	Top	-22.4	-19.2	-15.8	-16.9	10	36% ULSD:54% Kero	None
	Bottom	-6.6	-2.3					
B12	Top	-22.3	-17.6	-14.4	-14.2	10	36% ULSD:54% Kero	A
	Bottom	-7.9	-3.4					
B14	Top	-21.5	-16.2	-13.6	-11.4	10	36% ULSD:54% Kero	B
	Bottom	-7.9	-4.8					
B31	Top	-23.0	-18.6	-12.4	-14.4	0	24% ULSD:16% Lo Aro:60% Kero	None
	Bottom	-10.6	-4.2					
B32	Top	-21.8	-17.4	-10.4	-12.8	0	24% ULSD:16% Lo Aro:60% Kero	A
	Bottom	-11.4	-4.6					
B34	Top	-21.8	-16.1	-6.1	-9.2	0	24% ULSD:16% Lo Aro:60% Kero	B
	Bottom	-15.7	-6.9					
B36	Top	-19.2	-15.2	-3.5	-6.0	5	22.8% ULSD:15.2% Lo Aro:57% Kero	None
	Bottom	-15.7	-9.2					
B38	Top	-19.1	-13.5	-7.0	-6.4	5	22.8% ULSD:15.2% Lo Aro:57% Kero	A
	Bottom	-12.1	-7.1					
B40	Top	-18.5	-12.2	-1.8	-1.3	5	22.8% ULSD:15.2% Lo Aro:57% Kero	B
	Bottom	-16.7	-10.9					

Table A3: CP and FMT measurements for C Group Blends:

Sample		CP	FMT	CP(bottom-top)	FMT(bottom-top)
C1	Top	-22.8	-18.5	-3.6	-2.7
	Bottom	-19.2	-15.8		
C2	Top	-25.5	-21.0	-12.2	-15.8
	Bottom	-13.3	-5.2		
C4	Top	-24.2	-17.6	-3.8	-6.3
	Bottom	-20.4	-11.3		
C6	Top	-20.4	-16.9	-12.6	-11.9
	Bottom	-7.8	-5.0		
C7	Top	-23.4	-18.9	-18.2	-18.2
	Bottom	-5.2	-0.7		
C9	Top	-21.7	-15.8	-5.6	-8.9
	Bottom	-16.1	-6.9		
C11	Top	-18.2	-14.6	-12.9	-19.8
	Bottom	-5.3	5.2		
C12	Top	-20.5	-16.5	-15.6	-17.1
	Bottom	-4.9	0.6		
C14	Top	-19.1	-13.6	-7.3	-9.6
	Bottom	-11.8	-4.0		

In Tables 2 and 3, a large difference between the measured CP and FMT in the Top versus the Bottom samples would be indicative of wax settling. Samples that demonstrated a 10°C or more increase between the Top and Bottom for CP and/or FMT are highlighted in red. This large of a disparity between the measured values would indicate that wax settling is occurring. The type of diesel fuel used for blending along with the amount of Soy B100 present and the additive type are also listed. In Table 2, there are several conclusions that can be drawn from the groups of samples run:

- For the 40% ULSD:60% Kerosene with no B100 blended, additive B reduced the amount of wax settling significantly when compared to using no additive or additive A.
- For 38% ULSD:57% Kerosene with 5% Soy B100, there appears to be a fair amount of wax settling in all cases, however it is slightly reduced with both the A and B additives.
- For 36% ULSD:54% Kerosene with 10% Soy B100, neither additive A nor B seem to reduce the amount of wax settling to a large extent.
- For 24% ULSD:16% Low Aromatic:60% Kerosene with no B100, it appears that additive B once again reduced the amount of wax settling when compared to using no additive or additive A.
- In the case of 22.8% ULSD:15.2% Low Aromatic: 57% Kerosene with 5% B100, there did not appear the be as much wax settling in these samples as compared to the other blends with B100. Additive B reduced the amount of wax settling in these samples to a moderate extent when compared to using no additive or additive A.

In general, additive B appeared to reduce the amount of wax settling to the greatest extent. There did not appear to be a correlation between the amount of wax settling and the type of diesel fuel used. Even without B100 present, there was wax settling occurring as evidenced by the large differences in CP and FMT between the Top and the Bottom samples. Once the B100 blend level reached 10%, additive B did not appear to be as effective at reducing the amount of wax settling.

For samples received in the C group, information on the blend level, diesel fuel used, and the additive was not received. If these samples were to follow the same organization as those received in the B group, then it would appear that the second additive (possibly additive B) was most effective at reducing the amount of wax settling. This would be in agreement with the results from the B group of samples. Analysis of the GC diesel profile shows that the organization is most likely similar in organization because the first three samples (C1, C2, and C4) do not contain B100. The next six samples in the set have peaks that correspond to FAME peaks in the GC profile suggesting that the next set of three samples (C6, C7, and C9) would be a B5 blend and that the last three samples (C11, C12, and C14) would be B10 blends. The peaks corresponding to FAME in the GC profile are larger in the third (C11, C12, and C14) set also suggesting that these are B10 blends. Without definitive information on the sample compositions, it is difficult to draw accurate conclusions for this set of samples though they do appear to follow the same organization.

Ion Chromatography Results:

Tables 4 and 5 contain the IC results for all of the B and C group blend samples.

Table A4: IC Results for B Group Blend Samples

Sample		Free Glycerin (ppm)	Total Glycerin (ppm)	B100 %	Diesel	Additive
B1	Top	ND	ND	0	40% ULSD:60% Kero	None
	Bottom	ND	ND			
B2	Top	ND	ND	0	40% ULSD:60% Kero	A
	Bottom	ND	ND			
B4	Top	ND	ND	0	40% ULSD:60% Kero	B
	Bottom	ND	ND			
B6	Top	ND	18.07	5	38% ULSD:57% Kero	None
	Bottom	1.62	48.76			
B7	Top	ND	18.51	5	38% ULSD:57% Kero	A
	Bottom	1.65	34.60/37.23			
B9	Top	ND	21.2	5	38% ULSD:57% Kero	B
	Bottom	1.38	35.66			
B11	Top	1.30	53.40	10	36% ULSD:54% Kero	None
	Bottom	6.94	107.30			
B12	Top	0.95	46.80	10	36% ULSD:54% Kero	A
	Bottom	6.60	102.02			
B14	Top	1.12	56.02	10	36% ULSD:54% Kero	B
	Bottom	7.72	87.10			
B31	Top	ND	ND	0	24% ULSD:16% Lo Aro:60% Kero	None
	Bottom	ND	ND			
B32	Top	ND	ND	0	24% ULSD:16% Lo Aro:60% Kero	A
	Bottom	ND	ND			
B34	Top	ND	ND	0	24% ULSD:16% Lo Aro:60% Kero	B
	Bottom	ND	ND			
B36	Top	ND	22.47	5	22.8% ULSD:15.2% Lo Aro:57% Kero	None
	Bottom	2.40	45.18			
B38	Top	ND	19.72	5	22.8% ULSD:15.2% Lo Aro:57% Kero	A
	Bottom	2.32/2.856	49.83			
B40	Top	1.42	27.07	5	22.8% ULSD:15.2% Lo Aro:57% Kero	B
	Bottom	ND	35.69			

As with the increase of the CP and the FMT in the Top versus the Bottom samples, we might expect to see the glycerin settling out into the some of the Bottom samples. For the samples that did not contain any B100, there should be no measureable glycerin. Looking at the IC results, we see that there is more glycerin in the Bottom versus the Top samples as expected. This may be one of the reasons for the increase in the CP and FMT that was observed. It doesn't appear that the additives have as much of an effect on the amount of glycerin that settles out in Bottom samples however. Additive B does appear to reduce the amount of glycerin in the Bottom samples when compared with the Bottom samples for additive A and no additive used however.

For the C group of samples, we if we assume that the general organization used for the B group still applies (which GC supports), then we would assume that the lack of glycerin that is present in C1, C2, and C4 would indicate that these samples do not contain any B100 (which is in agreement with the GC). Once again, the Bottom samples contain more glycerin than the Top samples as would be expected. It would also appear that neither additive A nor B prevent the glycerin from settling to the bottom of the samples.

Table A5: IC Results for Group C Blend Samples

Sample		Free Glycerin (ppm)	Total Glycerin (ppm)
C1	Top	ND	ND
	Bottom	ND	ND
C2	Top	ND	ND
	Bottom	ND	ND
C4	Top	ND	ND
	Bottom	ND	ND
C6	Top	ND	18.66
	Bottom	1.19	40.77/40.76
C7	Top	ND	11.20
	Bottom	1.97	57.43
C9	Top	ND	13.78
	Bottom	2.30/2.20	36.68
C11	Top	1.45	52.24
	Bottom	5.34	101.31
C12	Top	ND	50.23/48.64
	Bottom	5.42	113.22
C14	Top	ND	56.87
	Bottom	5.75	97.42

GC Analysis of Diesel Profile:

Analysis of the GC profile allows us to look for the presence of heavier hydrocarbons in the Bottom versus the Top samples. As can clearly be seen in the overlay chromatograms in Figure 1, the Bottom sample (Red) contains a higher percentage of longer chain hydrocarbons than the Top (Black) sample. This would also explain the increase of the CP and FMT in the Bottom versus the Top samples. Analysis of all of the chromatograms indicates that these follow the same trends as was noted in the CP and FMT analysis. The Bottom samples contain higher amounts of heavier hydrocarbons which would lead to the increase in CP and FMT observed. Additive B appears to have the lowest amounts of the heavier hydrocarbons. In Figure 2, the black chromatogram is the sample with no additive, the red is additive A and the blue is additive B. As can be seen, the black (no additive) sample has the largest concentration of heavy hydrocarbons followed by the red (additive A) and the blue (additive B). The C group of samples also follows this trend.

Figure A1: Overlay of B4 Top and Bottom Samples

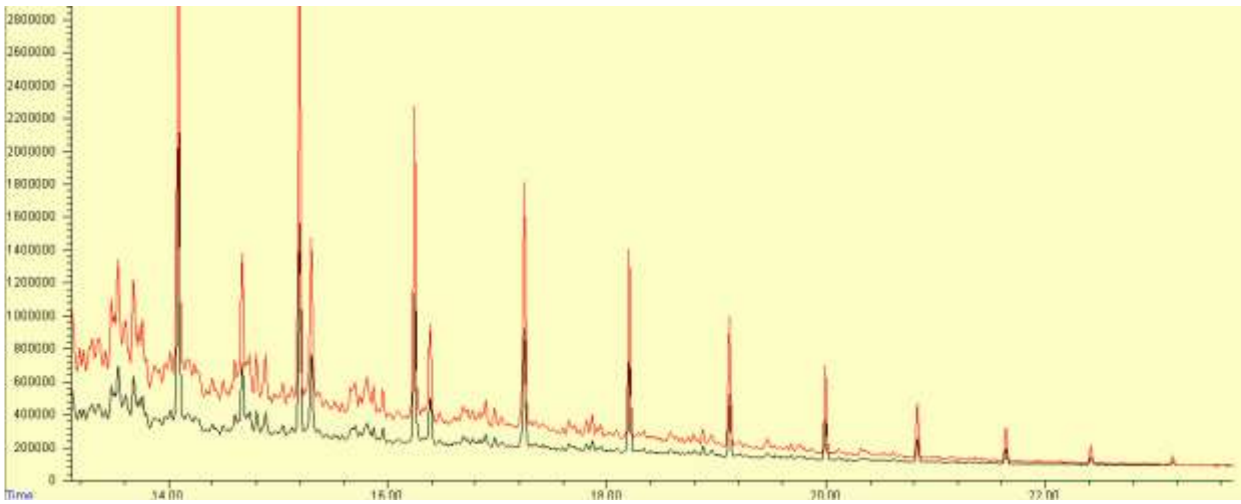
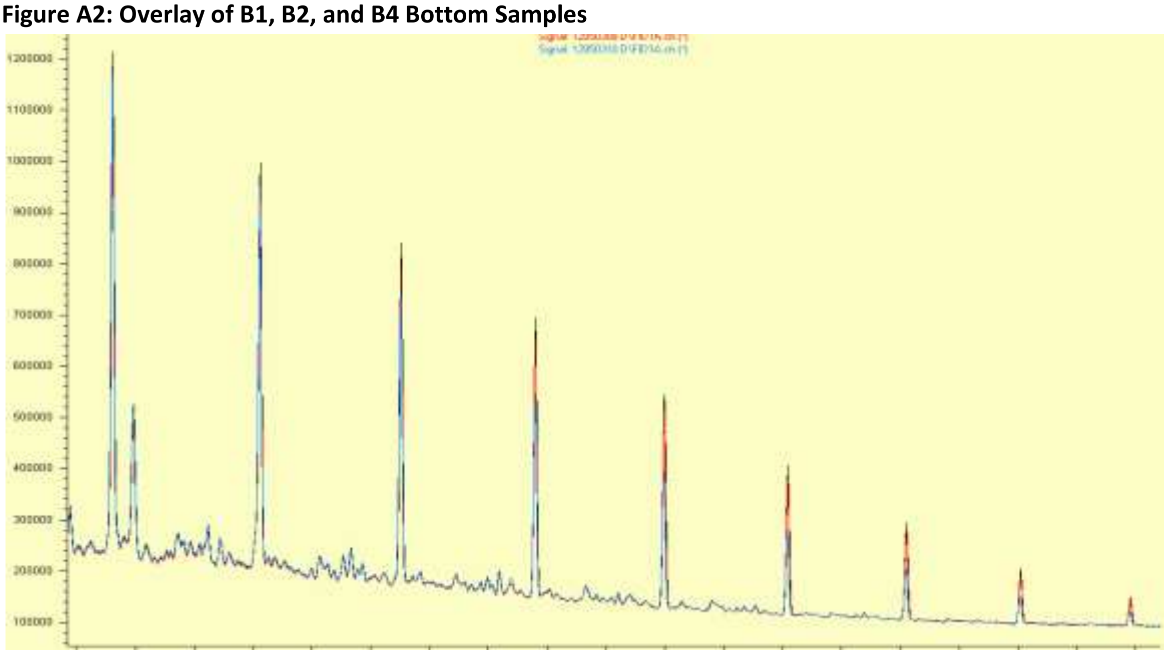


Figure A2: Overlay of B1, B2, and B4 Bottom Samples



Summary:

Additive B appears to give the best results as far as preventing wax settling based on the CP, FMT, and GC results. While it was not as effective at higher percentages of B100, it was effective at preventing wax settling especially in the pure diesel blends. The GC profile data supports the CP and FMT findings as far as a larger percentage of the heavier hydrocarbons being found in the samples that give higher CP and FMT measurements in the Bottom versus the Top samples. Neither additive appeared very effective at reducing the amount of glycerin that settled out into the Bottom samples. We were able to confirm the presence of B100 in the blends through the GC profile.

[1] Effect of Saturated Monoglyceride Polymorphism on Low-Temperature Performance of Biodiesel," Gina M. Chupka, Janet Yanowitz, Gordon Chiu, Teresa Alleman and Robert McCormick, *Energy Fuels,* **2011**, *25*, 398-405.

Appendix B: Vehicle Diurnal Cycle and Operability Results

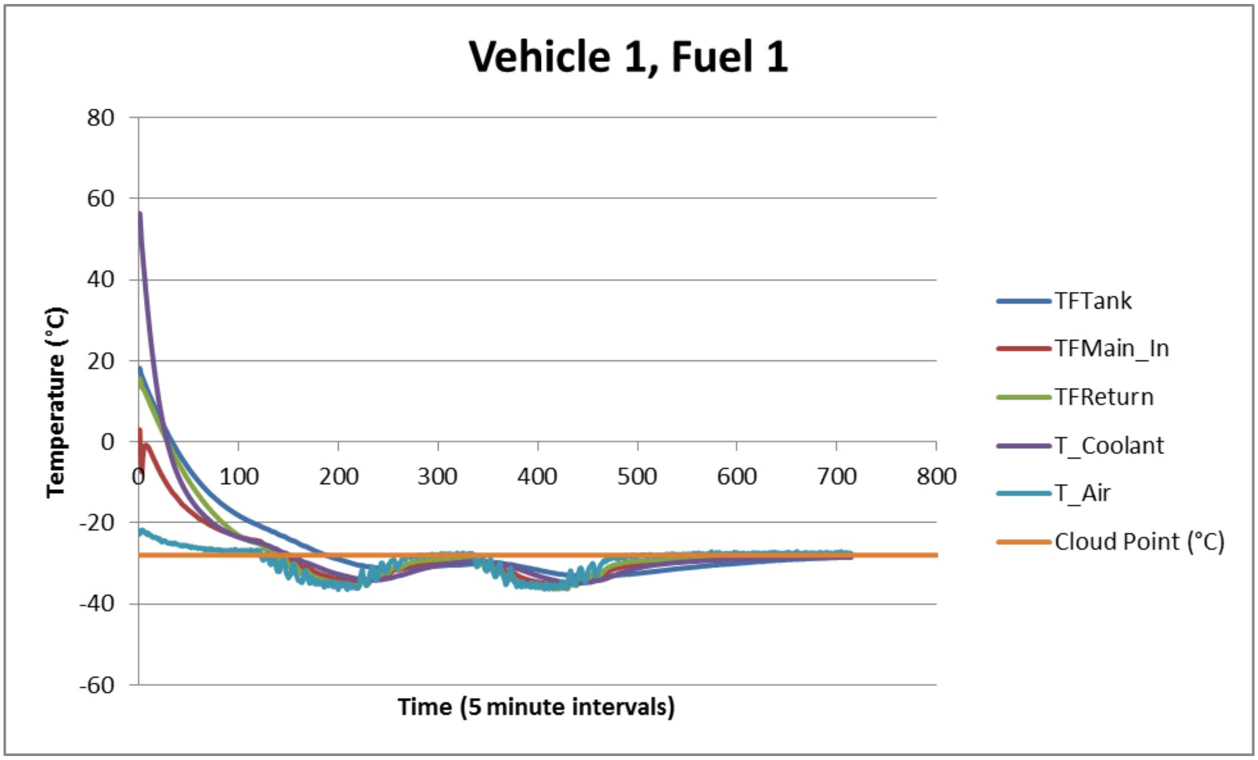


Figure B1: Diurnal Cold Soak of Fuel 1 Vehicle 1

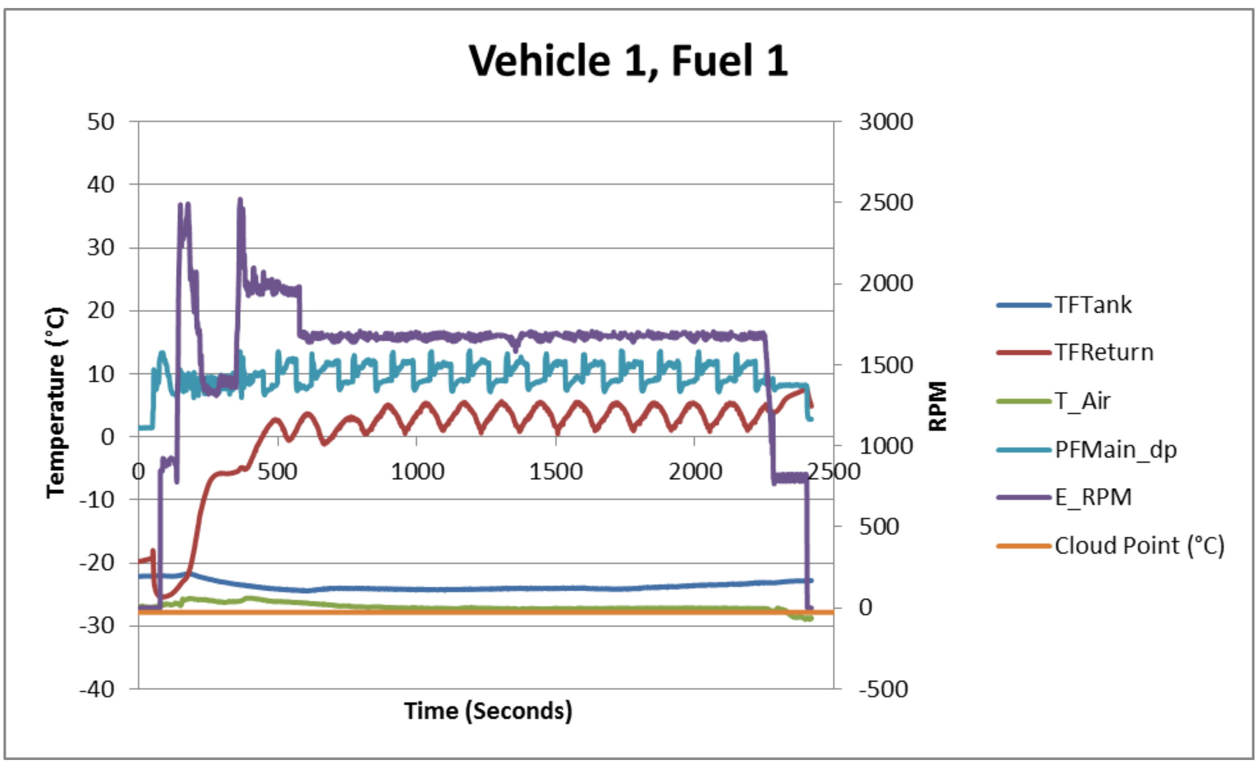


Figure B2: Select Data Series During AWCD Testing of Fuel 1 Vehicle 1

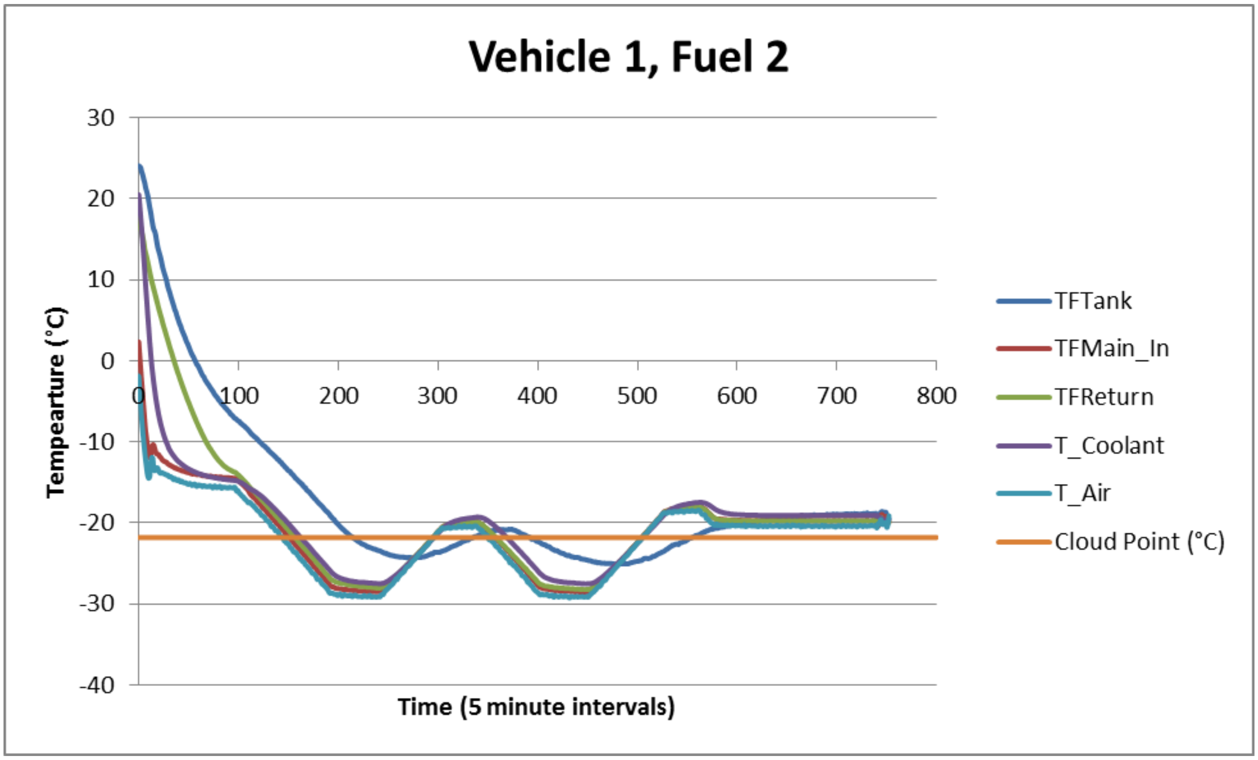


Figure B3: Diurnal Cold Soak of Fuel 2 Vehicle 1

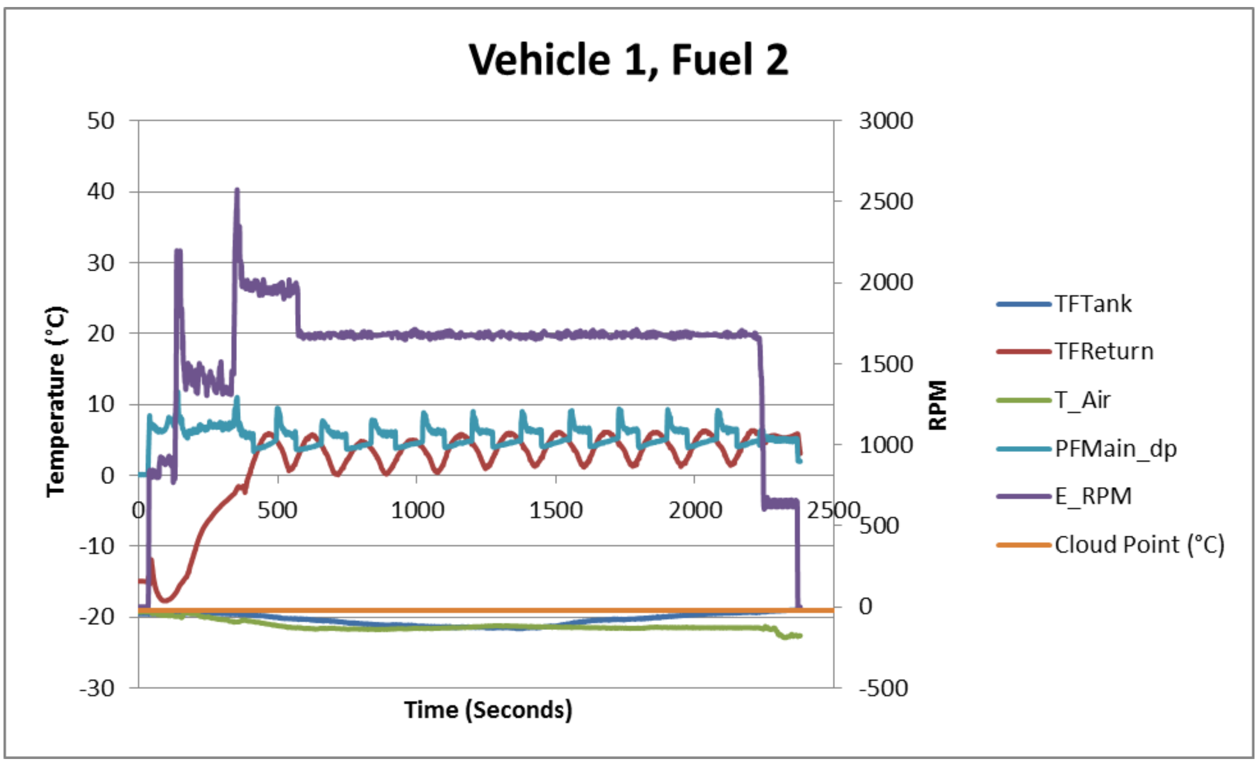


Figure B4: Select Data Series During AWCD Testing of Fuel 2 Vehicle 1

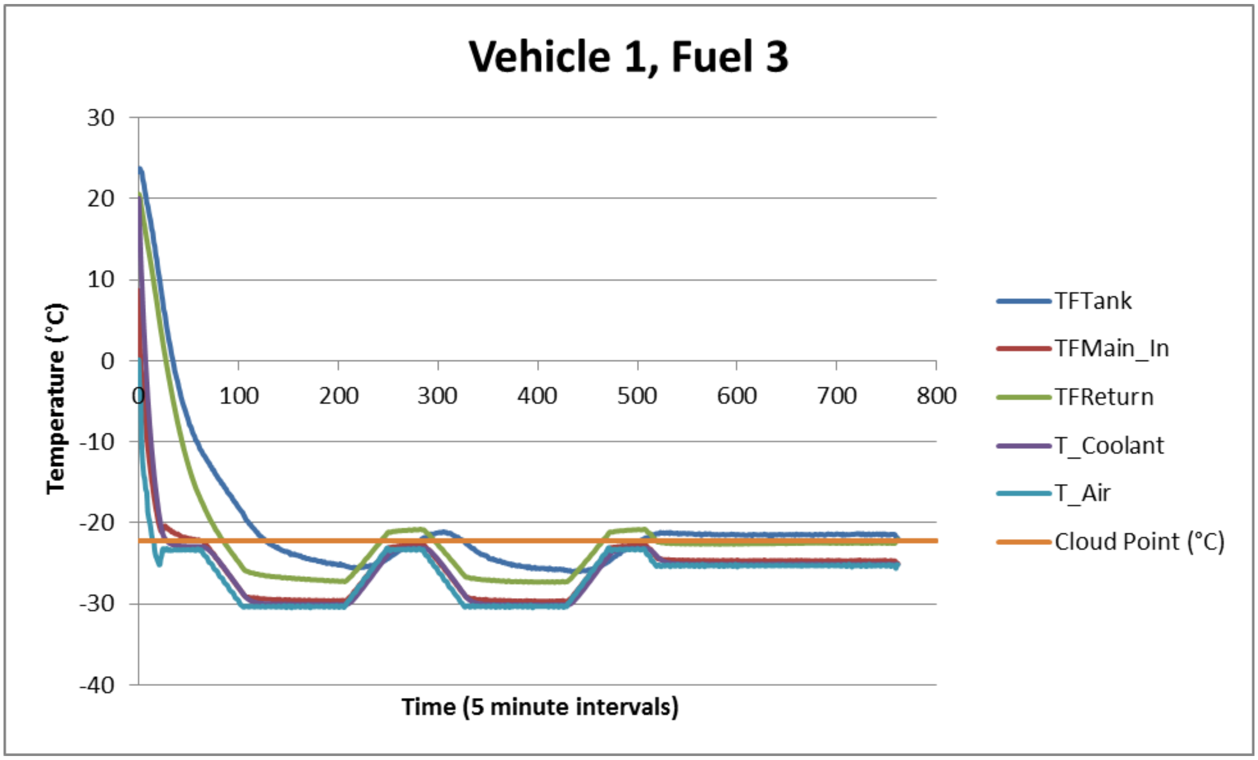


Figure B5: Diurnal Cold Soak of Fuel 3 Vehicle 1

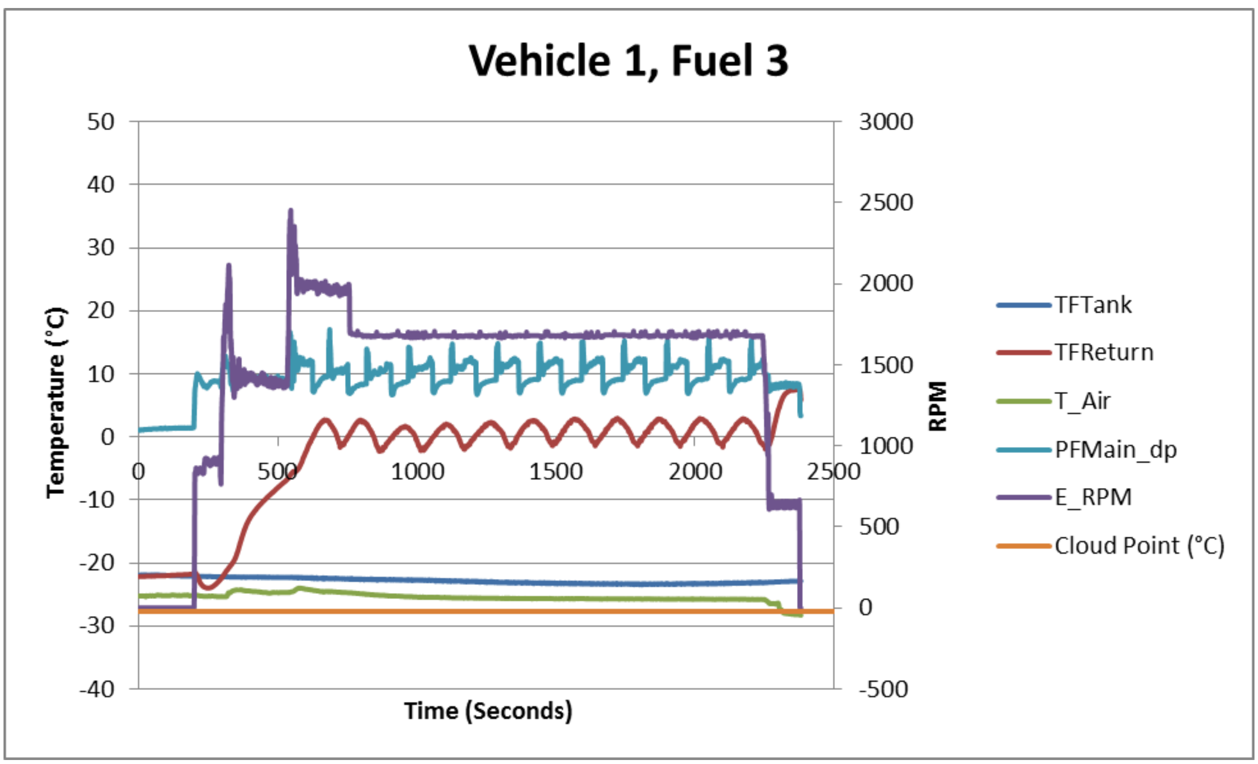


Figure B6: Select Data Series During AWCD Testing of Fuel 3 Vehicle 1

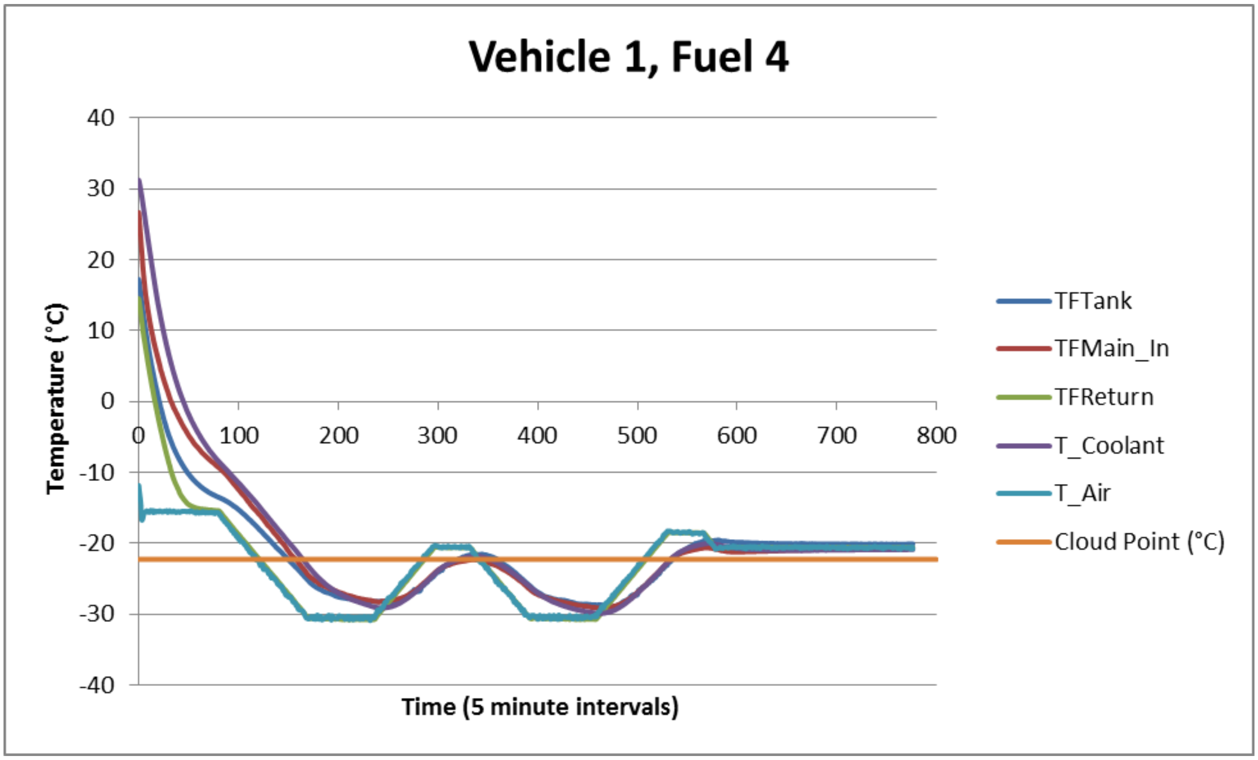


Figure B7: Diurnal Cold Soak of Fuel 4 Vehicle 1

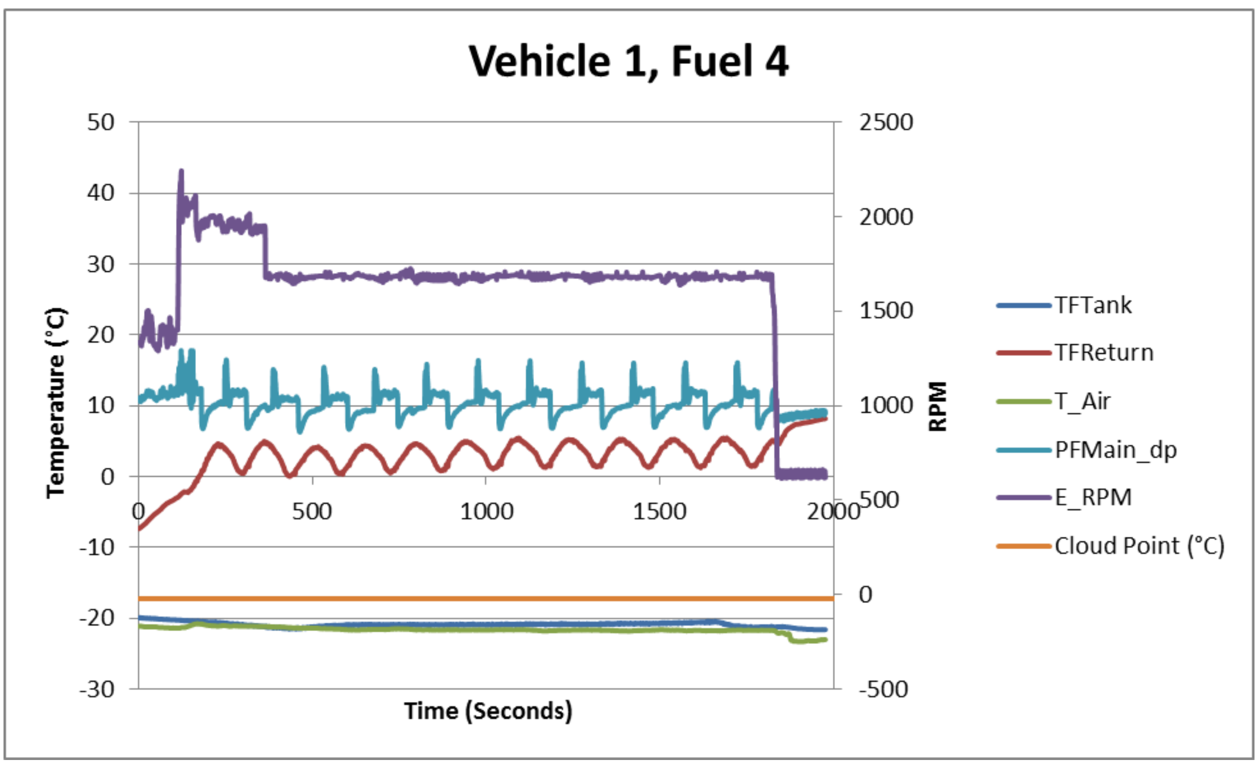


Figure B8: Select Data Series During AWCD Testing of Fuel 4 Vehicle 1

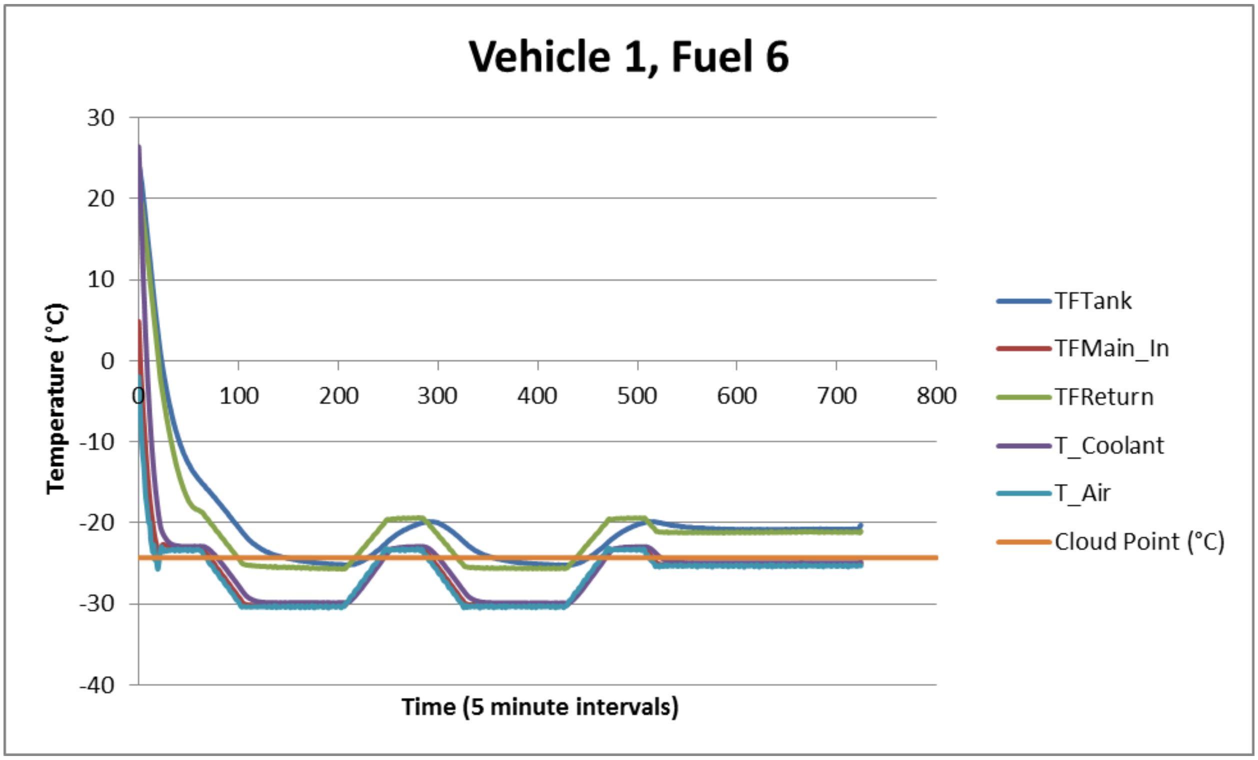


Figure B9: Diurnal Cold Soak of Fuel 6 Vehicle 1

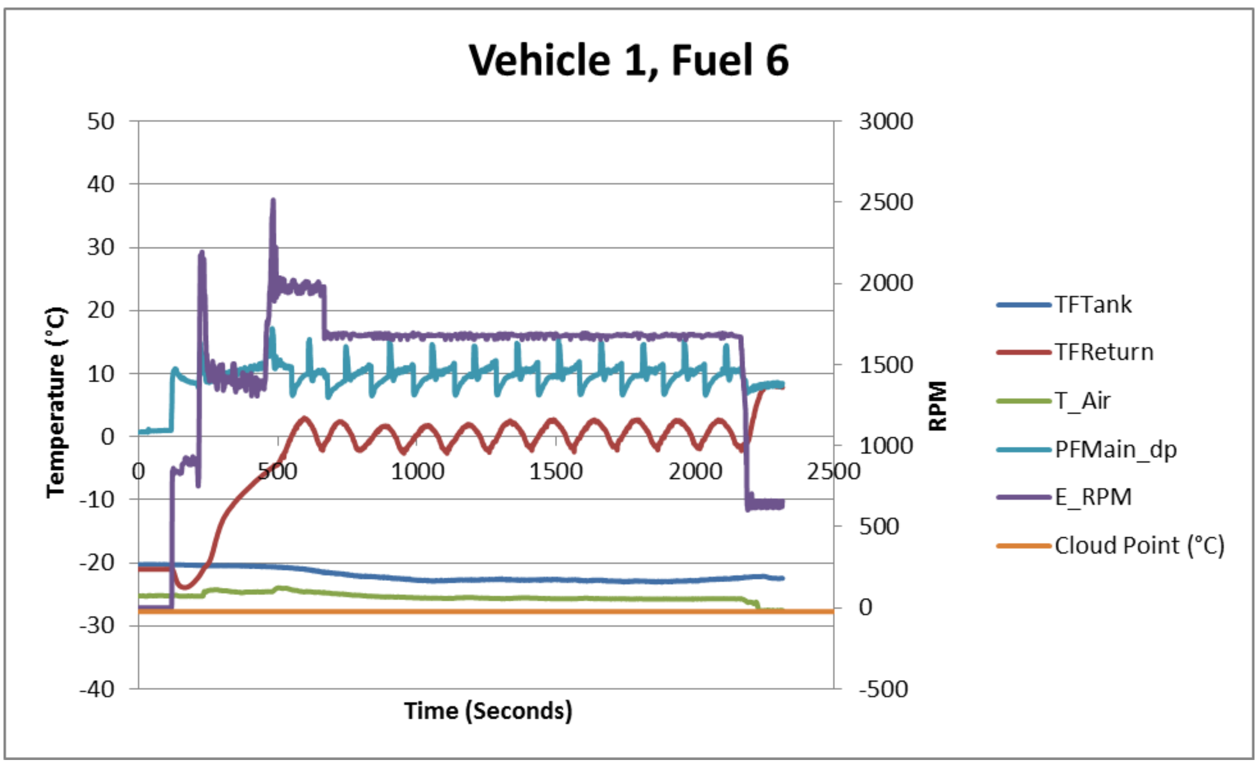


Figure B10: Select Data Series During AWCD Testing of Fuel 6 Vehicle 1

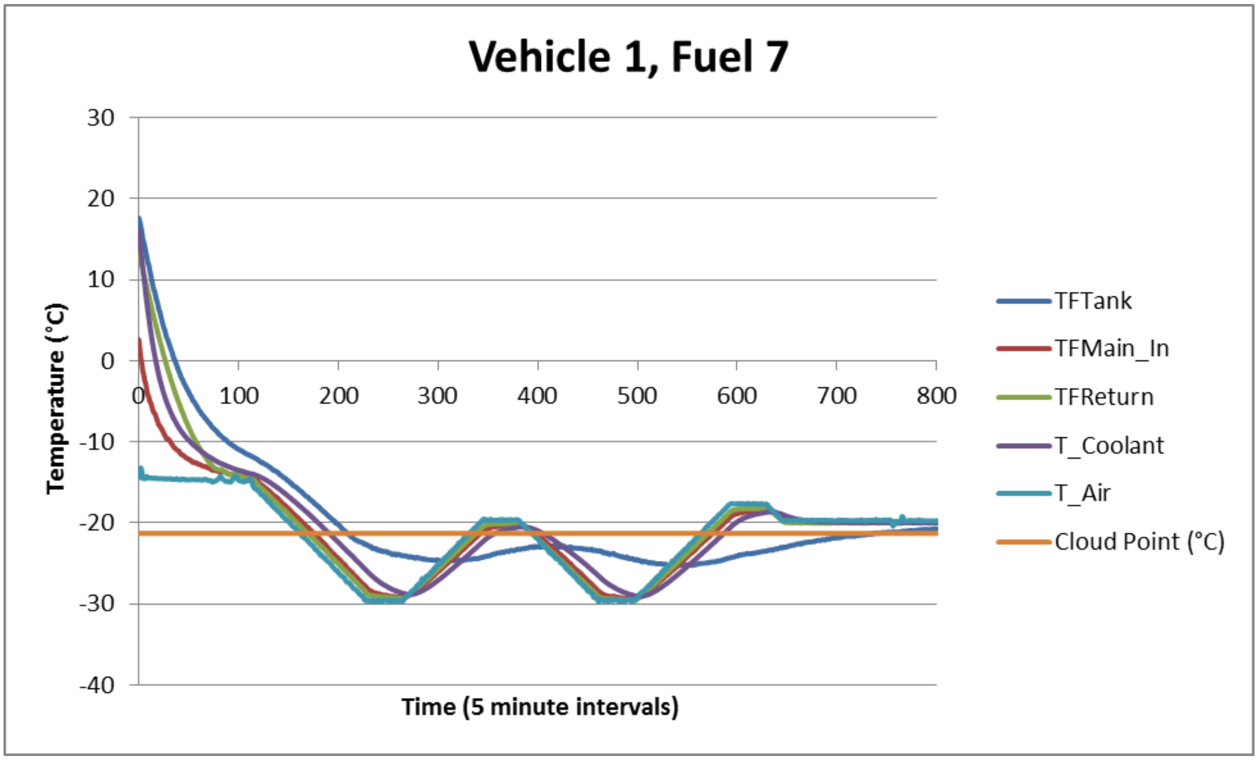


Figure B11: Diurnal Cold Soak of Fuel 7 Vehicle 1

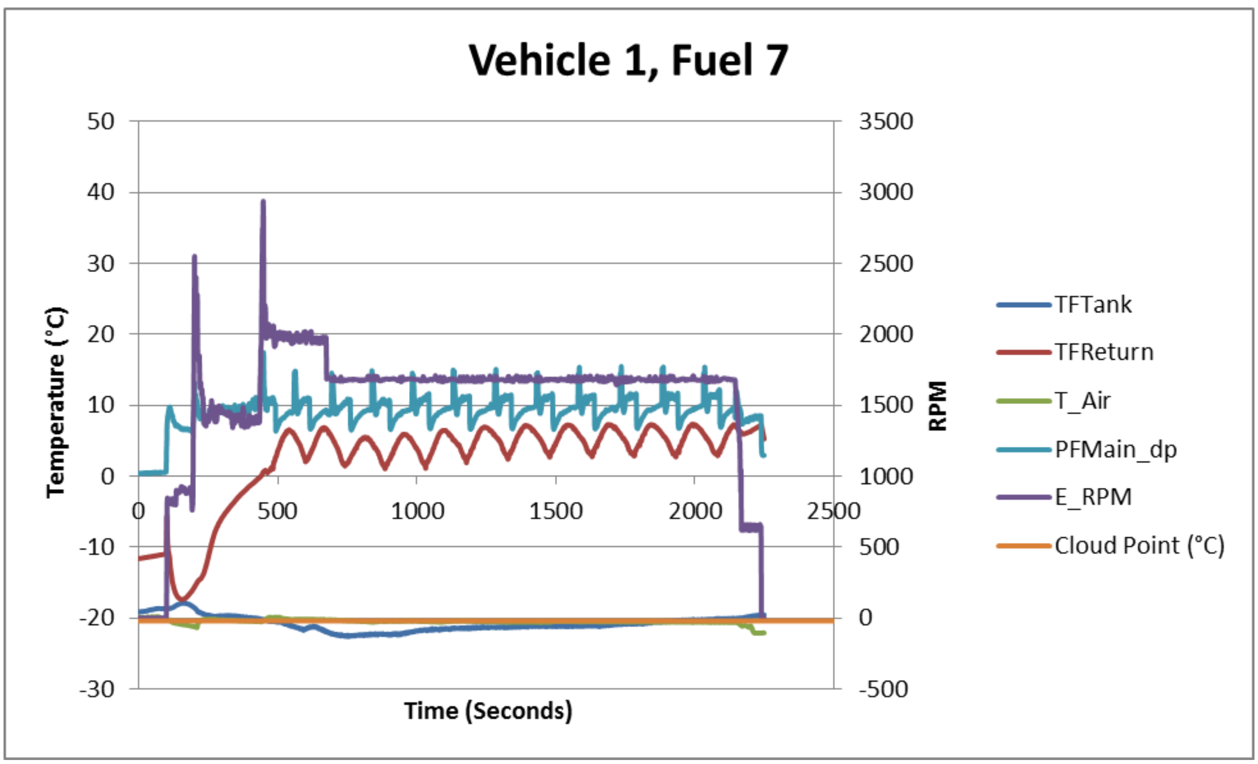


Figure B12: Select Data Series During AWCD Testing of Fuel 7 Vehicle 1

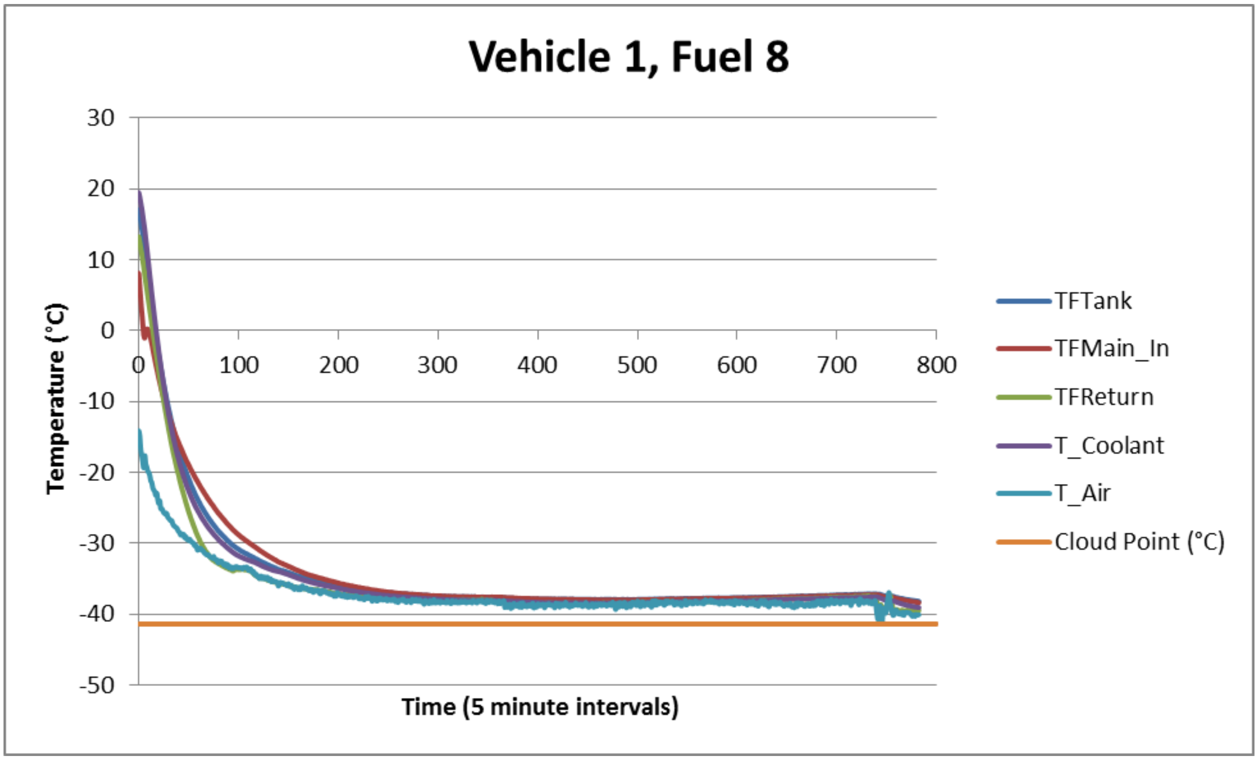


Figure B13: Diurnal Cold Soak of Fuel 8 Vehicle 1

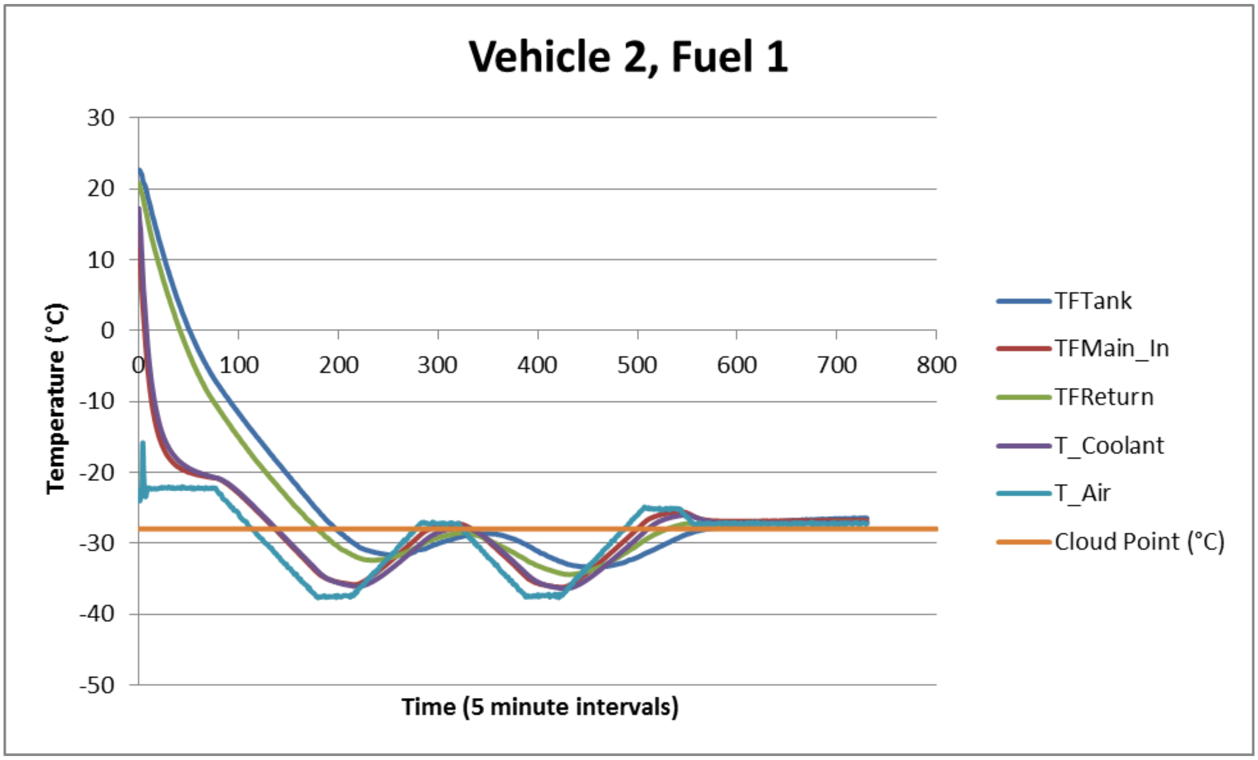


Figure B14: Diurnal Cold Soak of Fuel 1 Vehicle 2

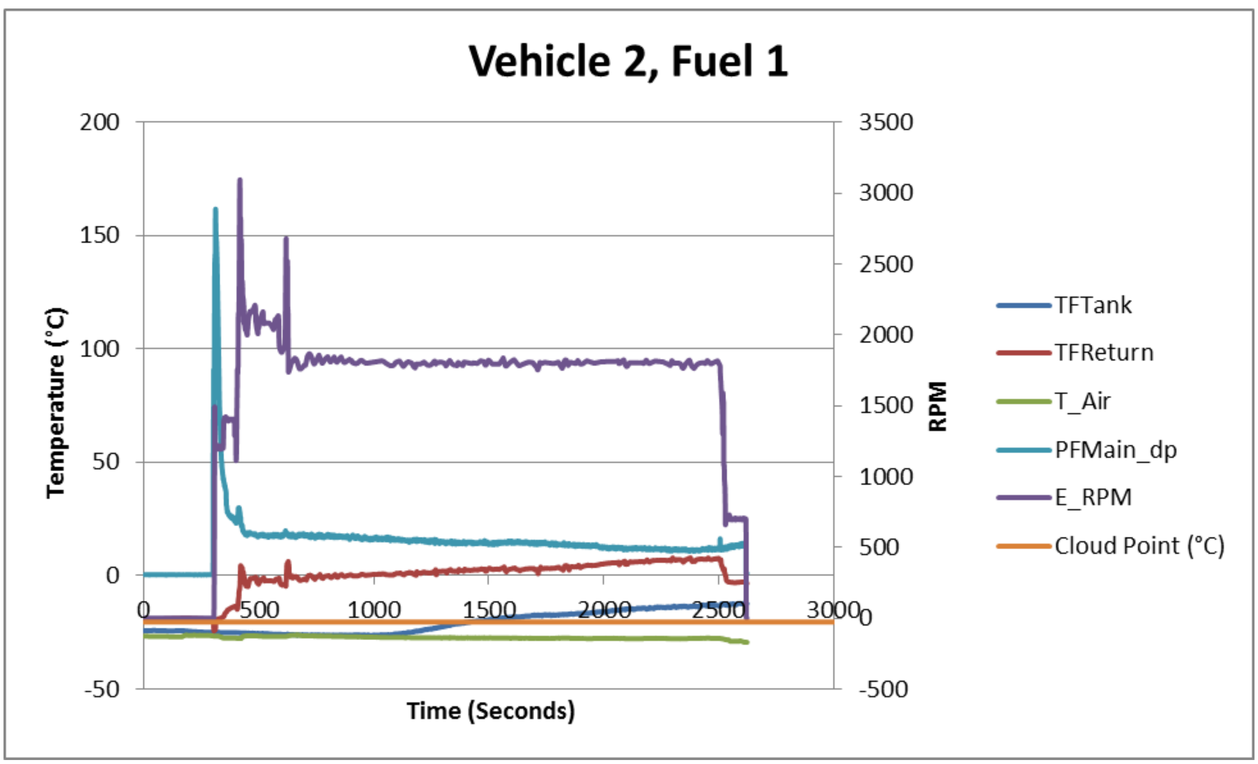


Figure B15: Select Data Series During AWCD Testing of Fuel 1 Vehicle 2

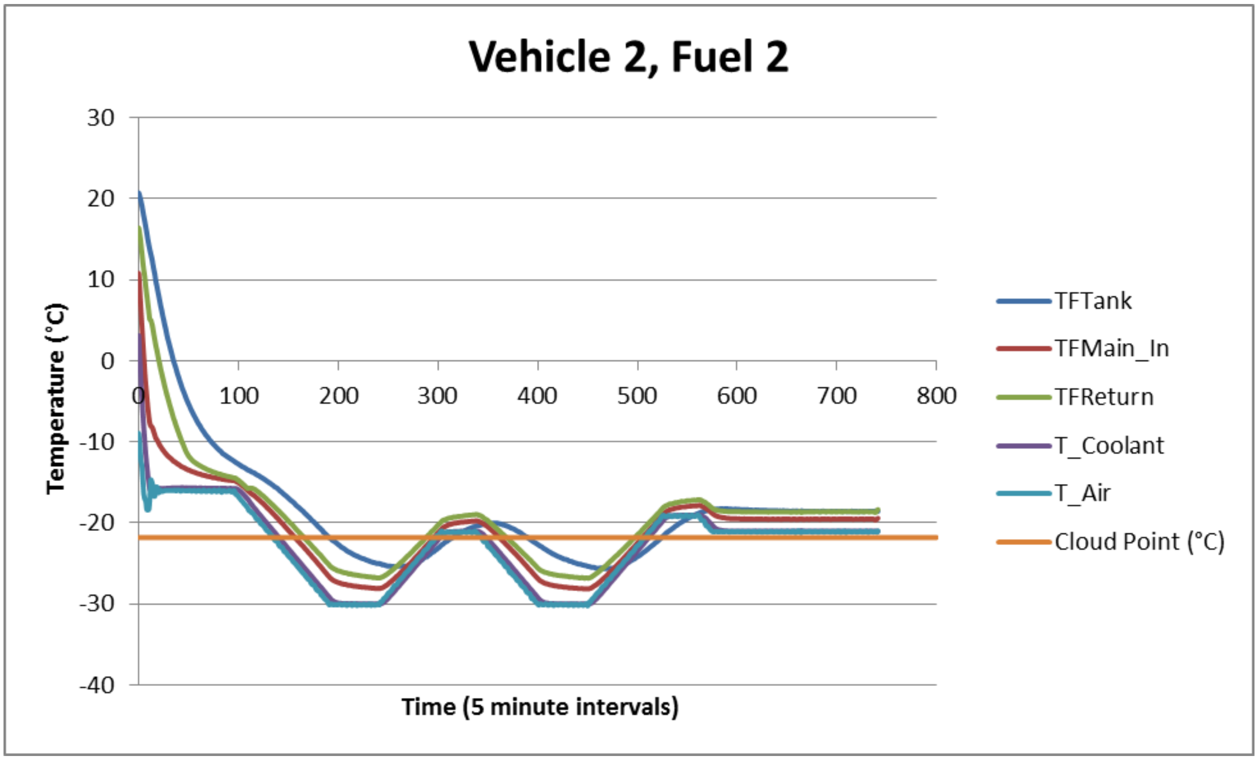


Figure B16: Diurnal Cold Soak of Fuel 2 Vehicle 2

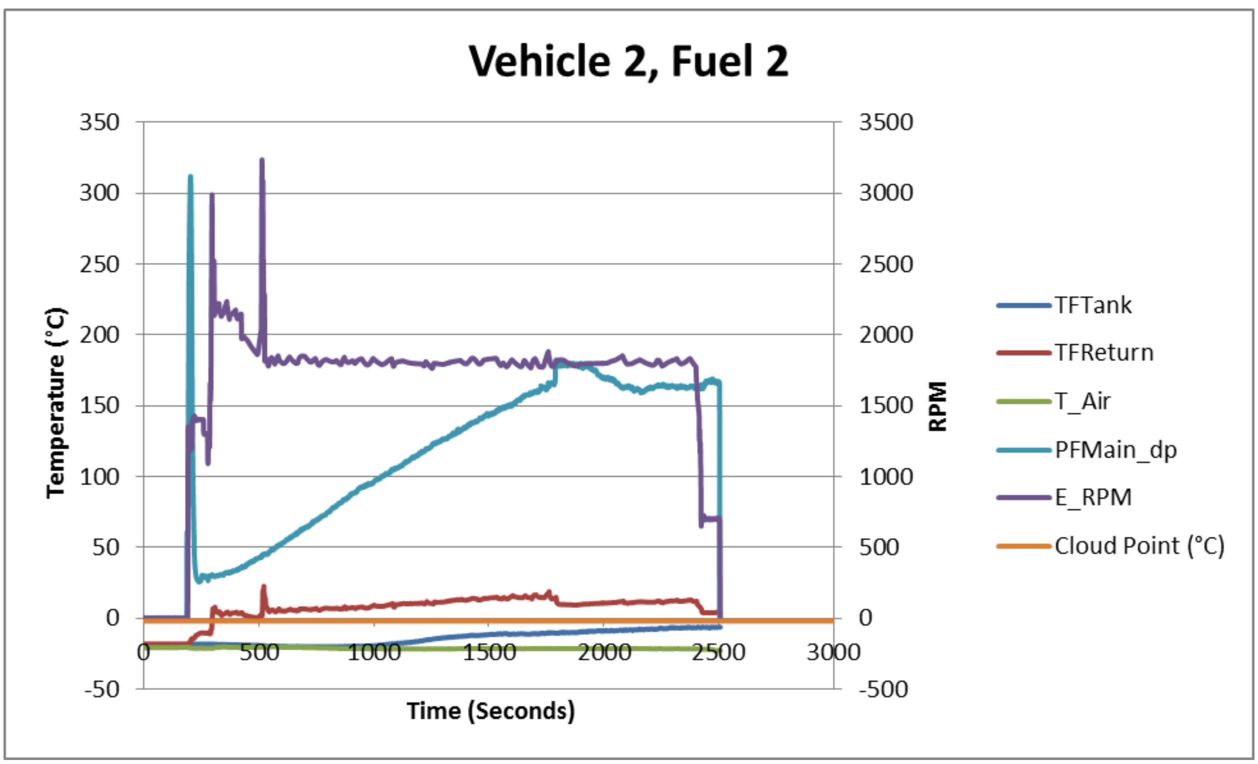


Figure B17: Select Data Series During AWCD Testing of Fuel 2 Vehicle 2

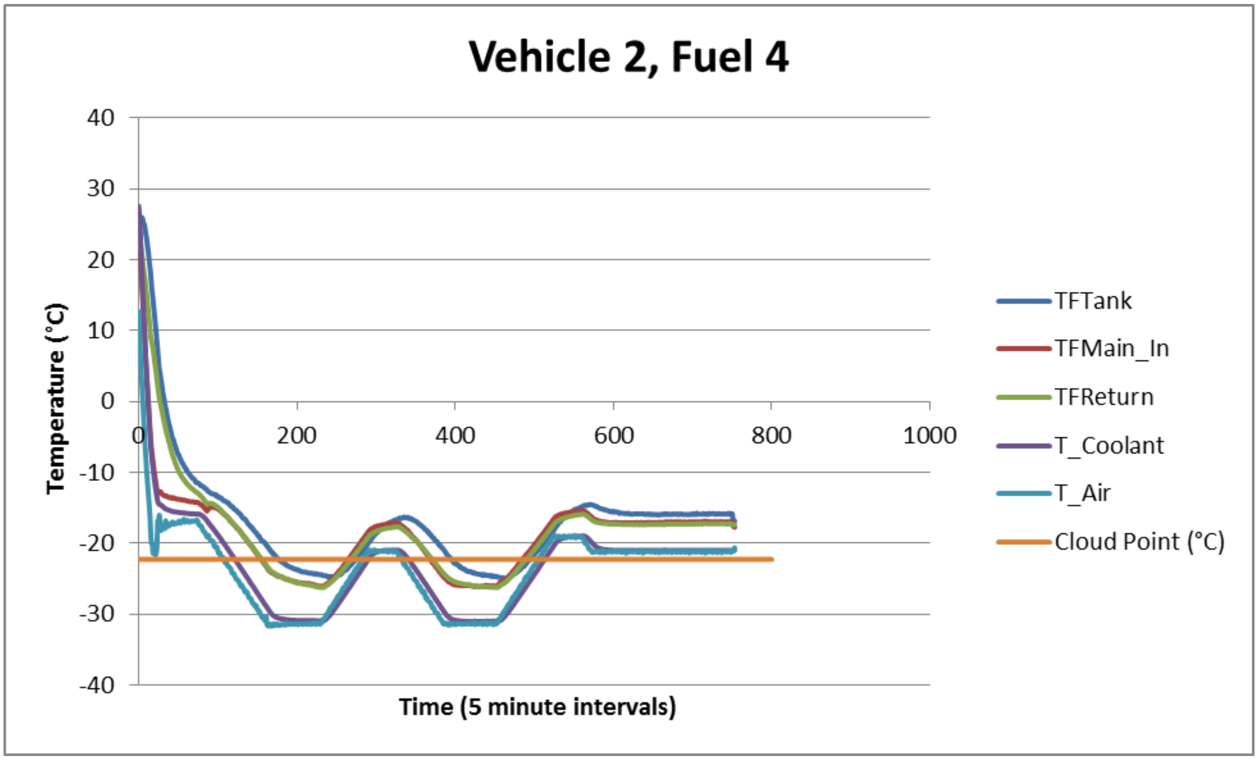


Figure B18: Diurnal Cold Soak of Fuel 4 Vehicle 2

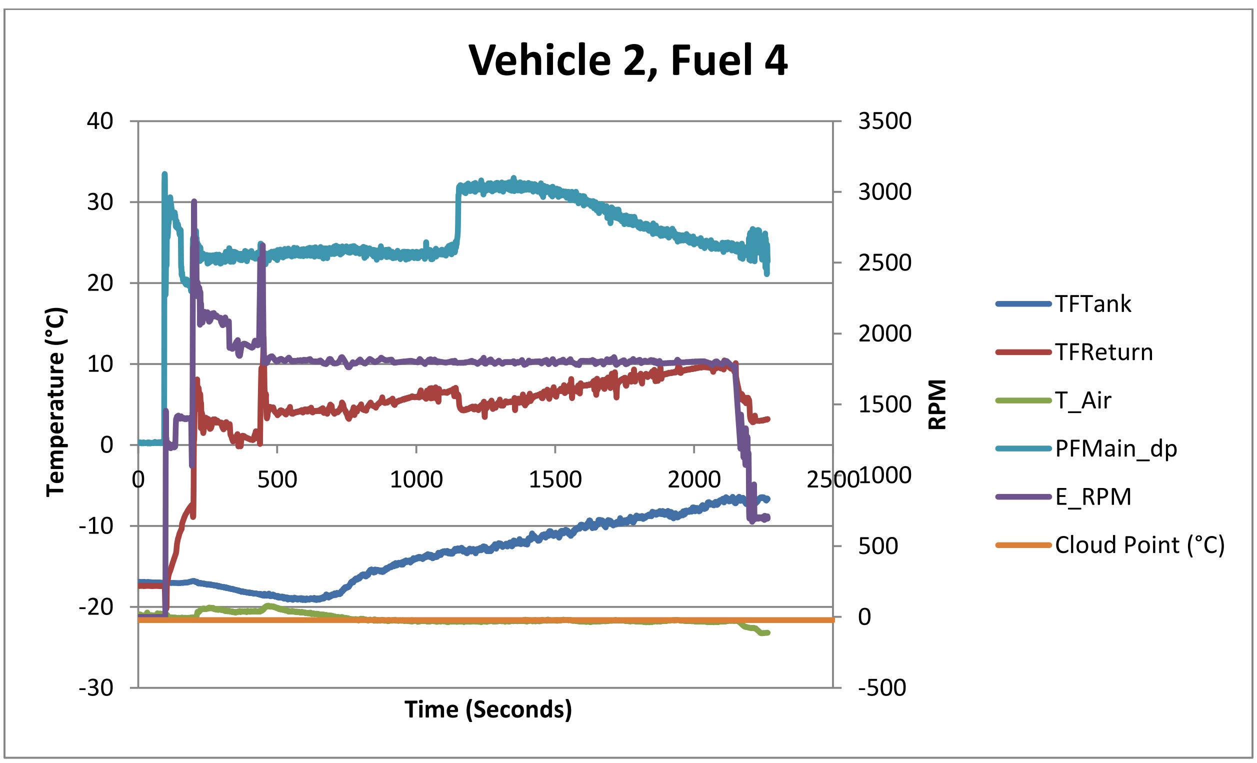


Figure B19: Select Data Series During AWCD Testing of Fuel 4 Vehicle 2

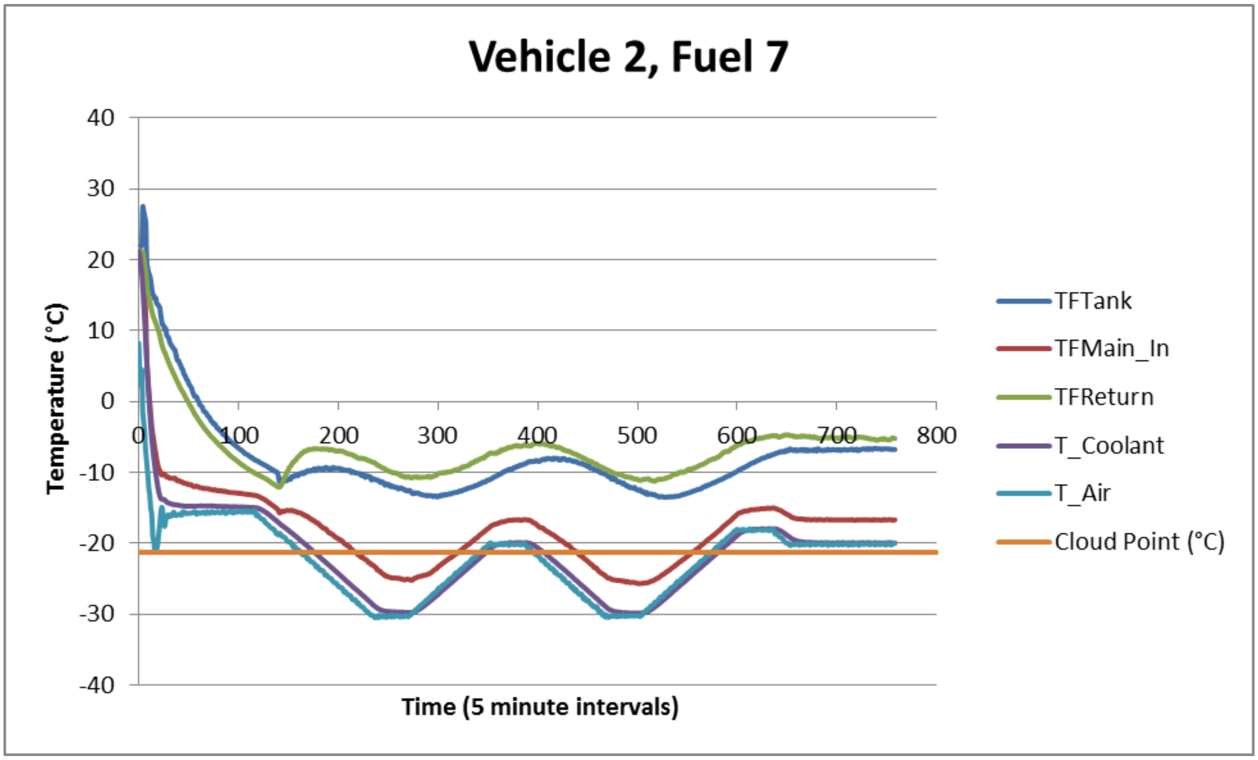


Figure B20: Diurnal Cold Soak of Fuel 7 Vehicle 2

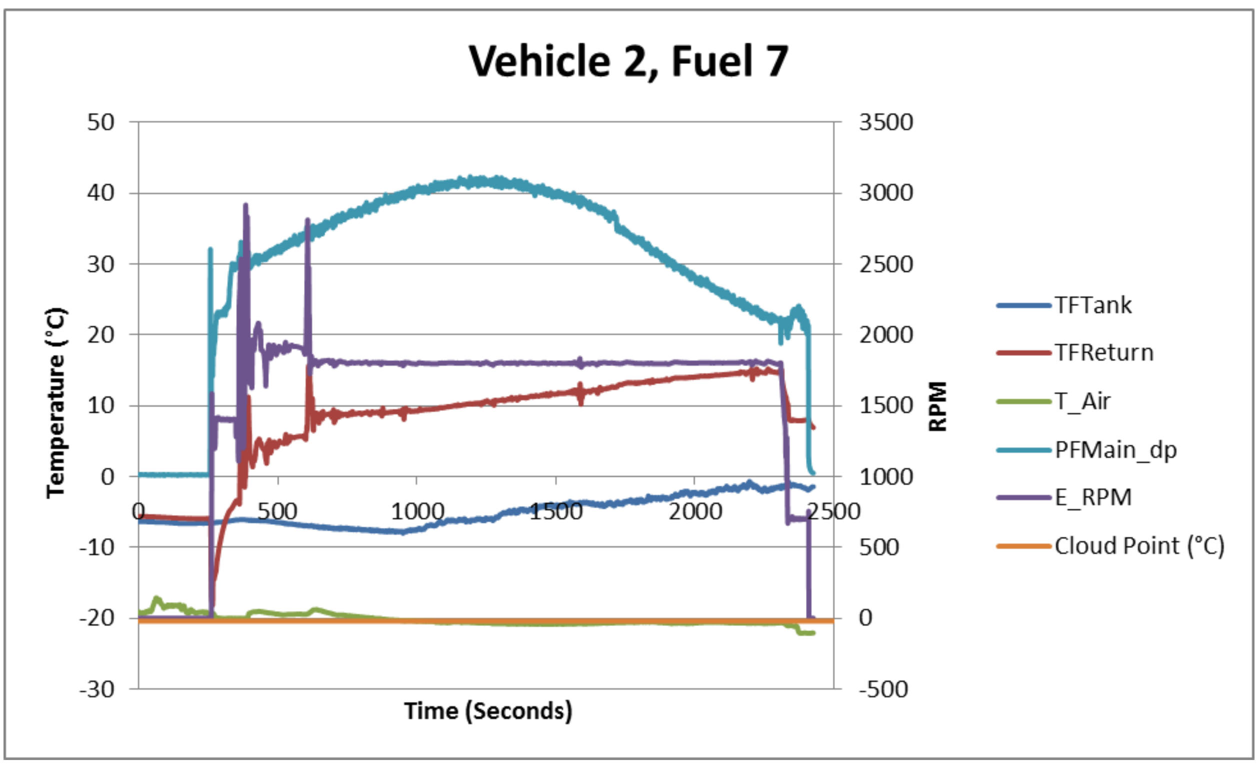


Figure B21: Select Data Series During AWCD Testing of Fuel 2 Vehicle 2

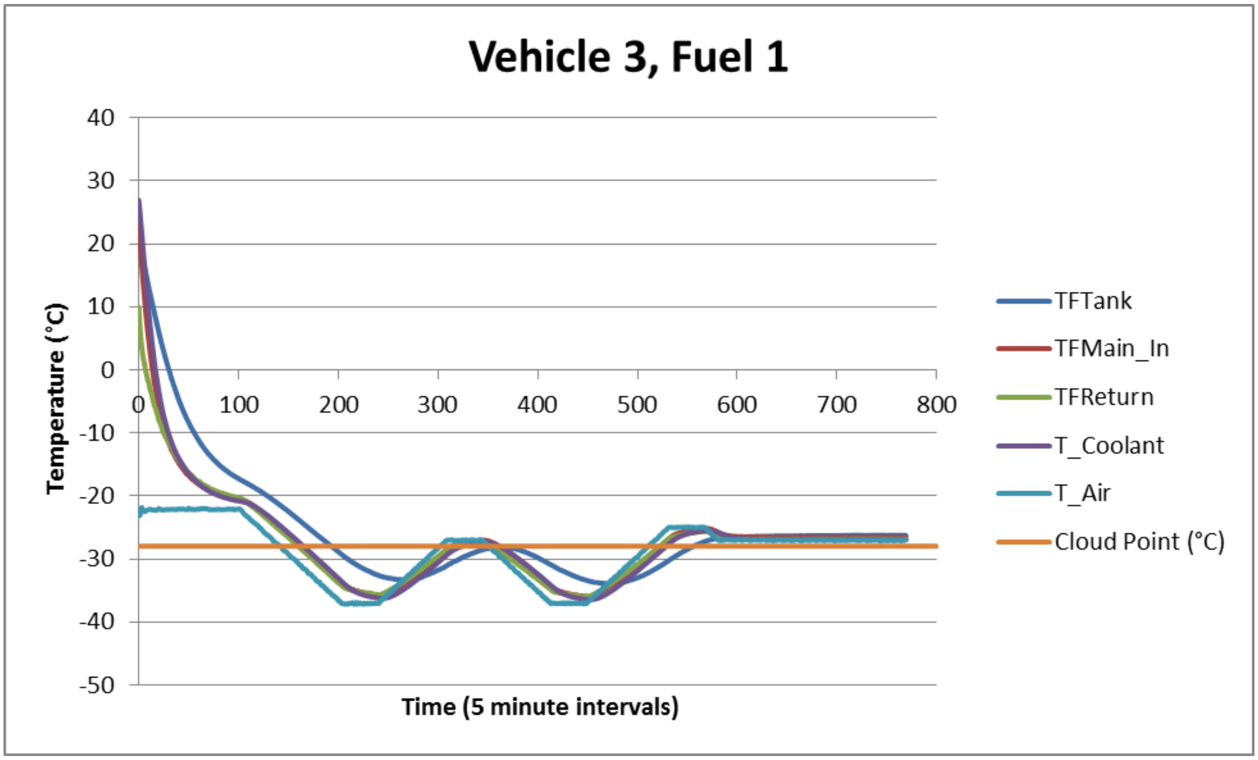


Figure B22: Diurnal Cold Soak of Fuel 1 Vehicle 3

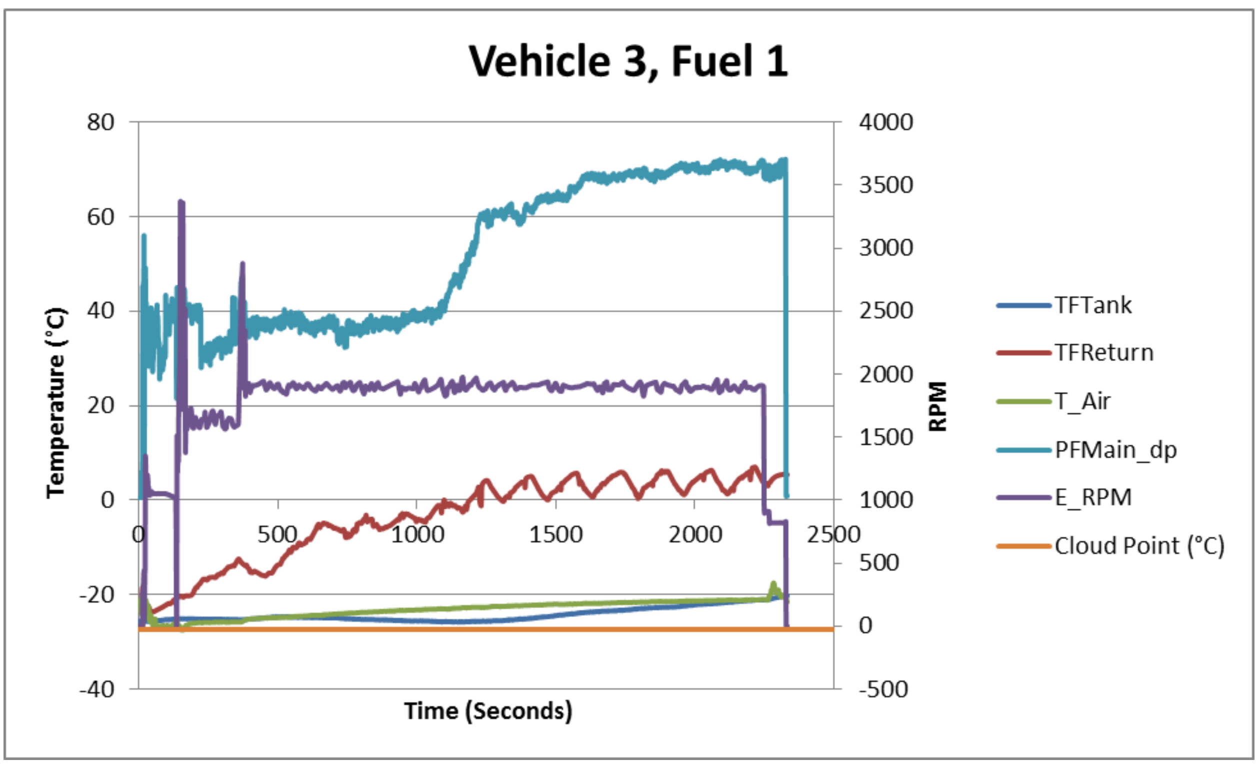


Figure B33: Select Data Series During AWCD Testing of Fuel 1 Vehicle 3

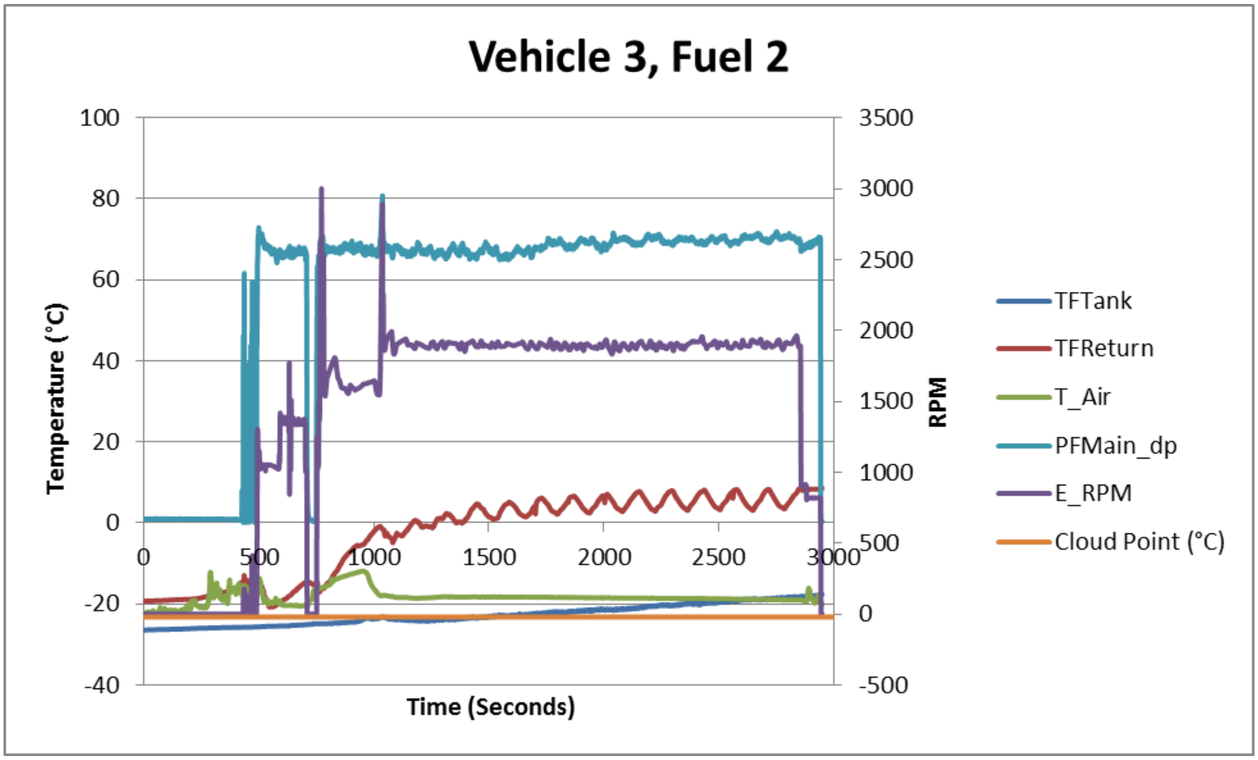


Figure B34: Select Data Series During AWCD Testing of Fuel 2 Vehicle 3

Note: Data capture for the diurnal cooling cycle failed to capture.

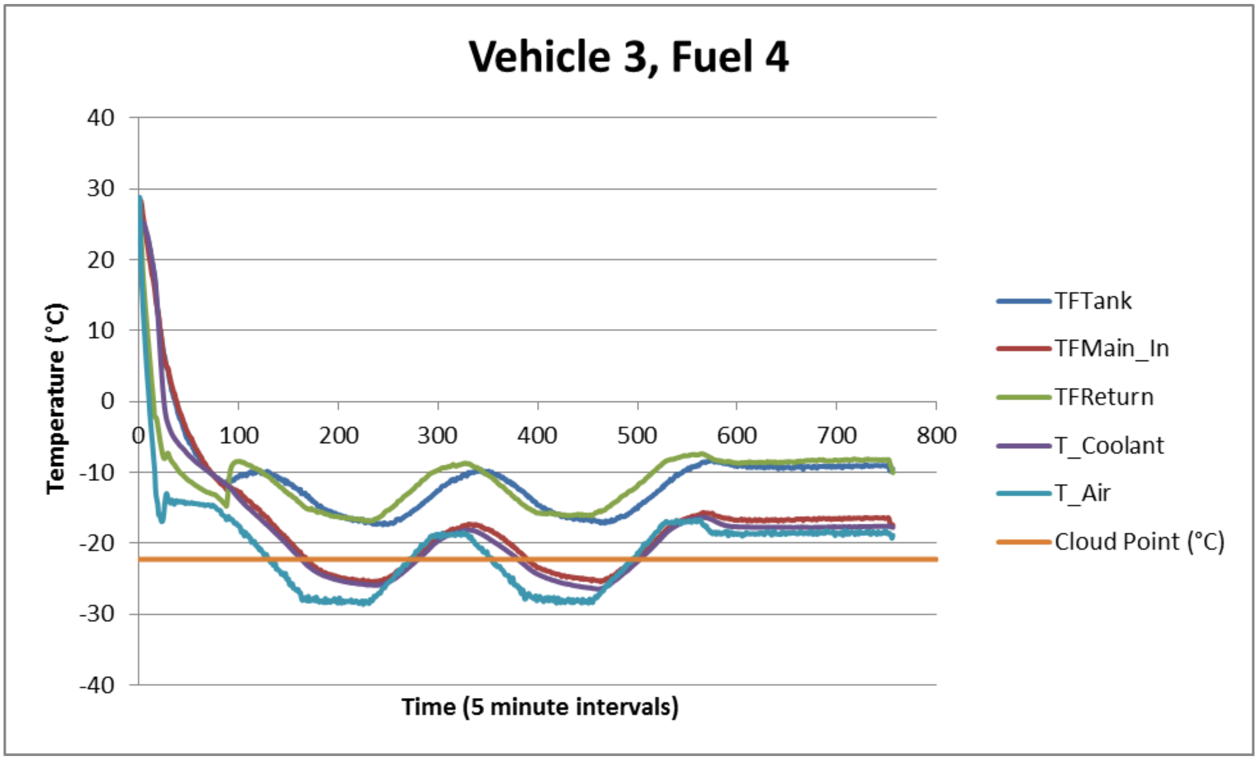


Figure B35: Diurnal Cold Soak of Fuel 4 Vehicle 3

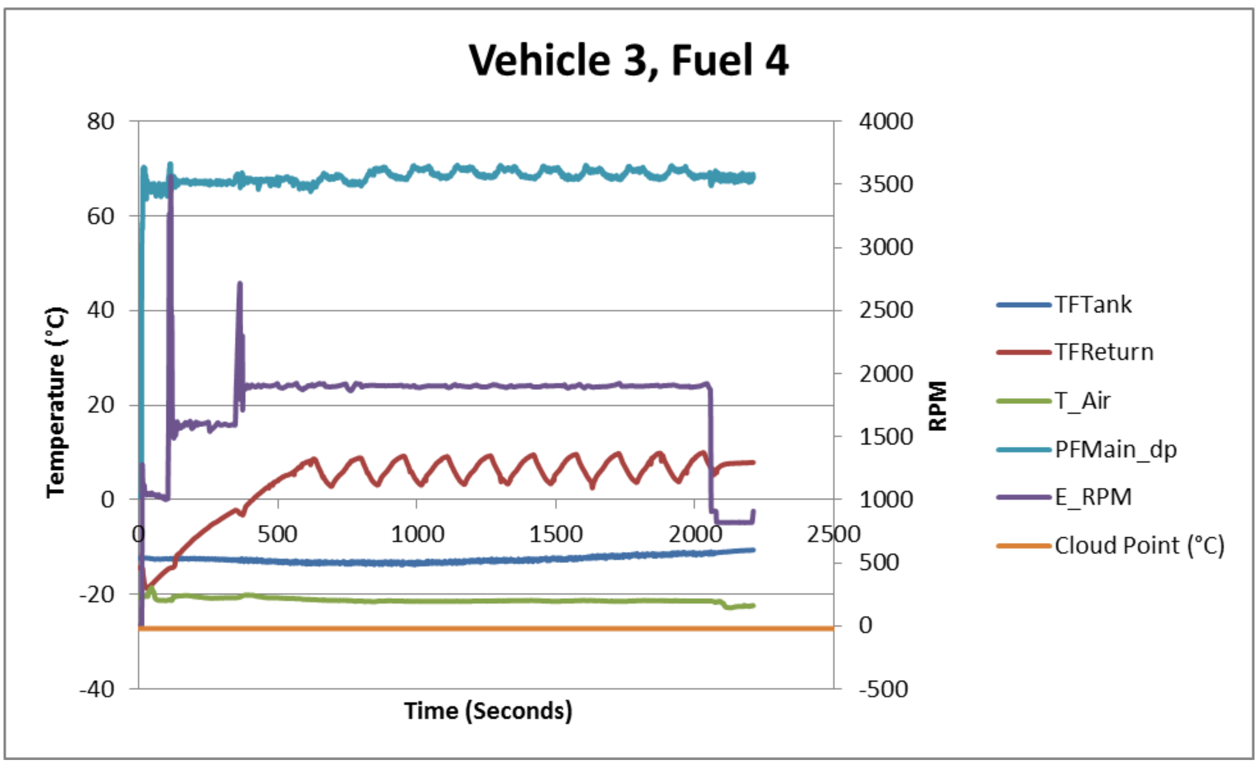


Figure B36: Select Data Series During AWCD Testing of Fuel 4 Vehicle 3

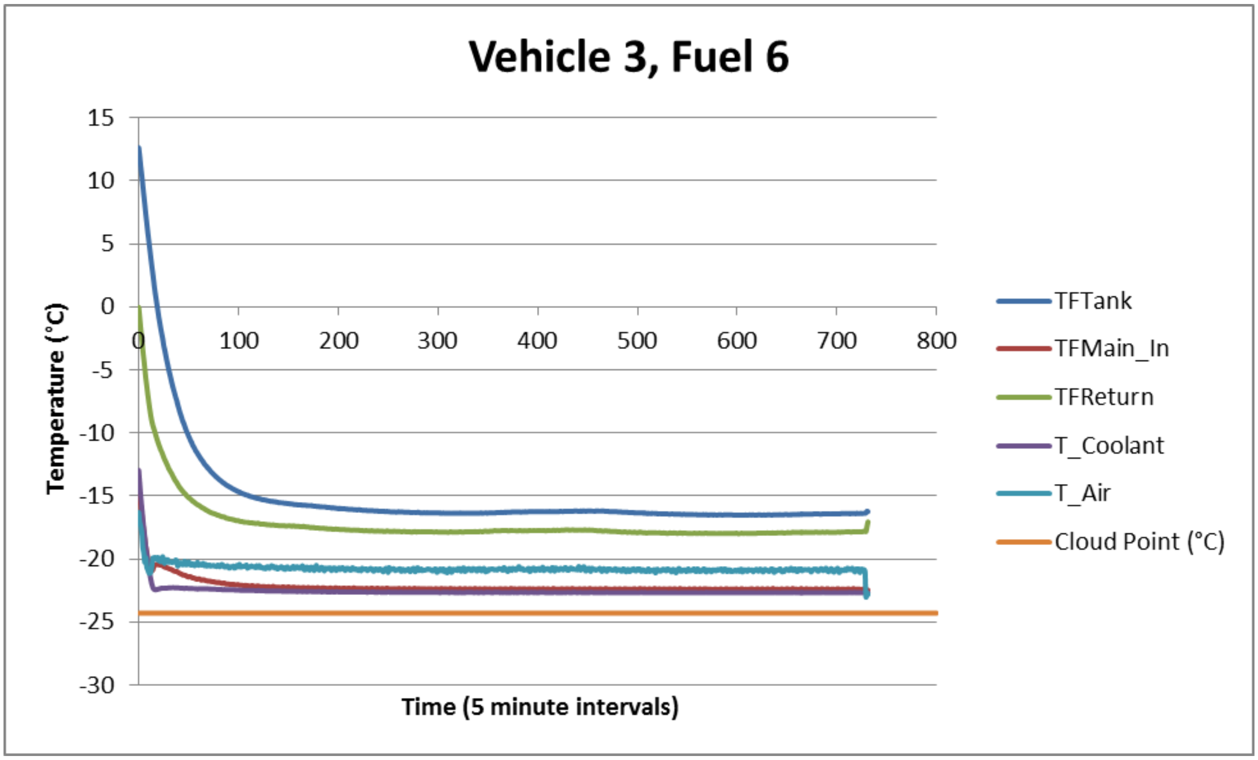


Figure B37: Diurnal Cold Soak of Fuel 6 Vehicle 3

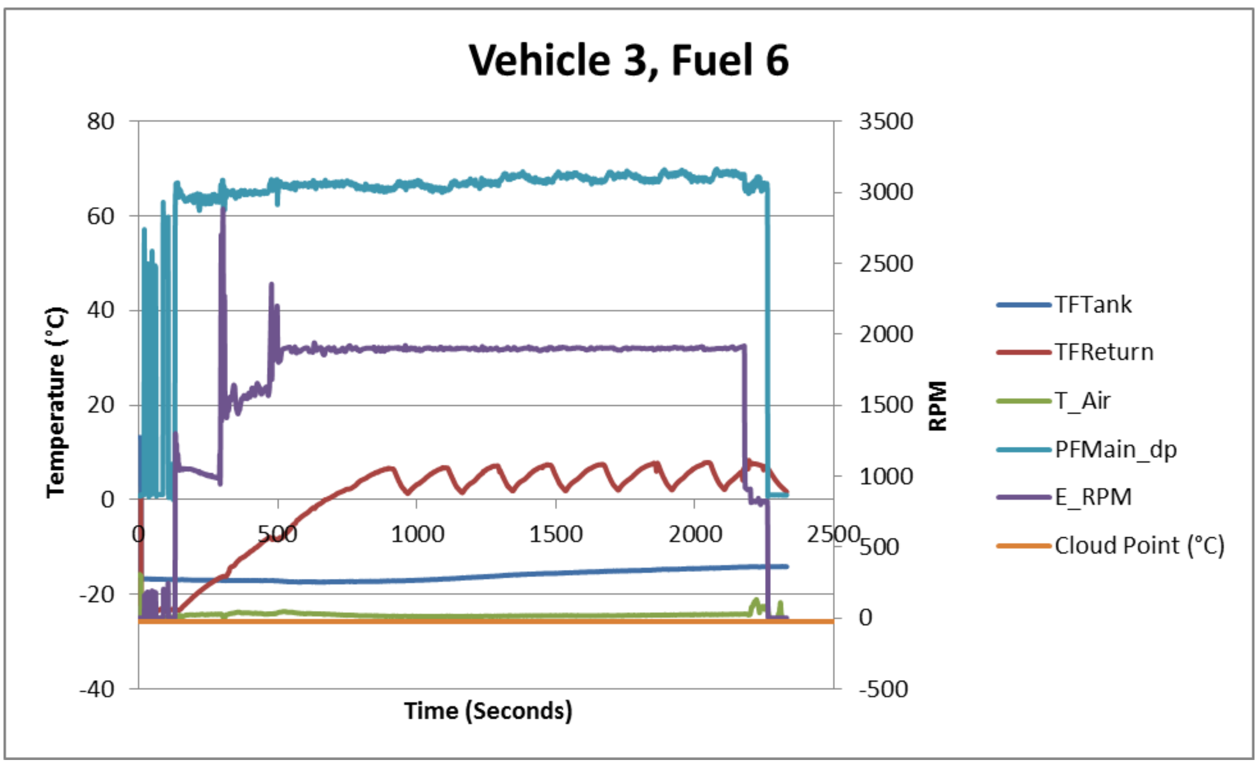


Figure B38: Select Data Series During AWCD Testing of Fuel 6 Vehicle 3

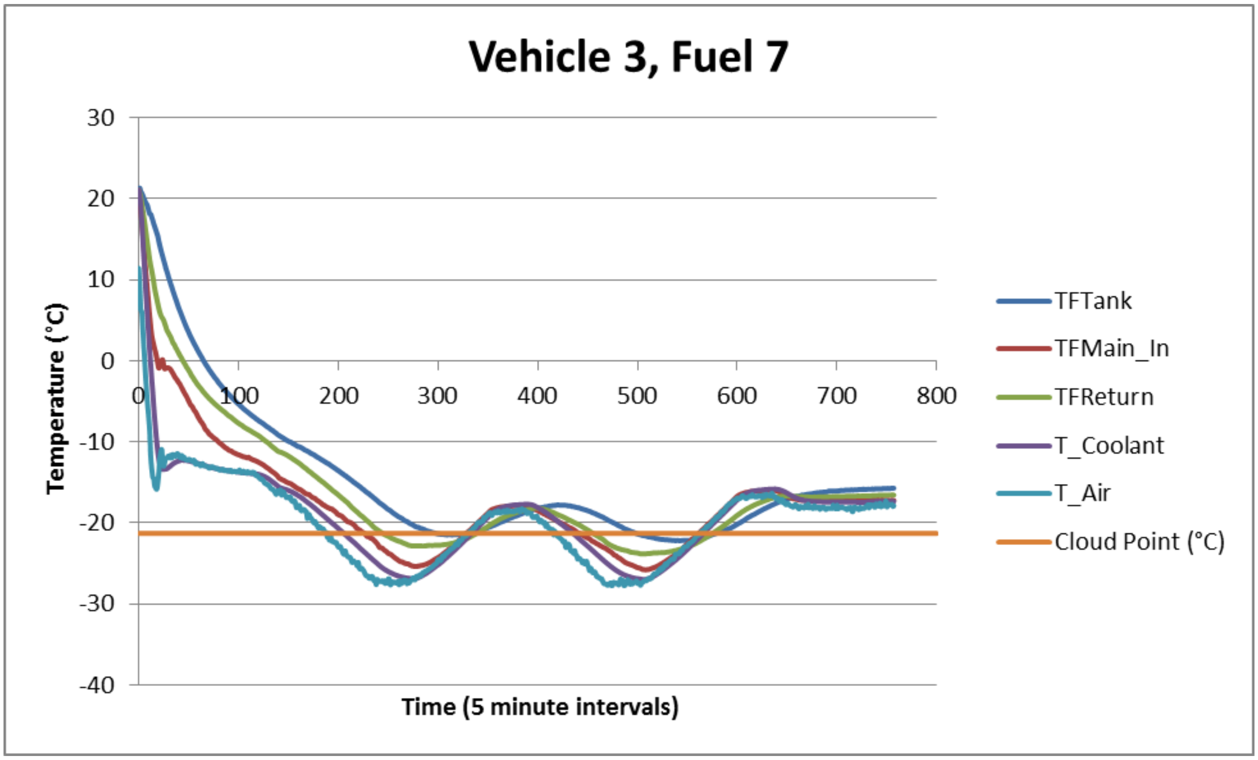


Figure B39: Diurnal Cold Soak of Fuel 7 Vehicle 3

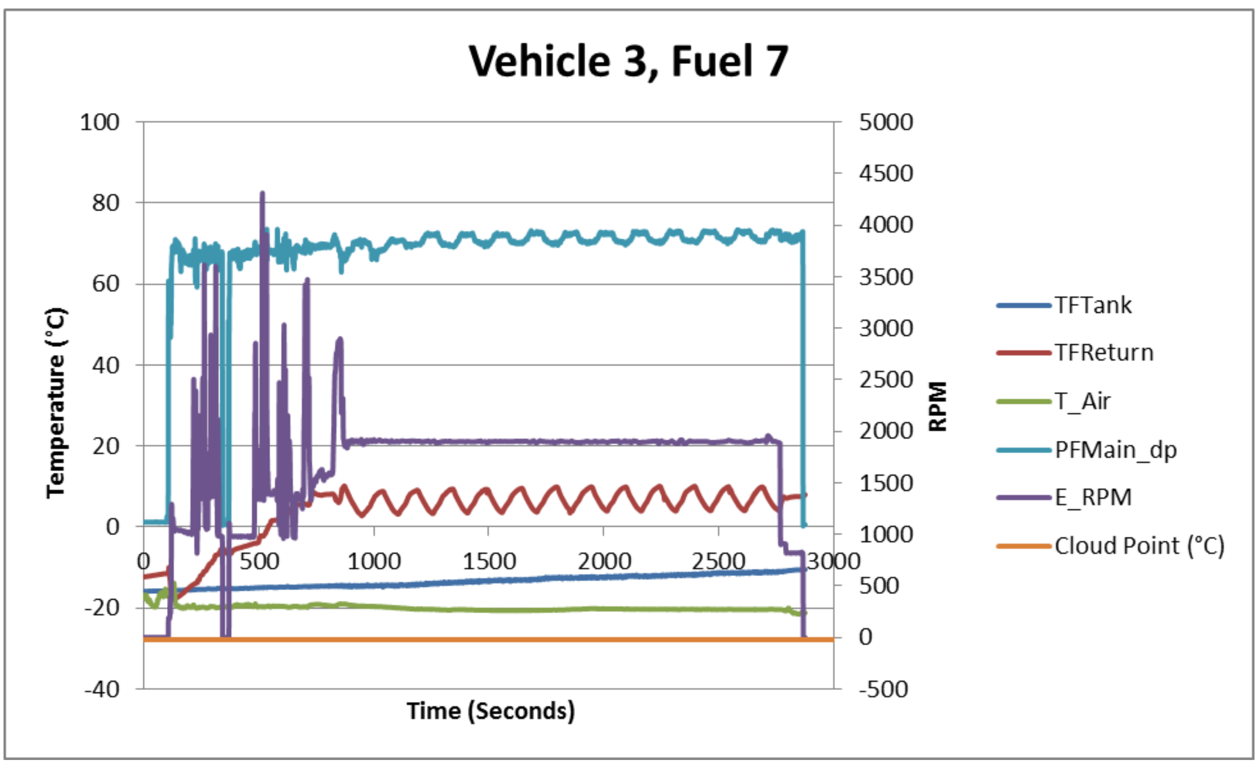


Figure B40: Select Data Series During AWCD Testing of Fuel 7 Vehicle 3

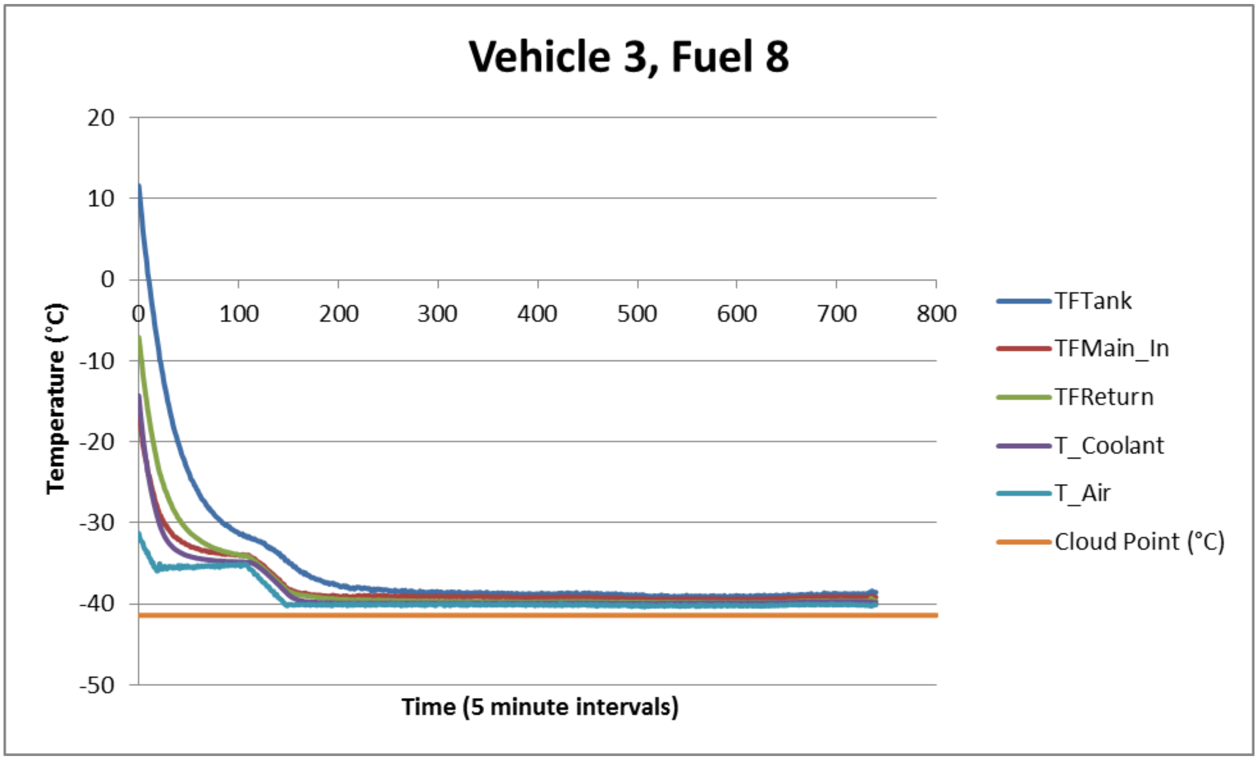


Figure B40: Diurnal Cold Soak of Fuel 8 Vehicle 3

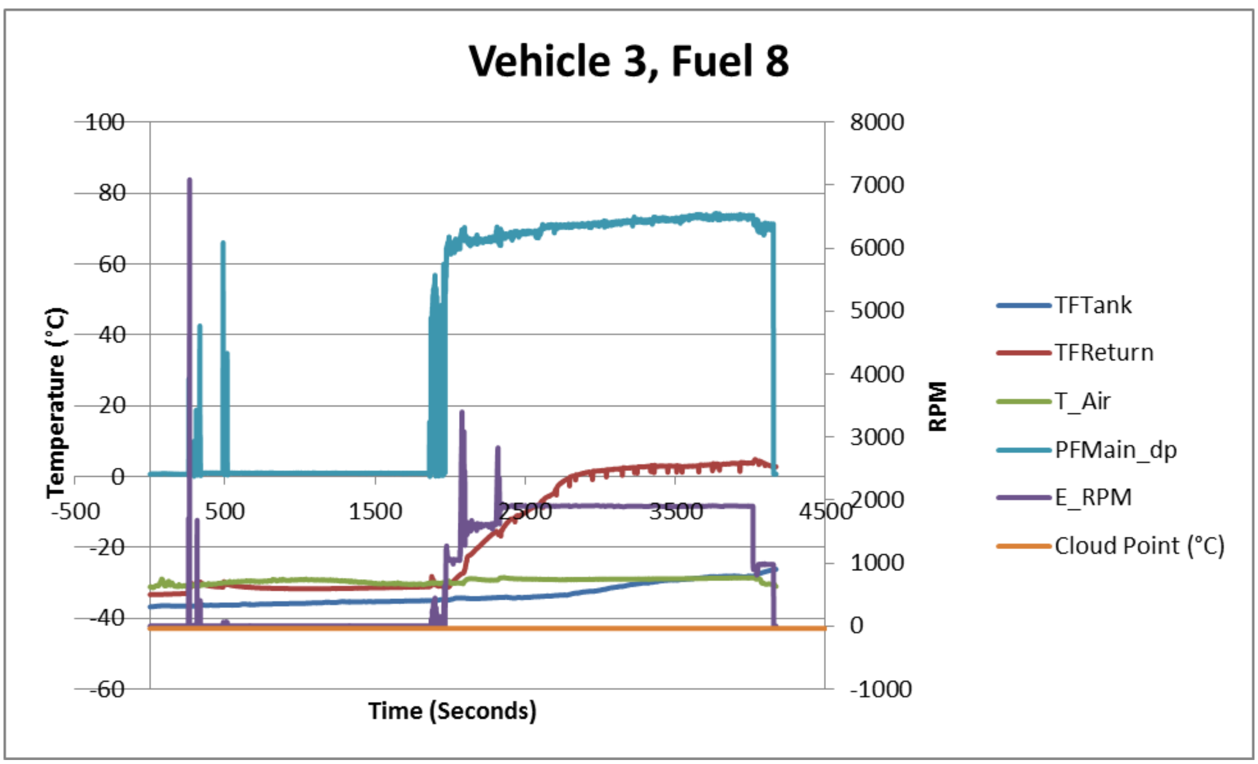


Figure B41: Select Data Series During AWCD Testing of Fuel 8 Vehicle 3

Appendix C: Comparison of Upper and Lower Fuels Samples n-paraffin content

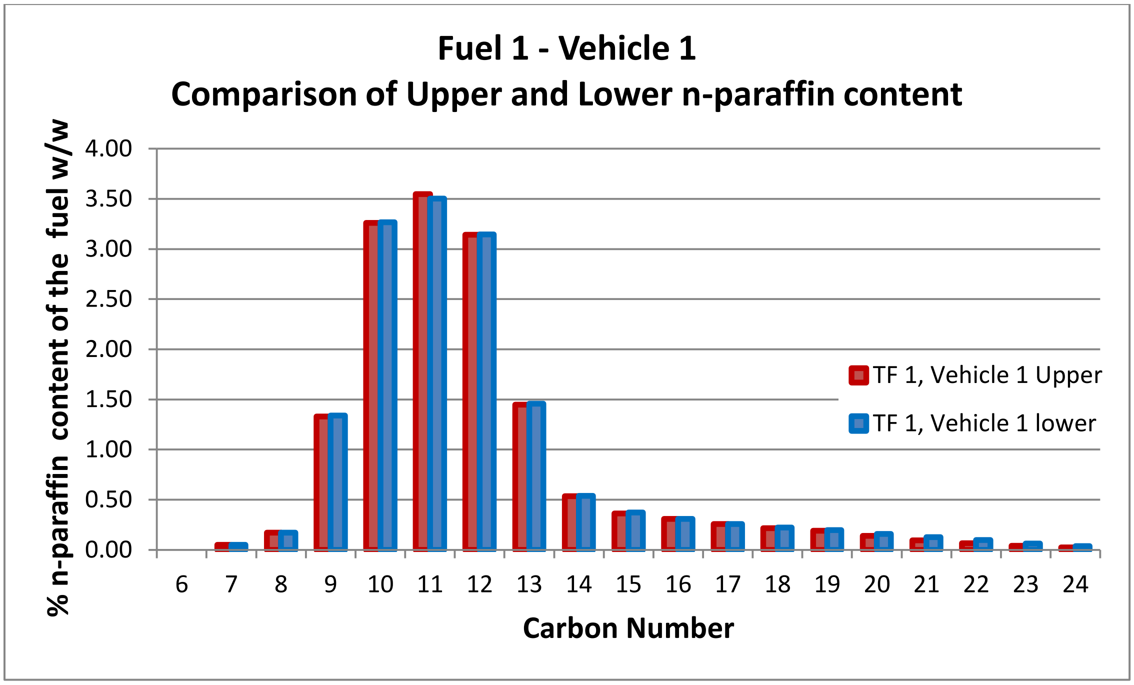


Figure C1a: Comparison of Upper sample and lower sample n-paraffin content (fuel taken just prior to vehicle test cycle). Fuel 1 Vehicle 1

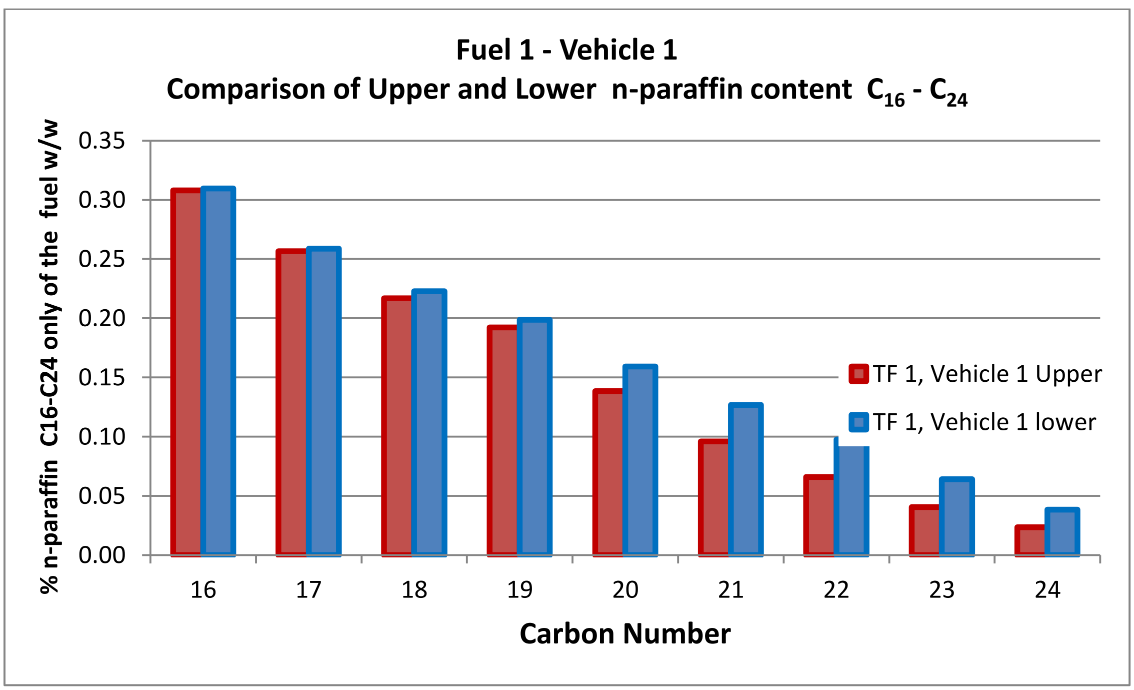


Figure C1b: Comparison of Upper sample and Lower sample n-paraffin content C_{16}-C_{24} taken just before vehicle test cycle. Fuel 1 Vehicle 1

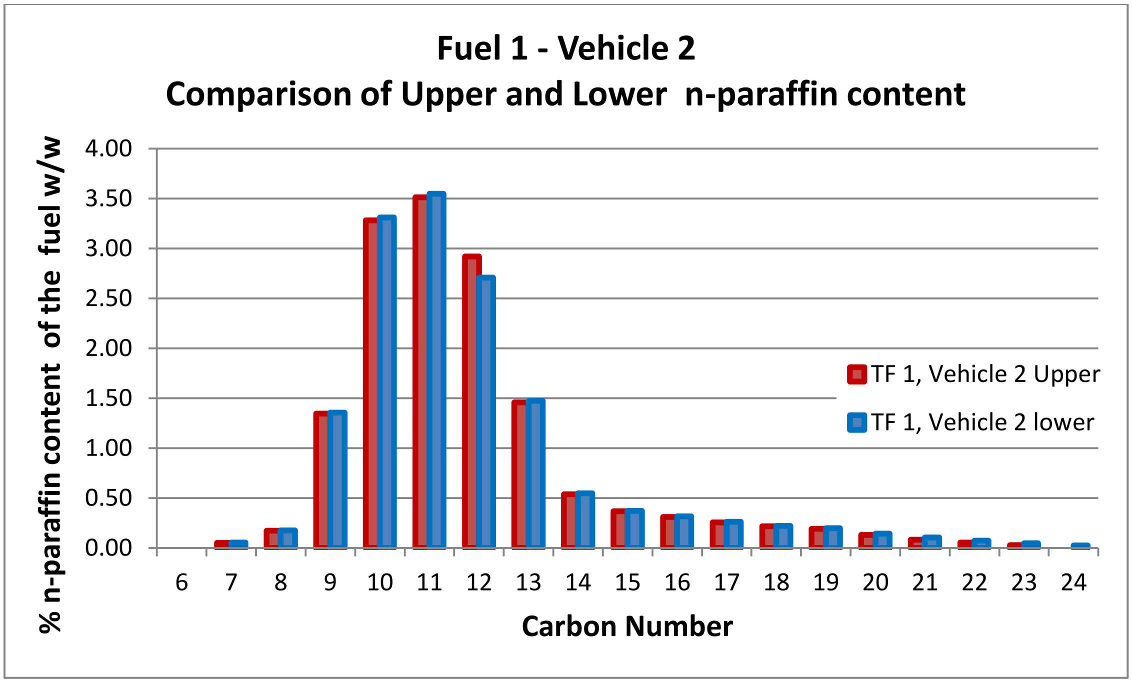


Figure C2a: Overlay of Upper sample and Lower sample n-paraffin content (fuel taken just prior to vehicle test cycle). Fuel 1 Vehicle 2

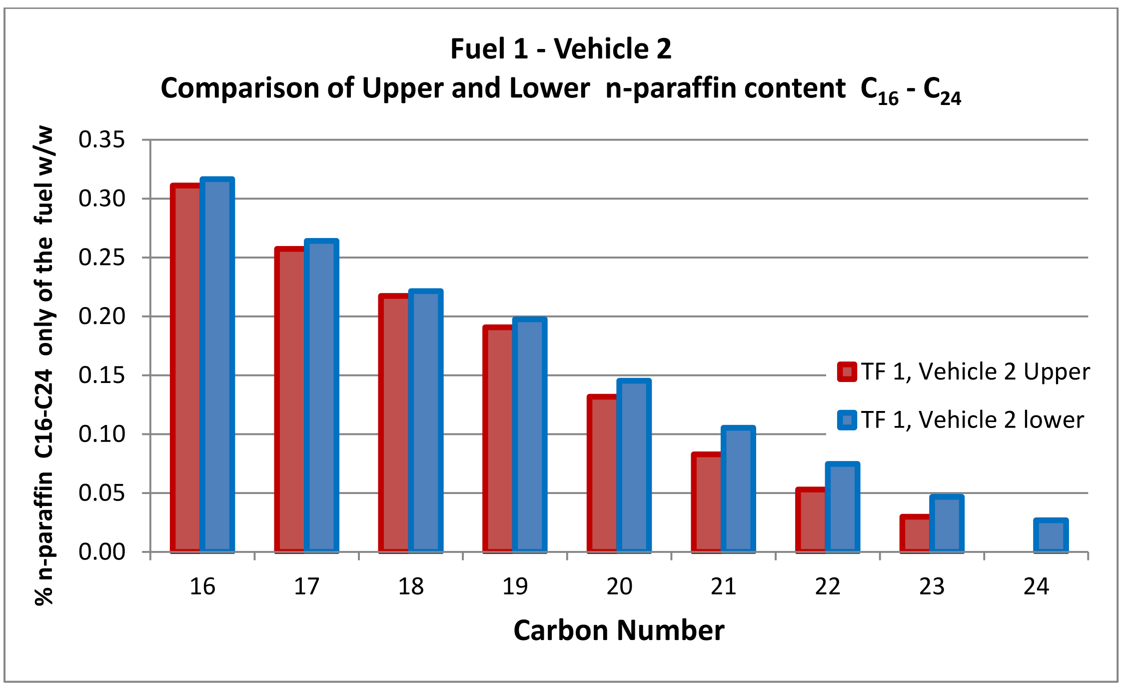


Figure C2b: Overlay of Upper sample and Lower sample n-paraffin content C_{16}-C_{24} (fuel taken just prior to vehicle test cycle). Fuel 1 Vehicle 2

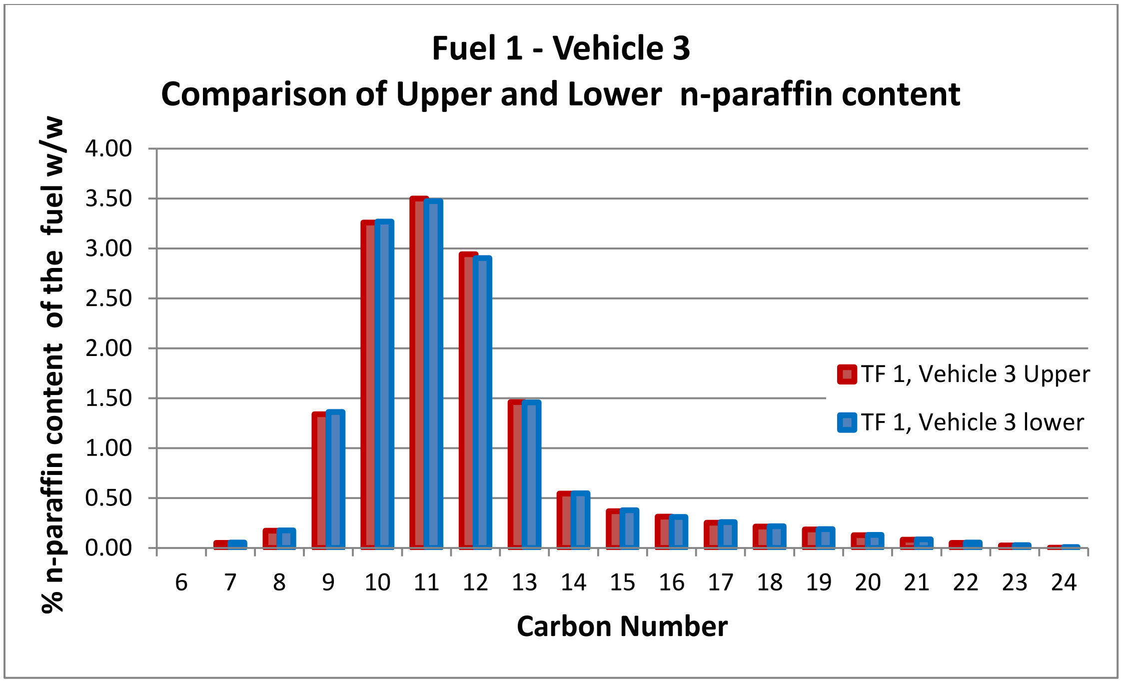


Figure C3a: Comparison of Upper sample and lower sample n-paraffin content (fuel taken just prior to vehicle test cycle). Fuel 1 Vehicle 3

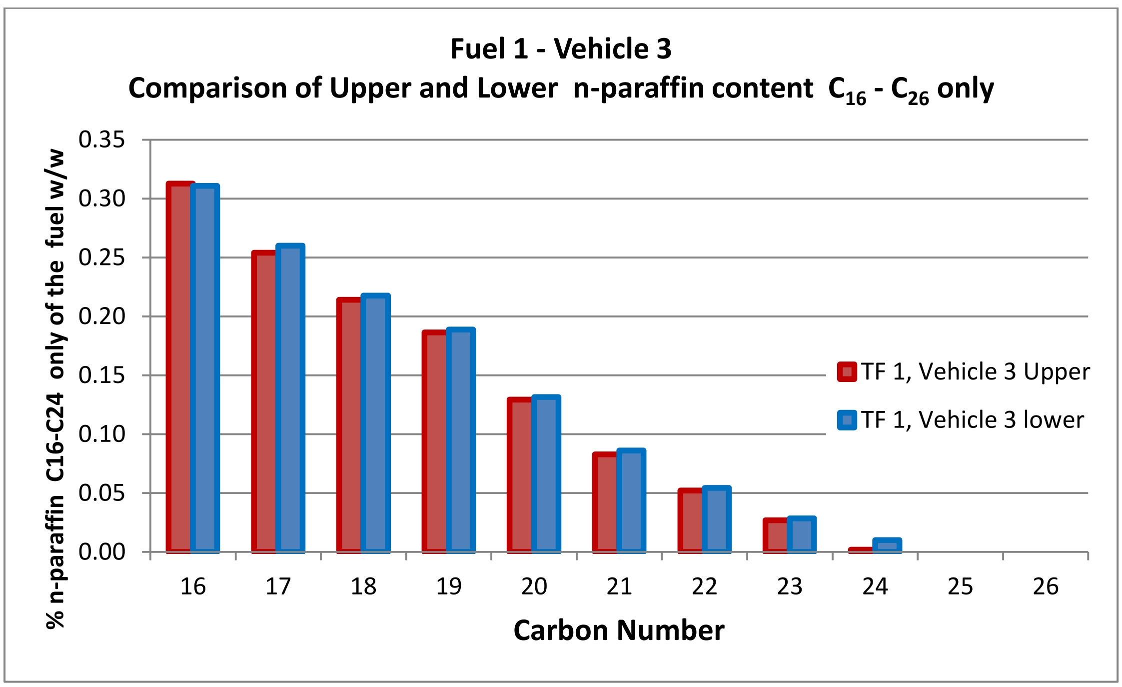


Figure C3b: Comparison of Upper sample and Lower sample n-paraffin content C_{16}-C_{24} taken just before vehicle test cycle. Fuel 1 Vehicle 3

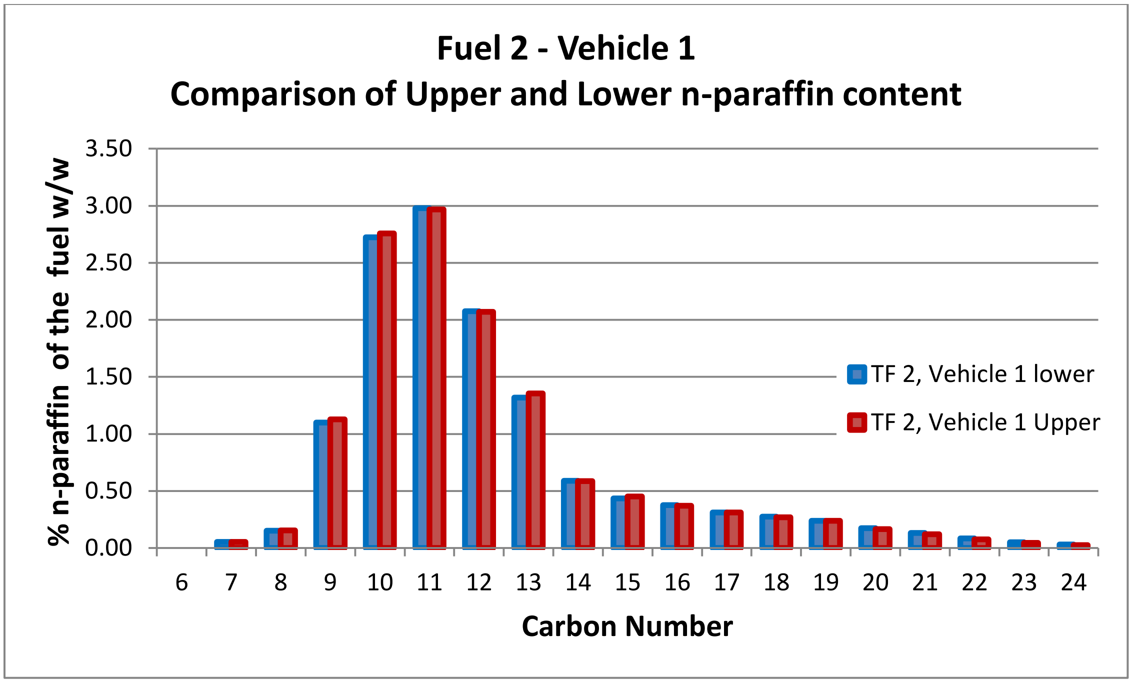


Figure C4a: Comparison of Upper sample and lower sample n-paraffin content (fuel taken just prior to vehicle test cycle). Fuel 2 Vehicle 1

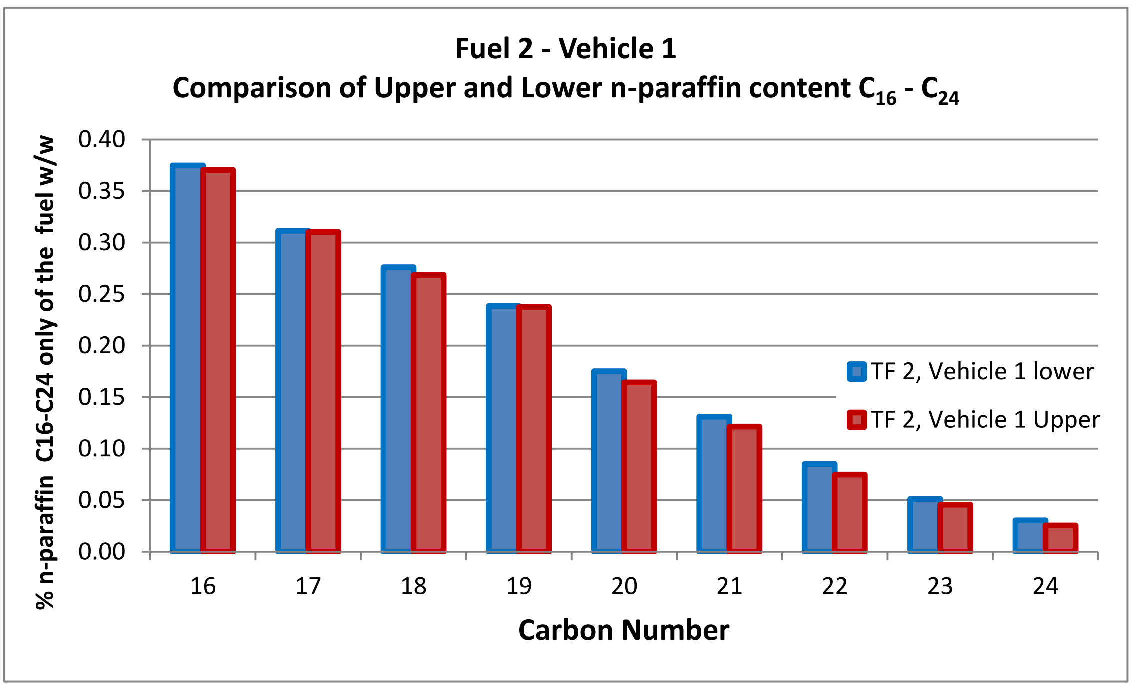


Figure C4b: Comparison of Upper sample and Lower sample n-paraffin content C_{16}-C_{24} taken just before vehicle test cycle. Fuel 2 Vehicle 1

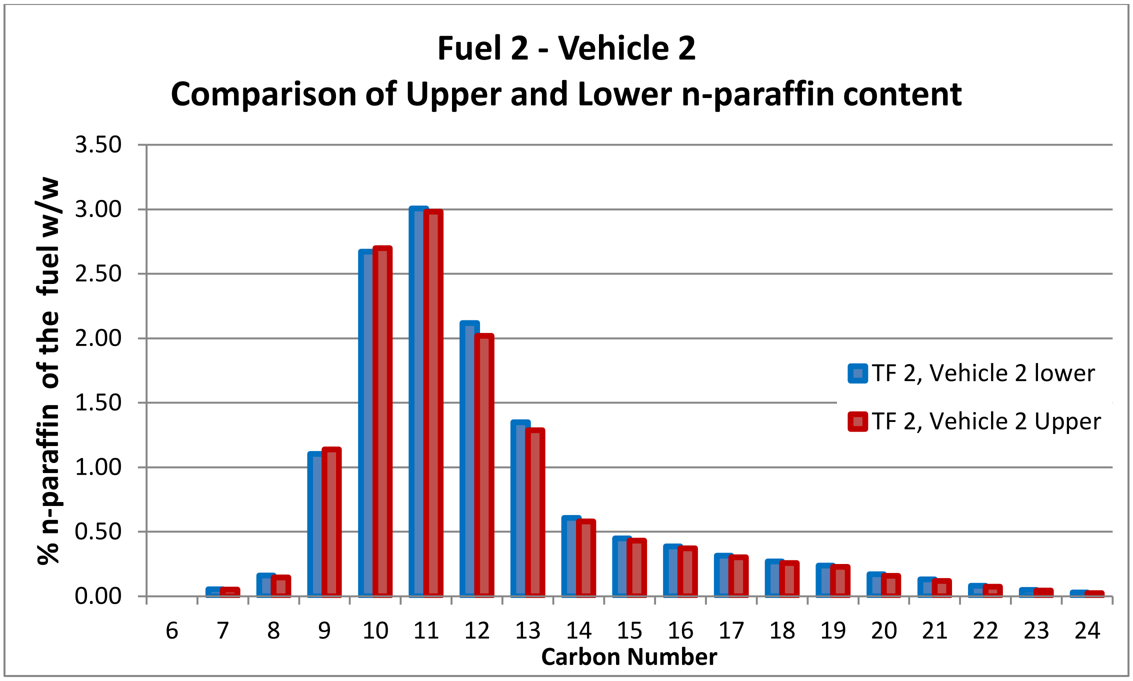


Figure C5a: Comparison of Upper sample and lower sample n-paraffin content (fuel taken just prior to vehicle test cycle). Fuel 2 Vehicle 2

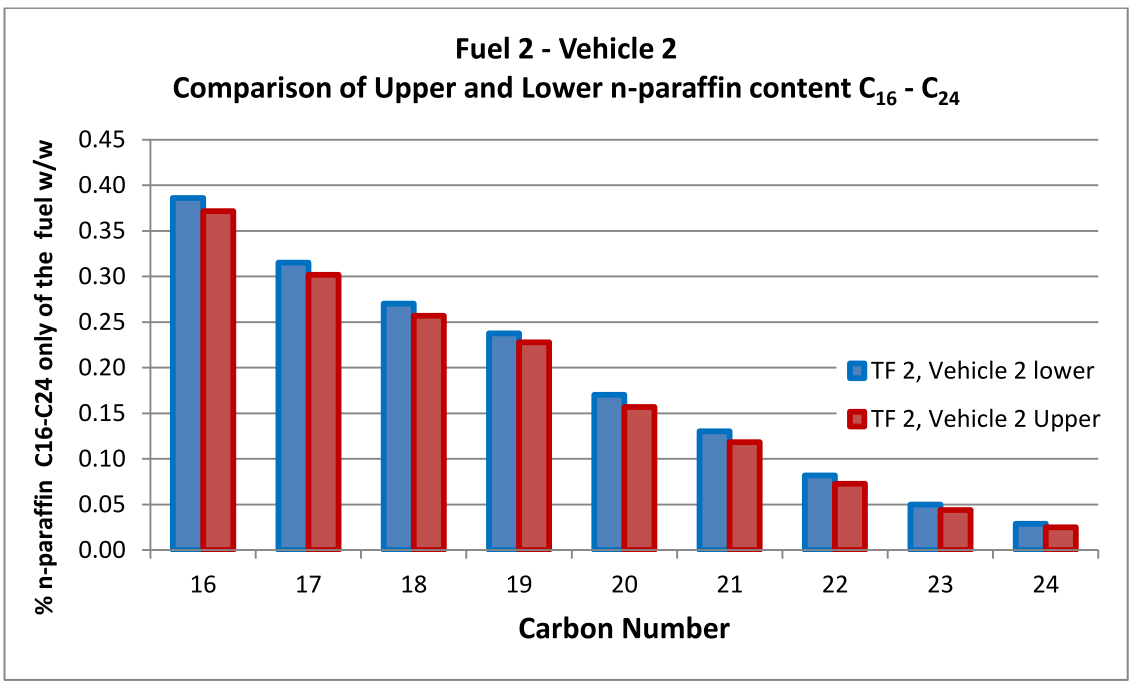


Figure C5b: Comparison of Upper sample and Lower sample n-paraffin content C_{16}-C_{24} taken just before vehicle test cycle. Fuel 2 Vehicle 2

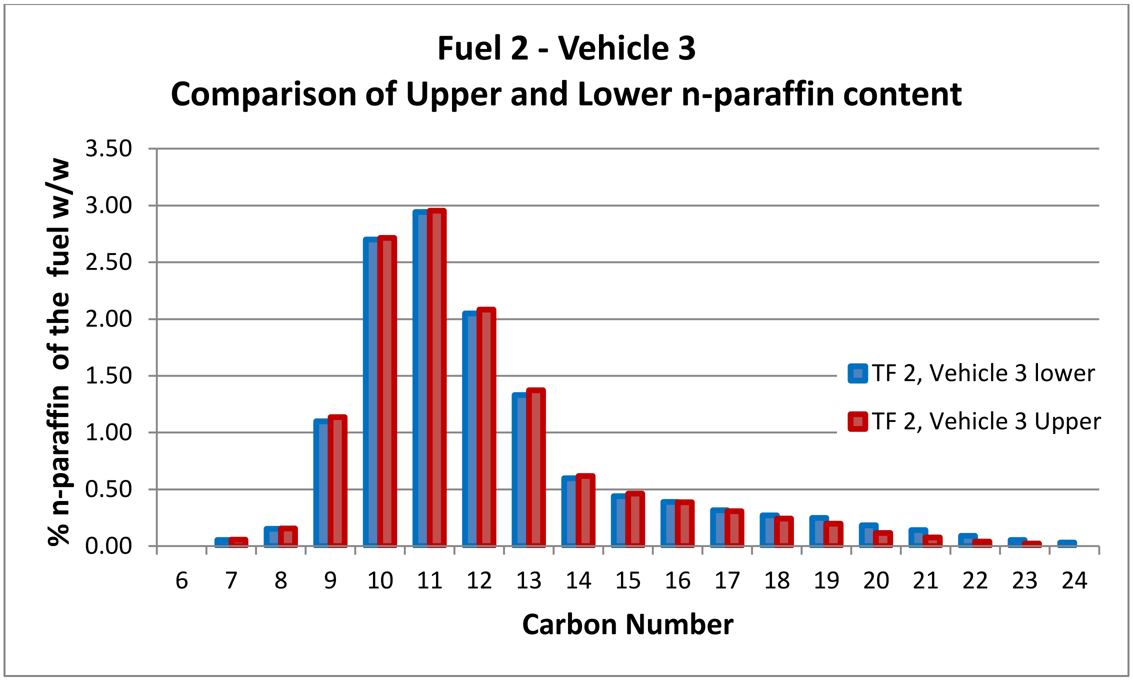


Figure C6a: Comparison of Upper sample and lower sample n-paraffin content (fuel taken just prior to vehicle test cycle). Fuel 2 Vehicle 3

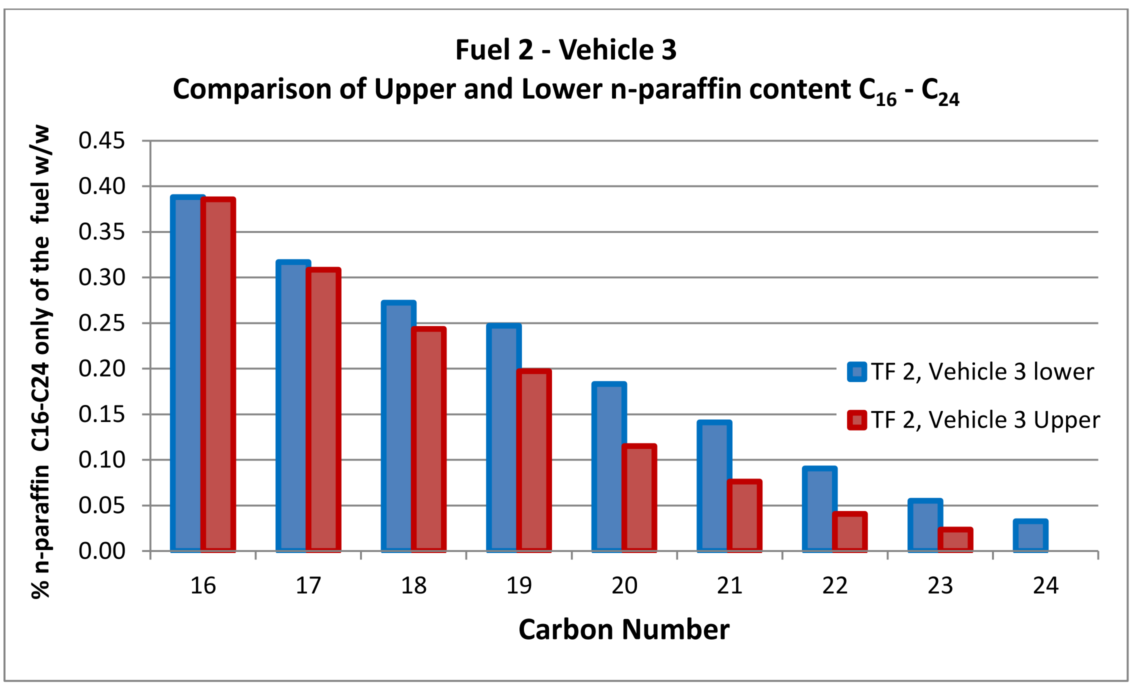


Figure C6b: Comparison of Upper sample and Lower sample n-paraffin content C_{16}-C_{24} taken just before vehicle test cycle. Fuel 2 Vehicle 3

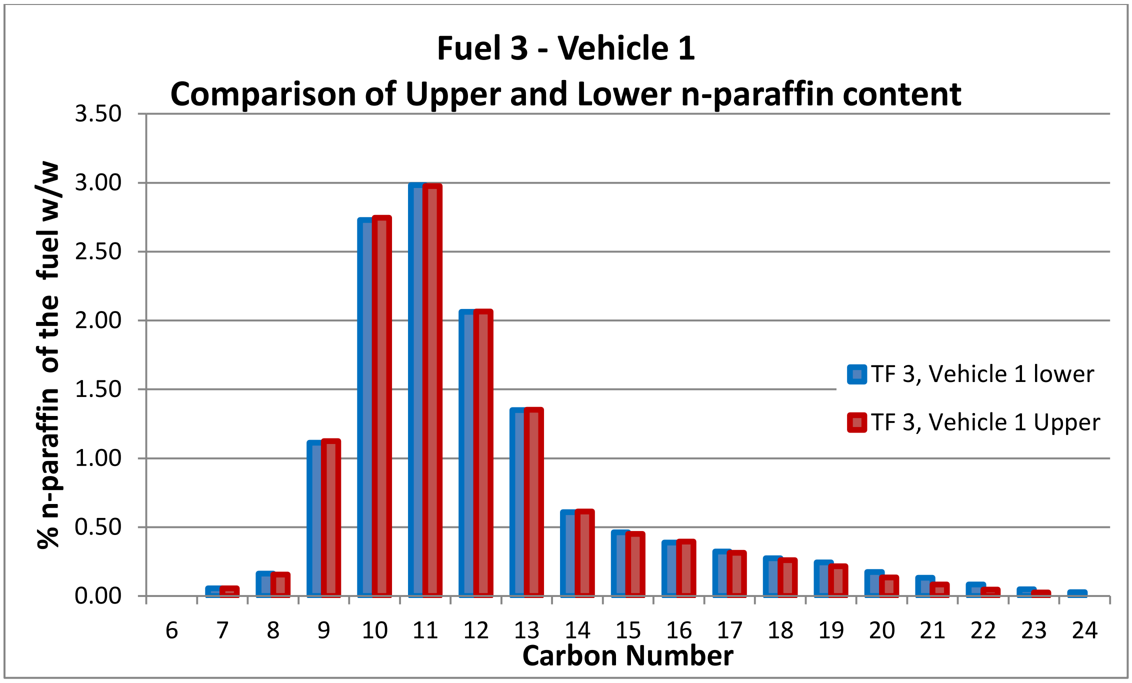


Figure C7a: Comparison of Upper sample and lower sample n-paraffin content (fuel taken just prior to vehicle test cycle). Fuel 3 Vehicle 1

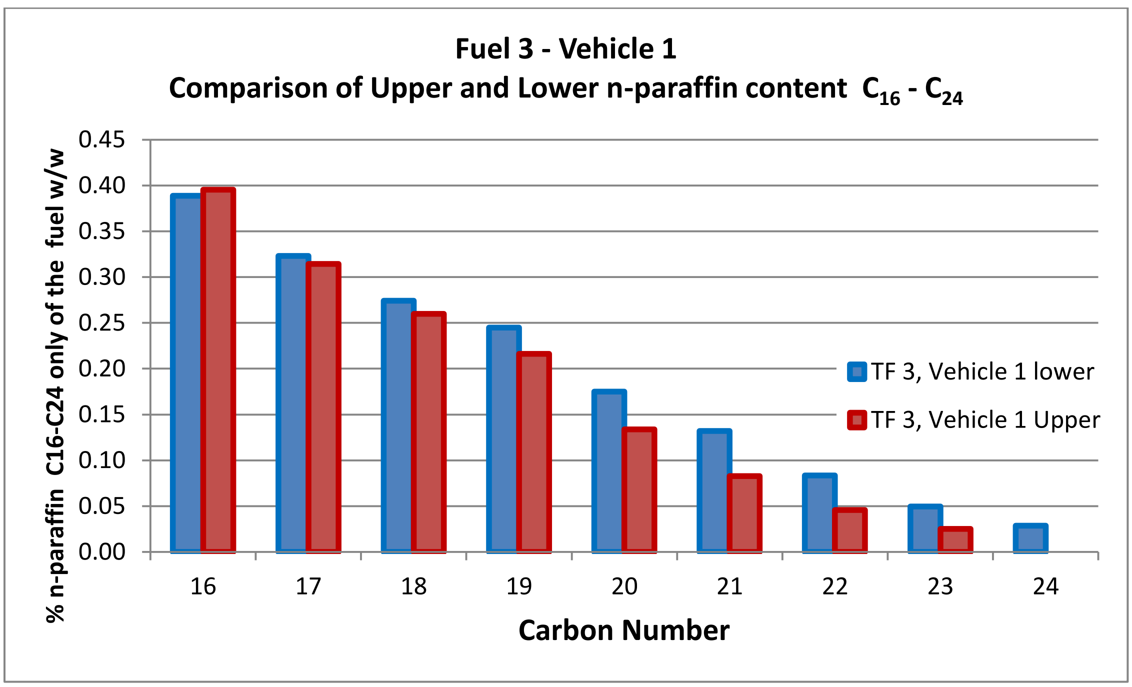


Figure C7b: Comparison of Upper sample and Lower sample n-paraffin content C_{16}-C_{24} taken just before vehicle test cycle. Fuel 3 Vehicle 1

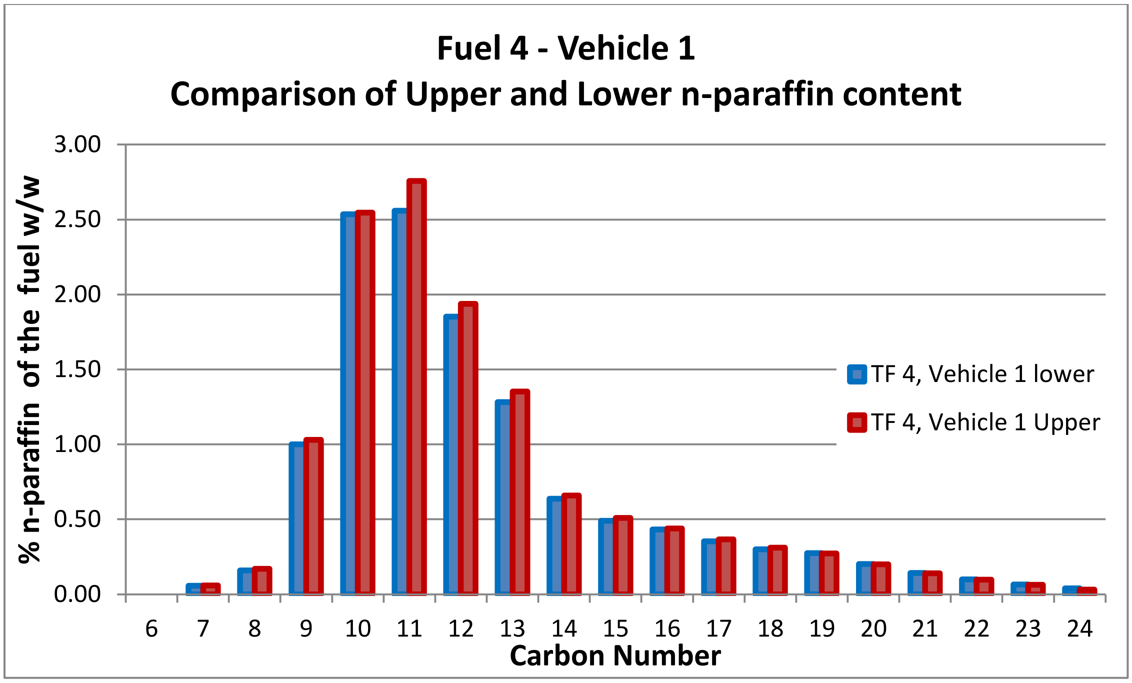


Figure C8a: Comparison of Upper sample and lower sample n-paraffin content (fuel taken just prior to vehicle test cycle). Fuel 4 Vehicle 1

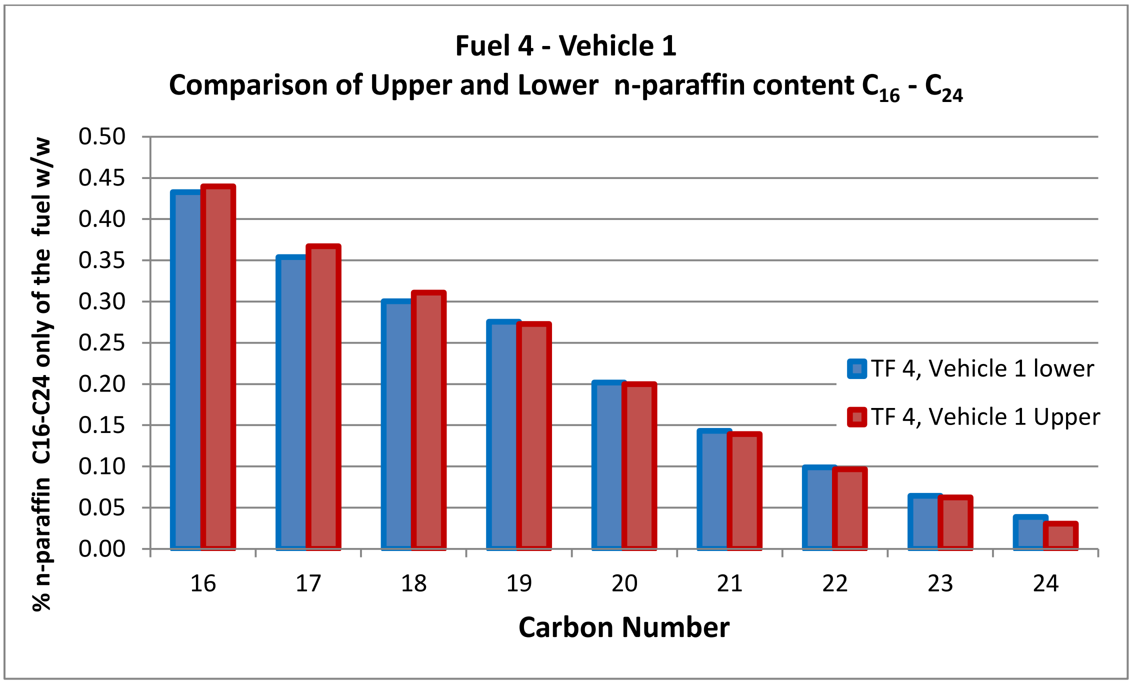


Figure C8b: Comparison of Upper sample and Lower sample n-paraffin content C_{16}-C_{24} taken just before vehicle test cycle. Fuel 4 Vehicle 1

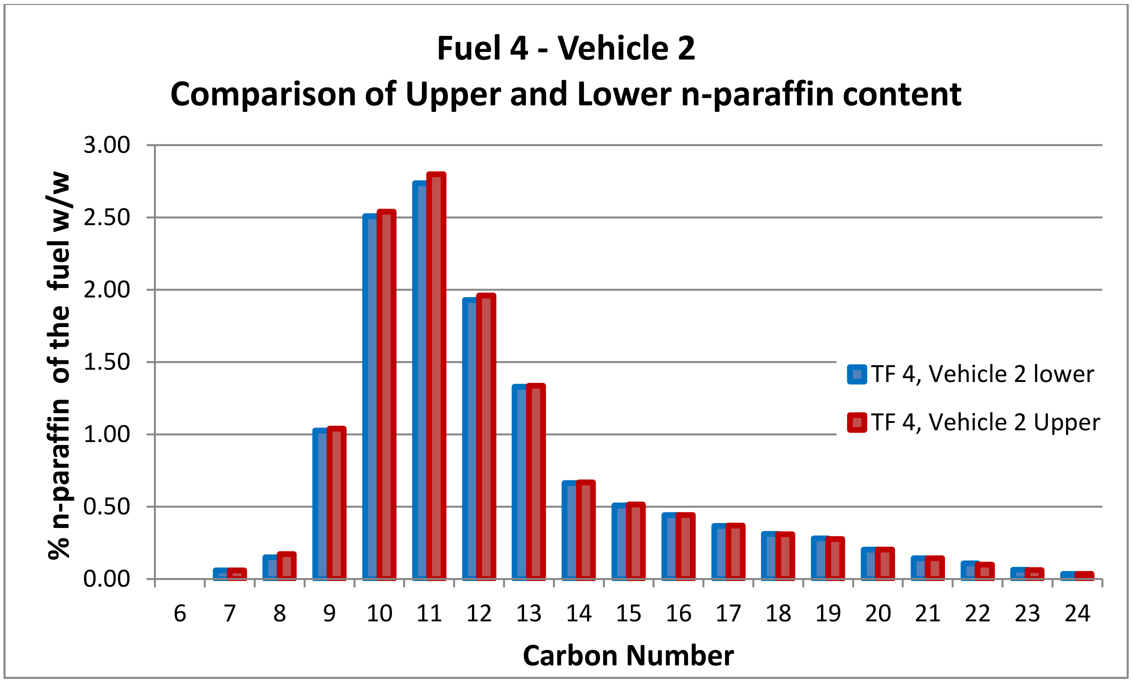


Figure C9a: Comparison of Upper sample and lower sample n-paraffin content (fuel taken just prior to vehicle test cycle). Fuel 4 Vehicle 2

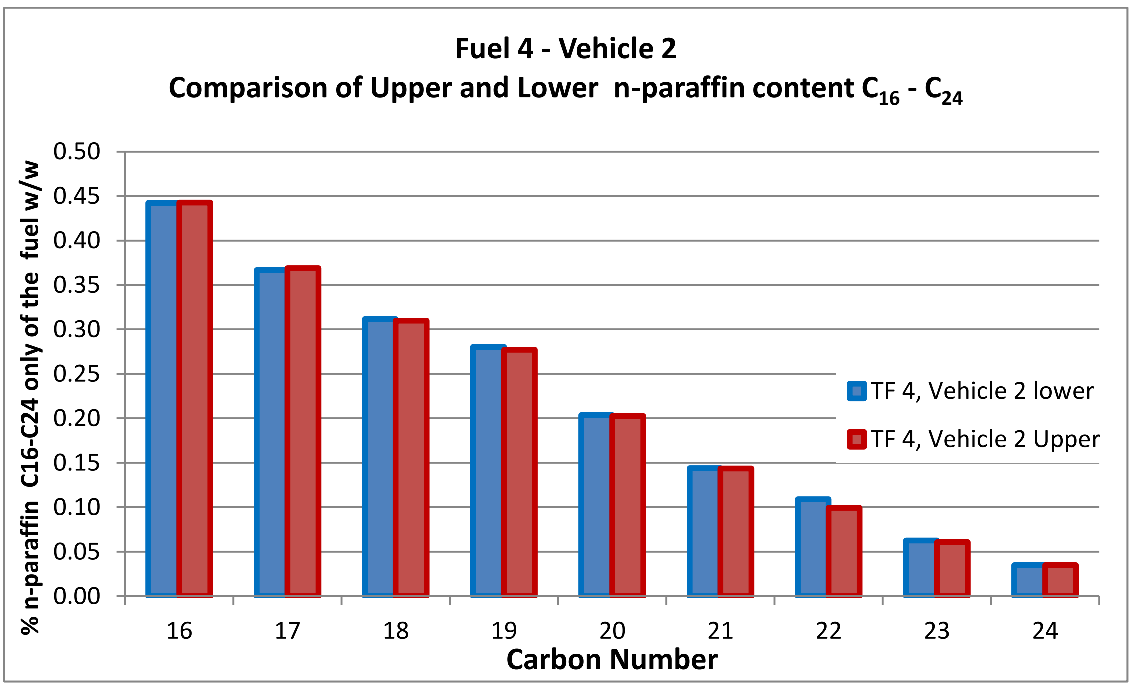


Figure C9b: Comparison of Upper sample and Lower sample n-paraffin content C_{16}-C_{24} taken just before vehicle test cycle. Fuel 4 Vehicle 2

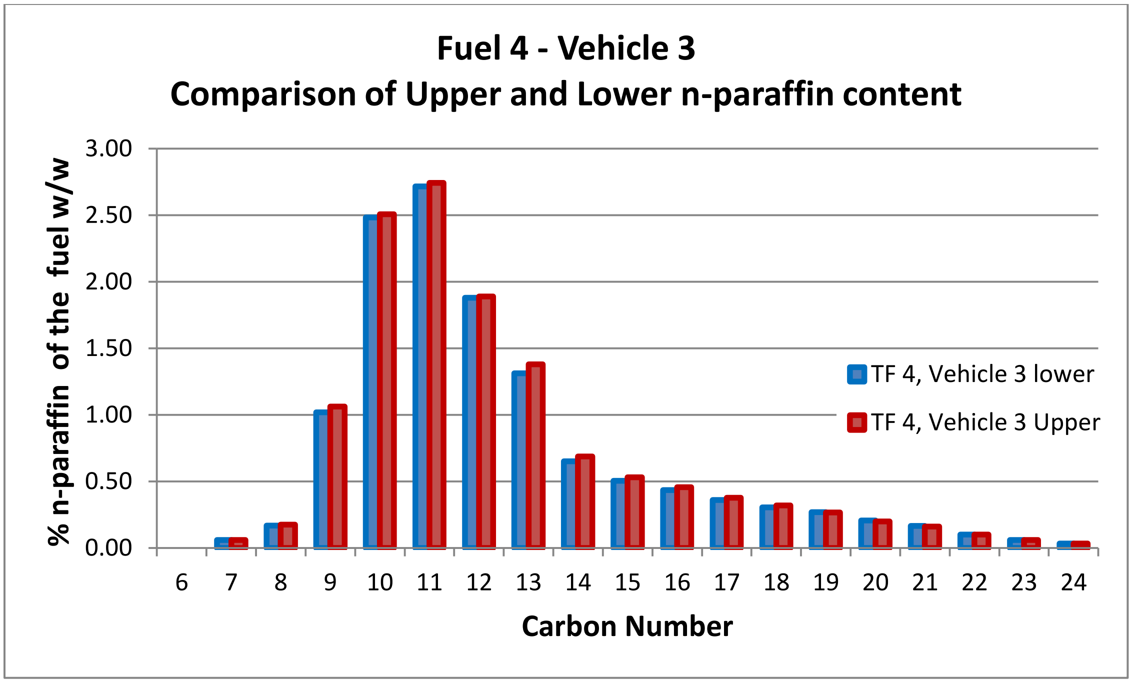


Figure C10a: Comparison of Upper sample and lower sample n-paraffin content (fuel taken just prior to vehicle test cycle). Fuel 4 Vehicle 3

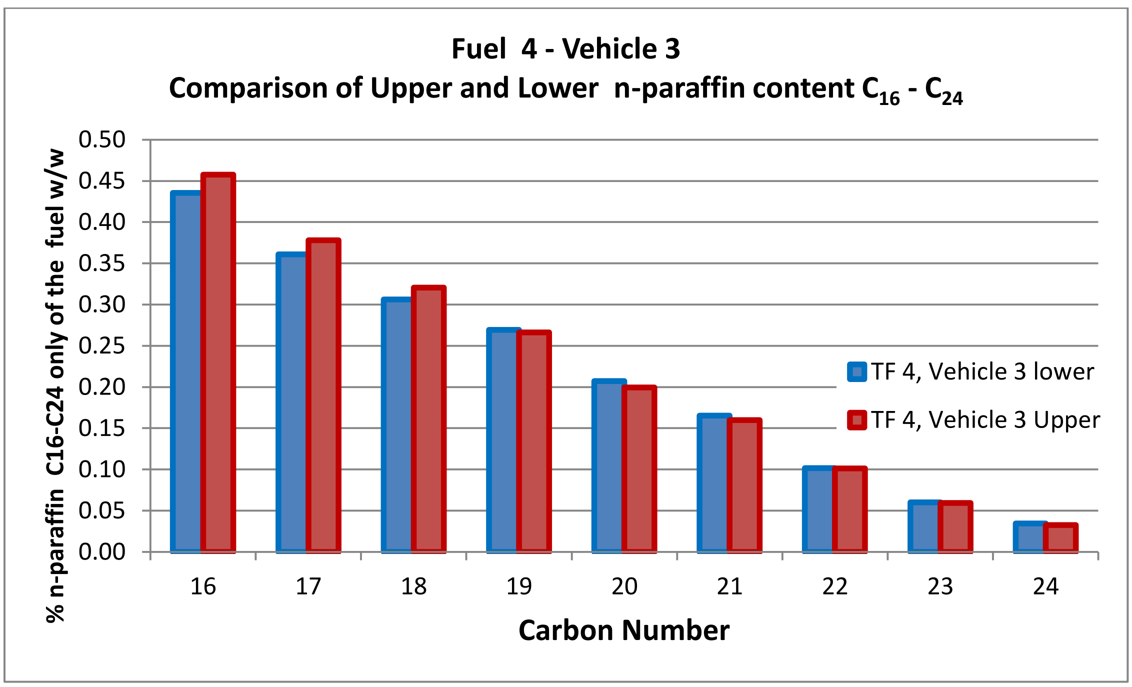


Figure C10b: Comparison of Upper sample and Lower sample n-paraffin content C_{16}-C_{24} taken just before vehicle test cycle. Fuel 4 Vehicle 3

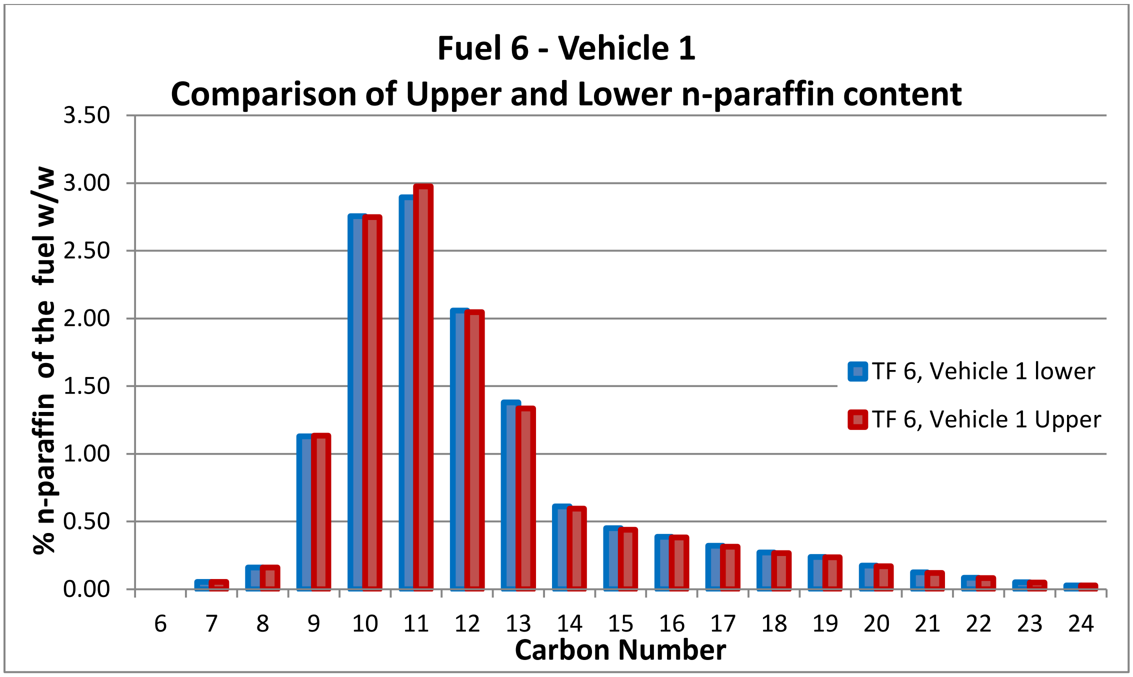


Figure C11a: Comparison of Upper sample and lower sample n-paraffin content (fuel taken just prior to vehicle test cycle). Fuel 6 Vehicle 1

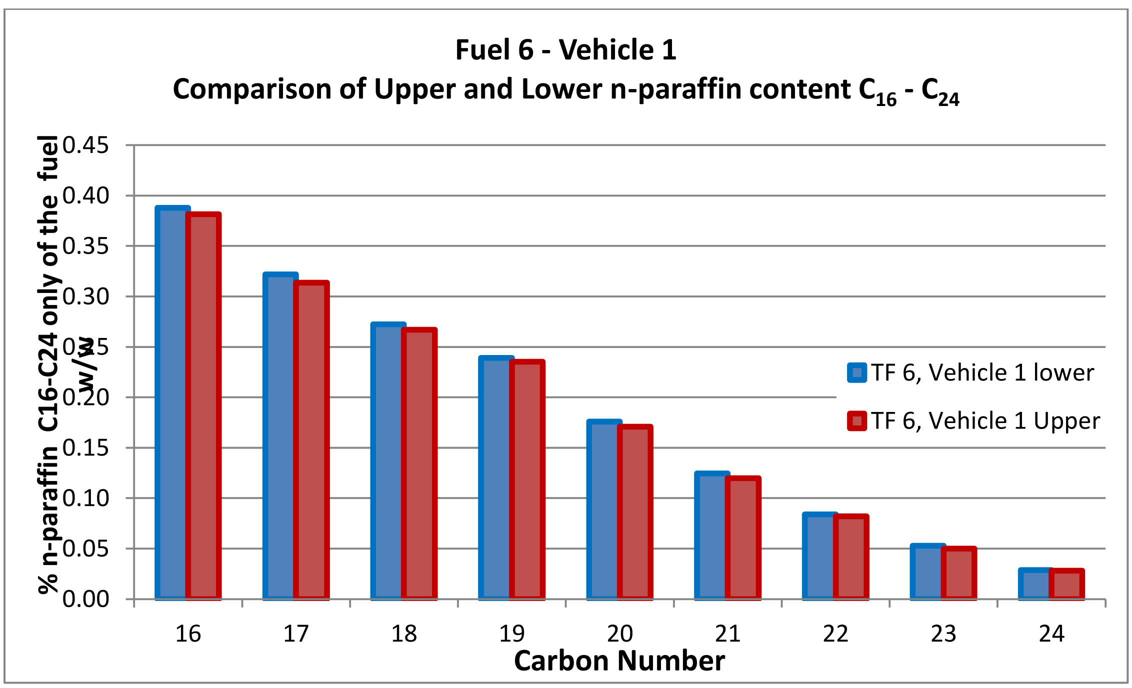


Figure C11b: Comparison of Upper sample and Lower sample n-paraffin content C_{16}-C_{24} taken just before vehicle test cycle. Fuel 6 Vehicle 1

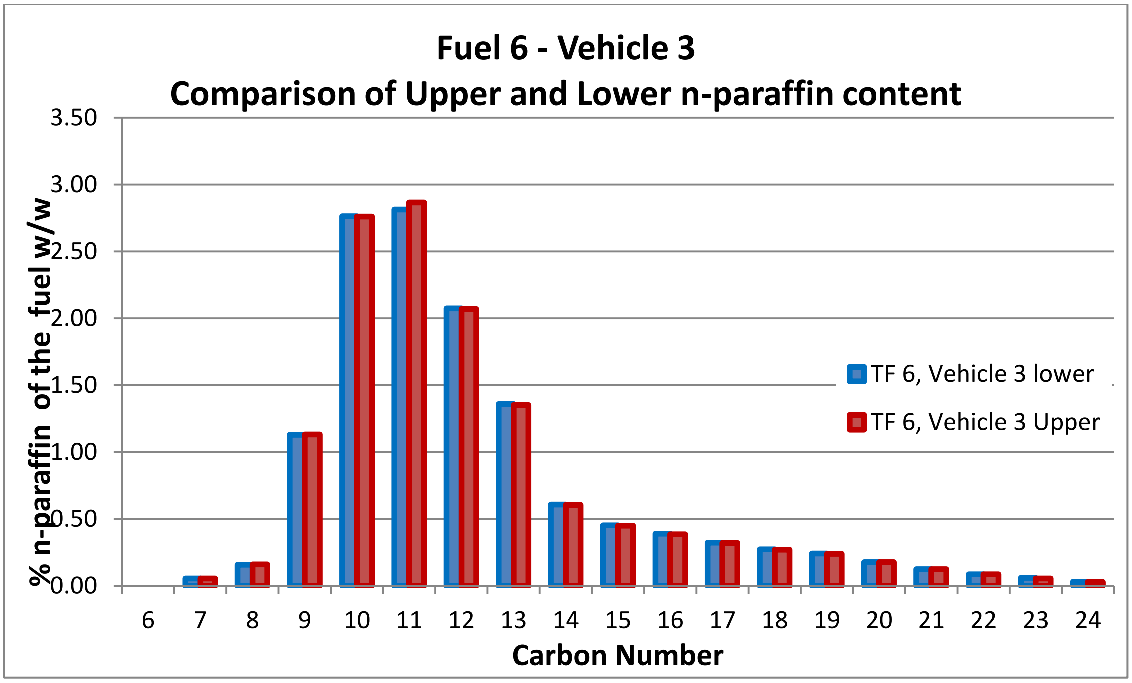


Figure C12a: Comparison of Upper sample and lower sample n-paraffin content (fuel taken just prior to vehicle test cycle). Fuel 6 Vehicle 3

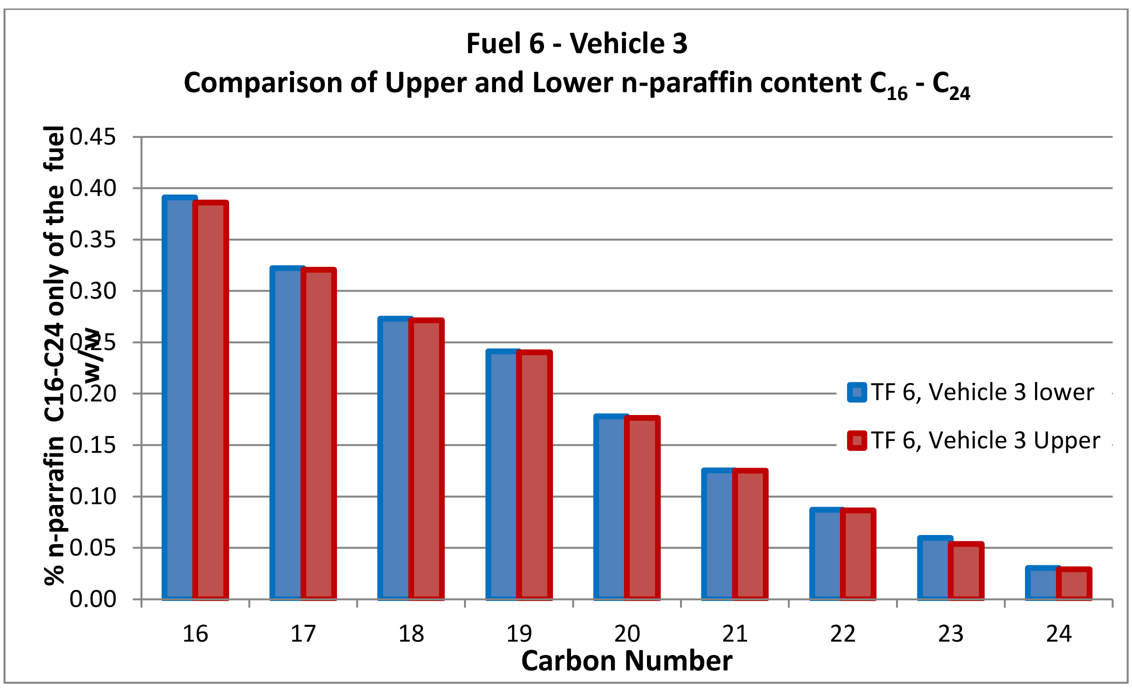


Figure C12b: Comparison of Upper sample and Lower sample n-paraffin content C_{16}-C_{24} taken just before vehicle test cycle. Fuel 6 Vehicle 3

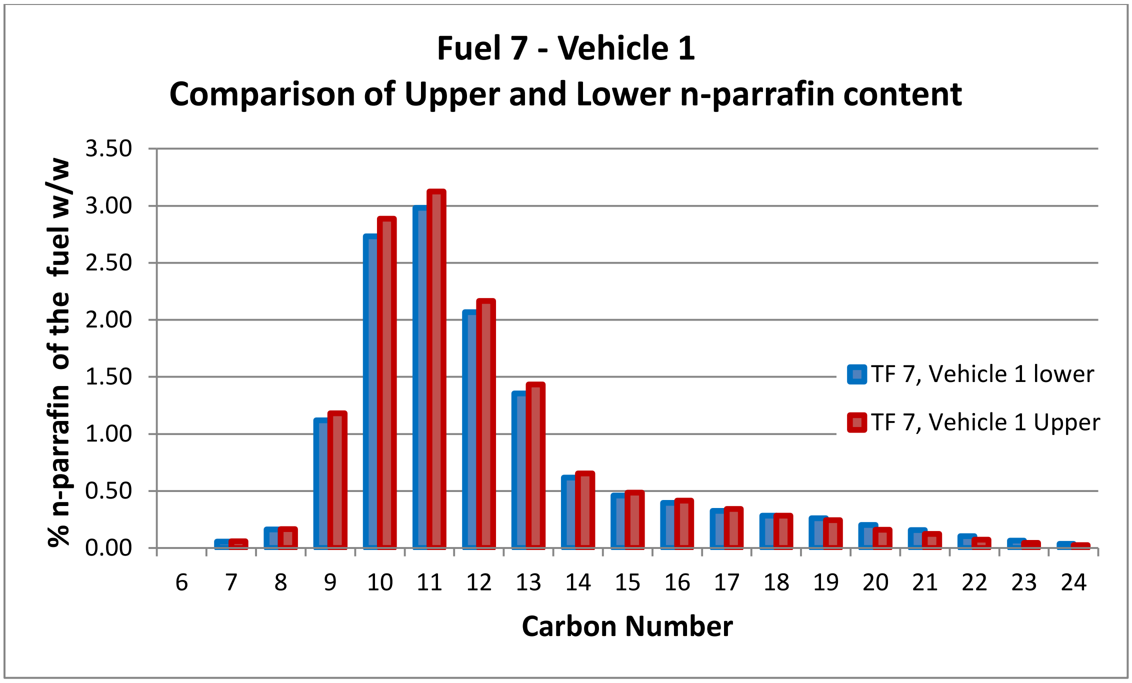


Figure C13a: Comparison of Upper sample and lower sample n-paraffin content (fuel taken just prior to vehicle test cycle). Fuel 7 Vehicle 1

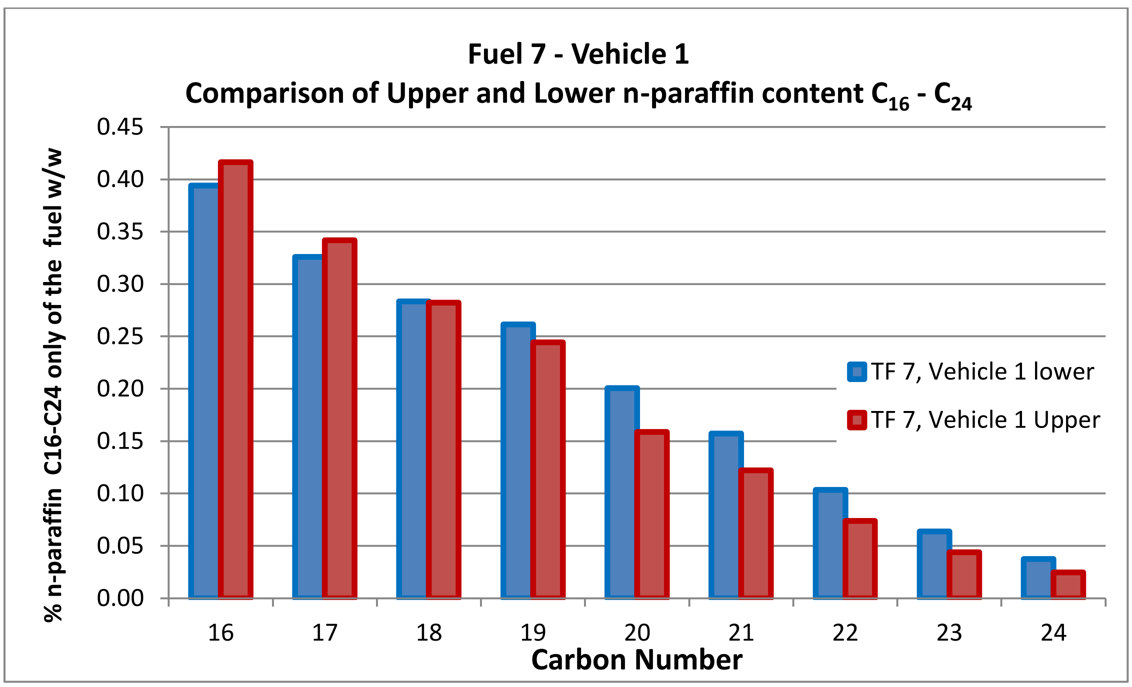


Figure C13b: Comparison of Upper sample and Lower sample n-paraffin content C_{16}-C_{24} taken just before vehicle test cycle. Fuel 7 Vehicle 1

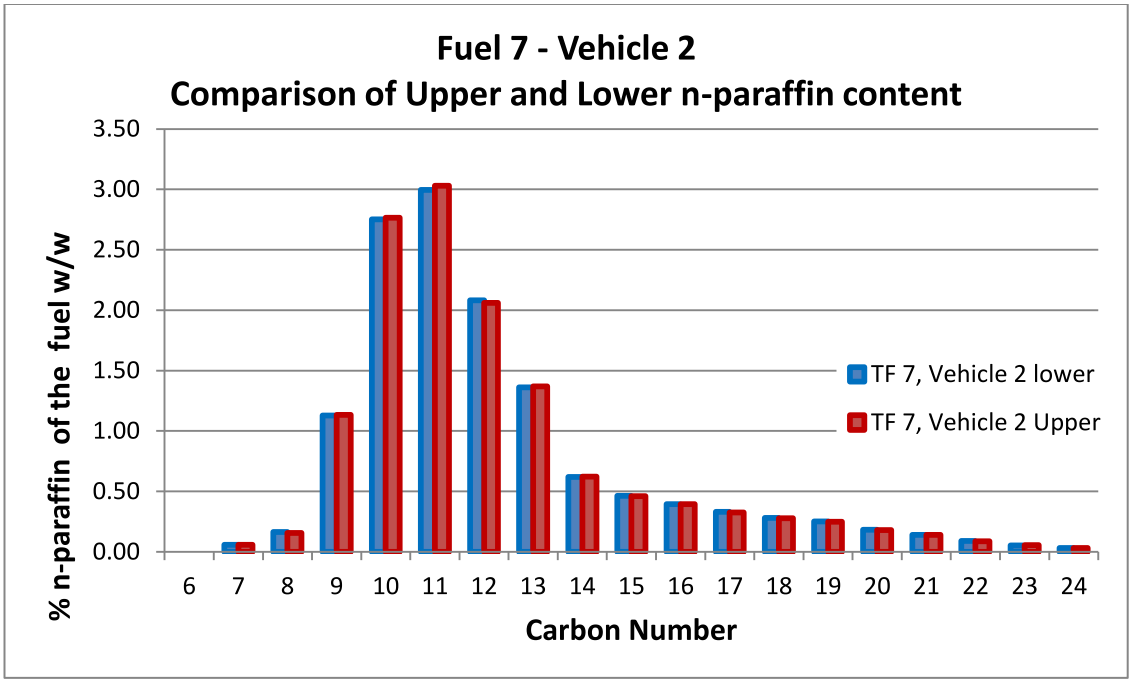


Figure C14a: Comparison of Upper sample and lower sample n-paraffin content (fuel taken just prior to vehicle test cycle). Fuel 7 Vehicle 2

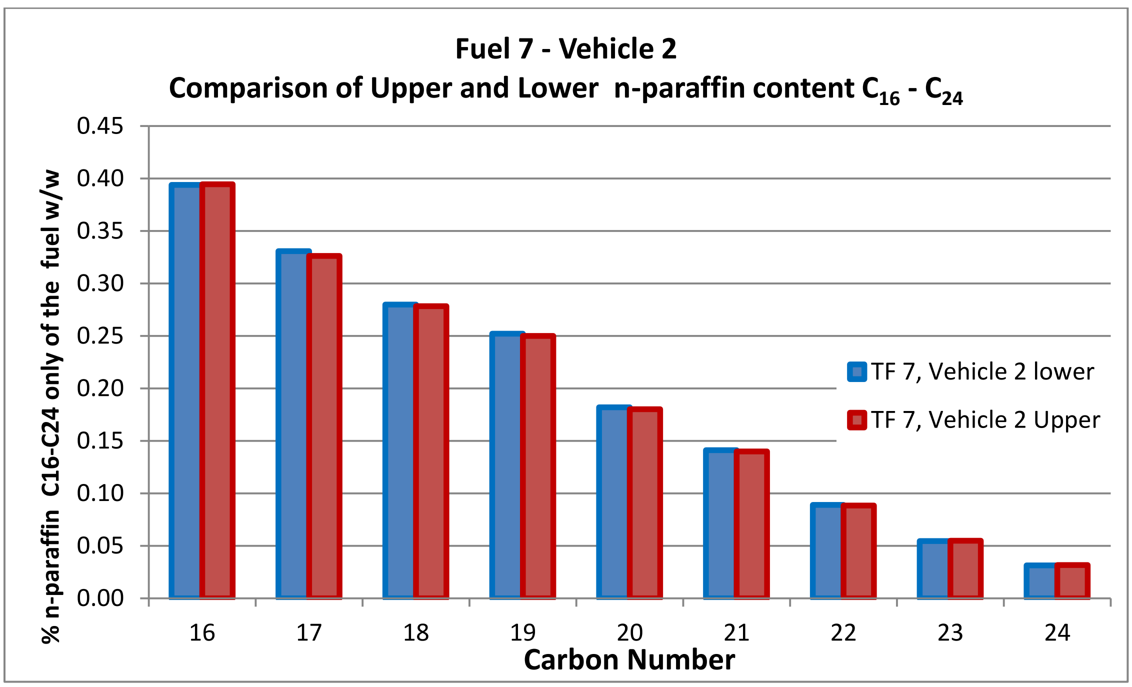


Figure C14b: Comparison of Upper sample and Lower sample n-paraffin content C_{16}-C_{24} taken just before vehicle test cycle. Fuel 7 Vehicle 2

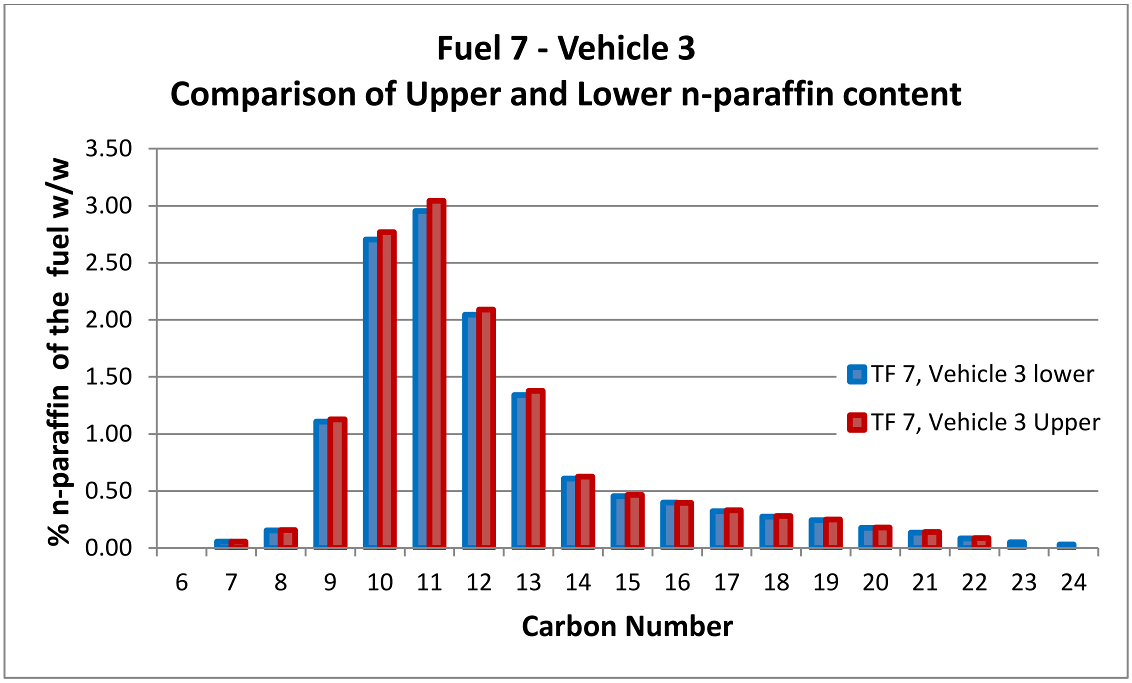


Figure C15a: Comparison of Upper sample and lower sample n-paraffin content (fuel taken just prior to vehicle test cycle). Fuel 7 Vehicle 3

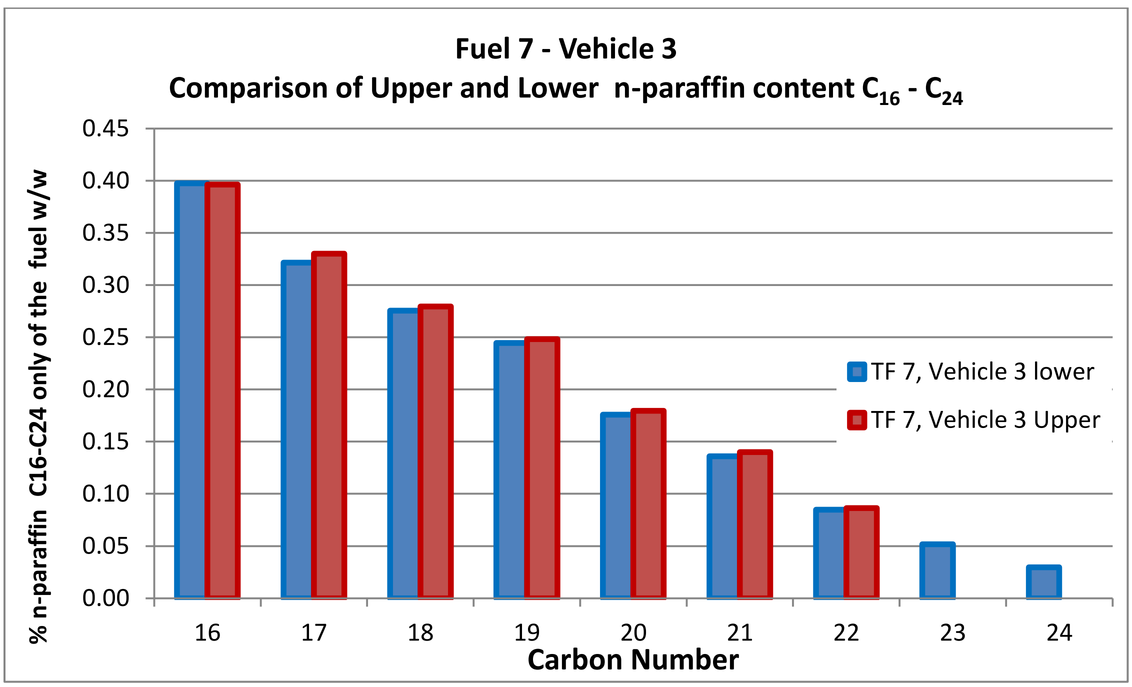


Figure C15b: Comparison of Upper sample and Lower sample n-paraffin content C_{16}-C_{24} taken just before vehicle test cycle. Fuel 7 Vehicle 3

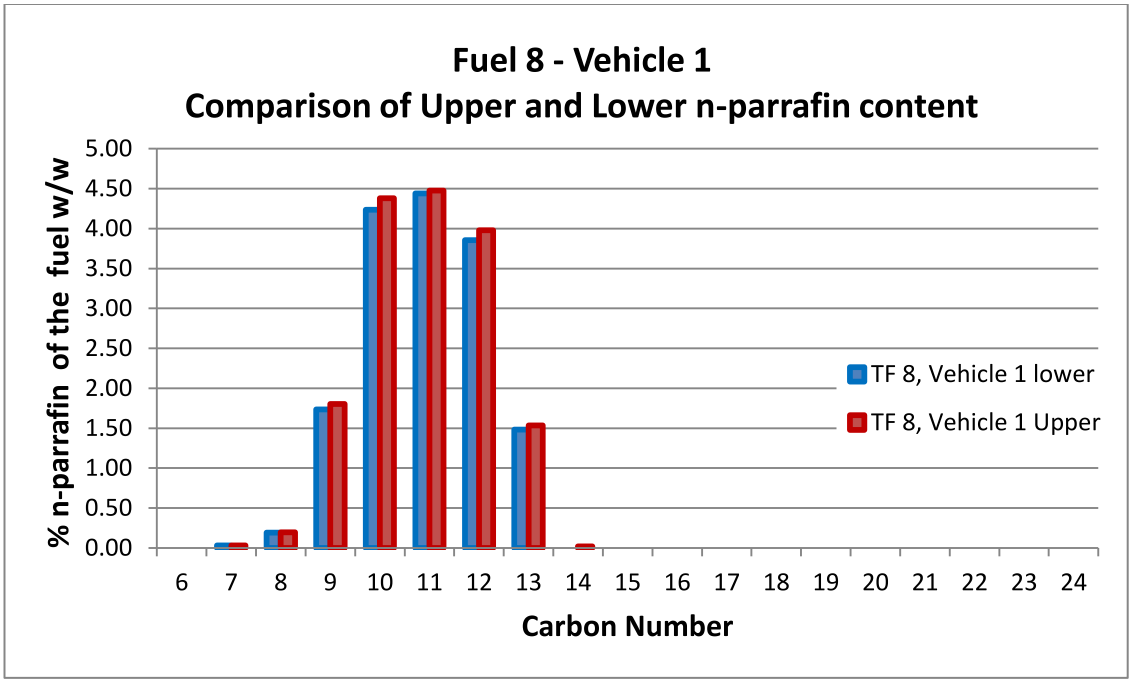


Figure C16a: Comparison of Upper sample and lower sample n-paraffin content (fuel taken just prior to vehicle test cycle). Fuel 8 Vehicle 1

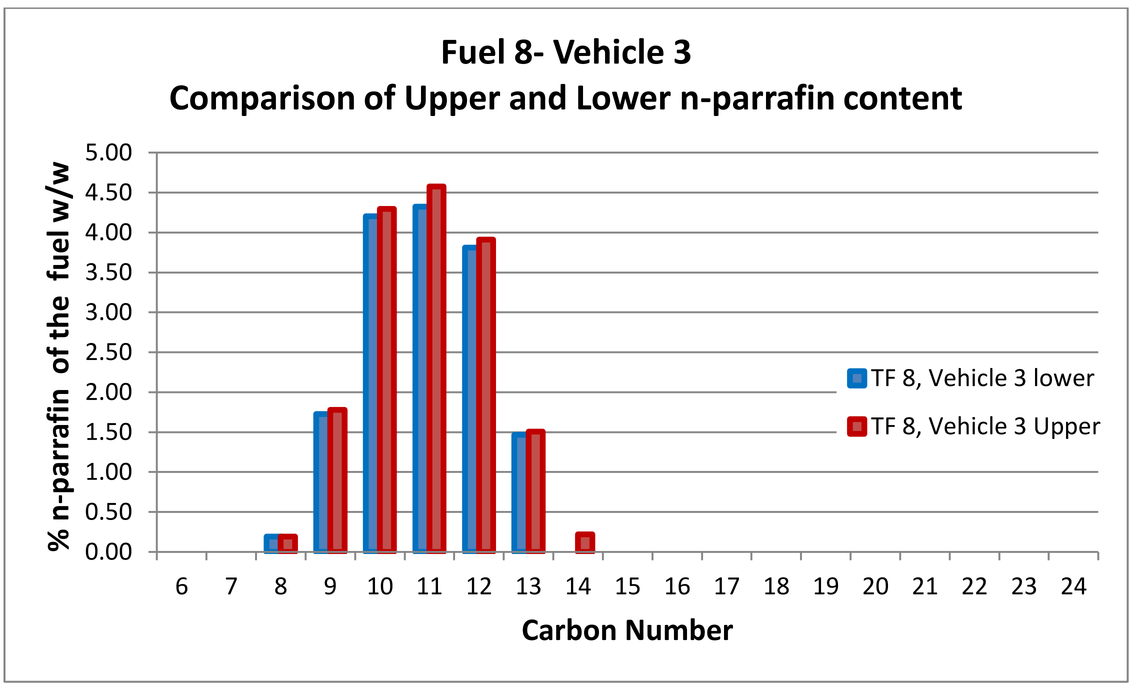


Figure C17a: Comparison of Upper sample and lower sample n-paraffin content (fuel taken just prior to vehicle test cycle). Fuel 8 Vehicle 3

Appendix D: Comparison of Upper and Lower Fuels Samples DSC Curves

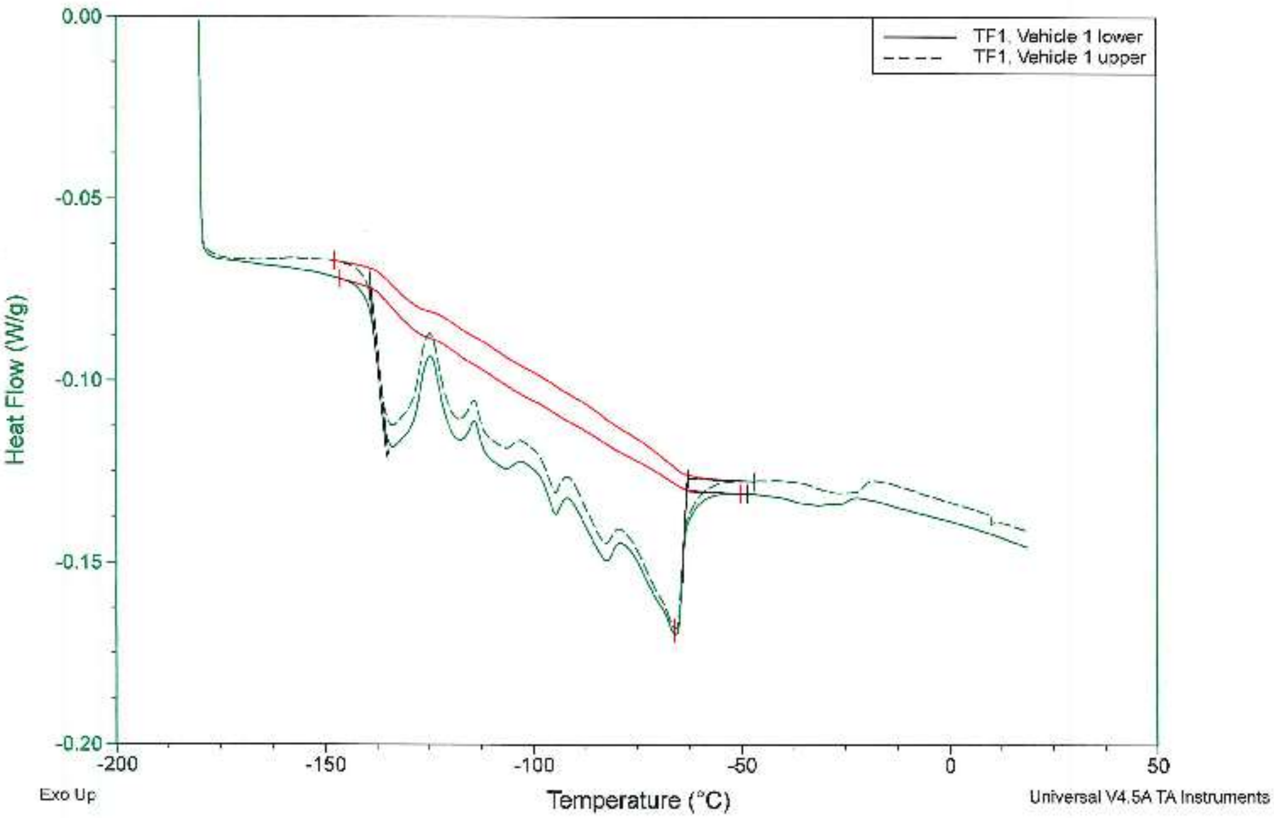


Figure D1: Comparison of upper sample and lower sample of DSC curves taken just before vehicle test cycle. Fuel 1 Vehicle 1

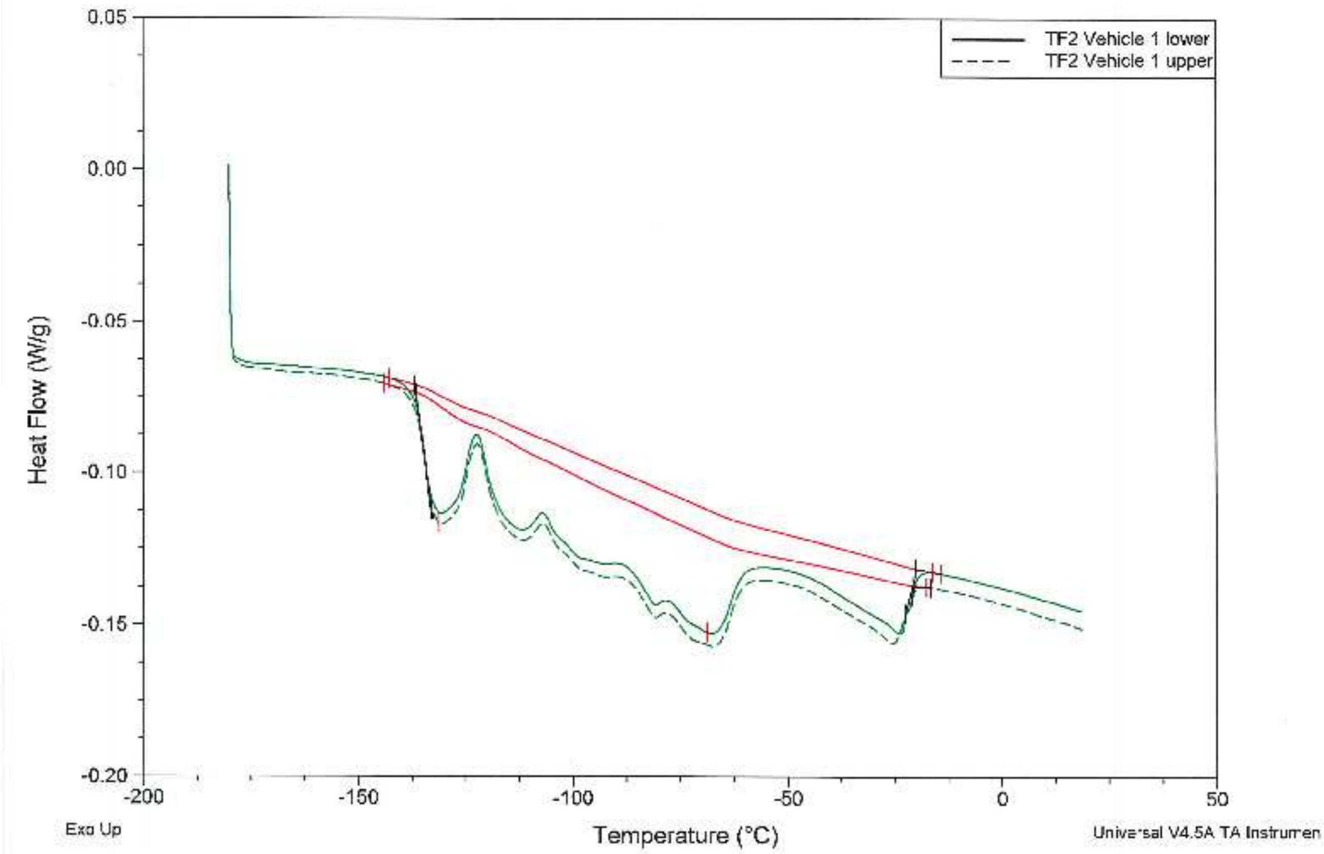


Figure D2: Comparison of upper sample and lower sample of DSC curves taken just before vehicle test cycle. Fuel 2 Vehicle 1

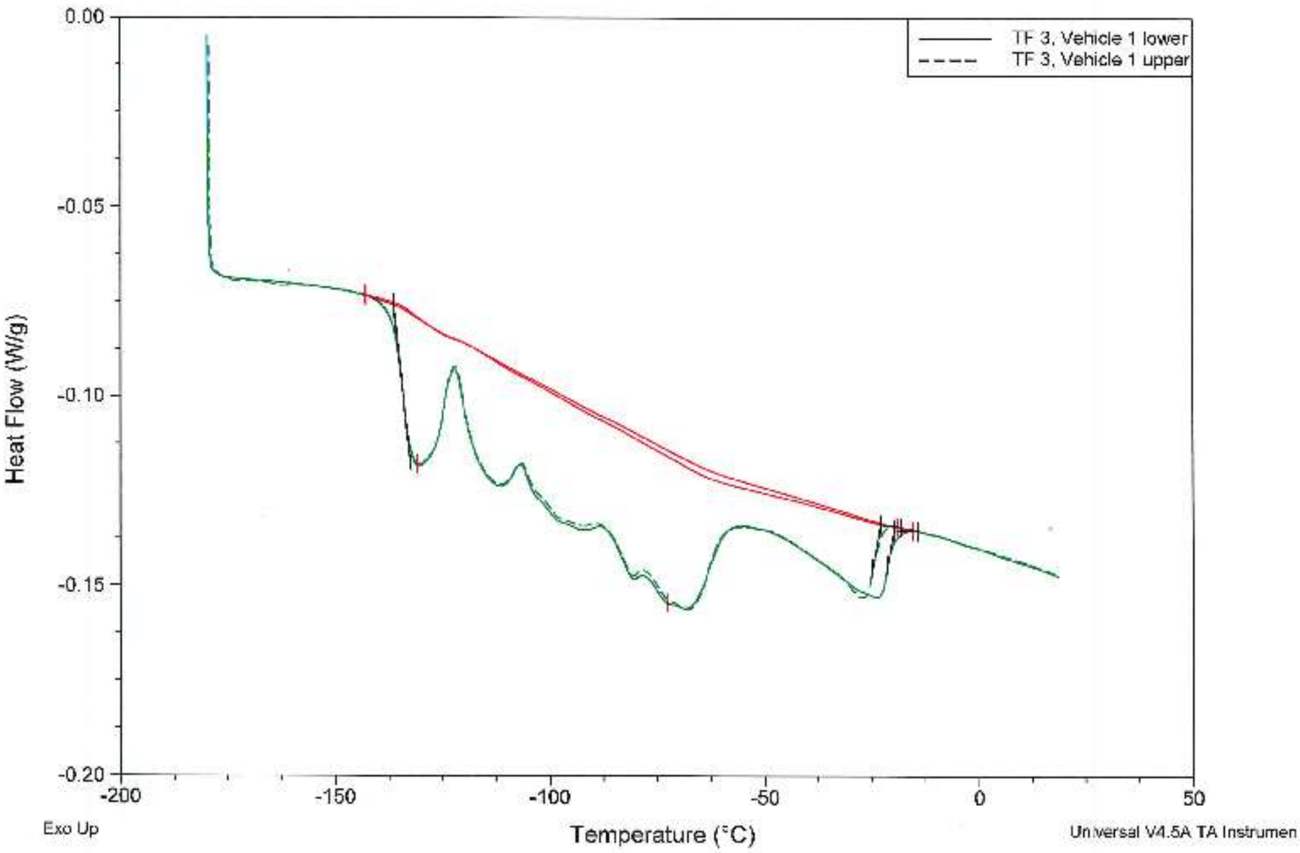


Figure D3: Comparison of upper sample and lower sample of DSC curves taken just before vehicle test cycle. Fuel 3 Vehicle 1

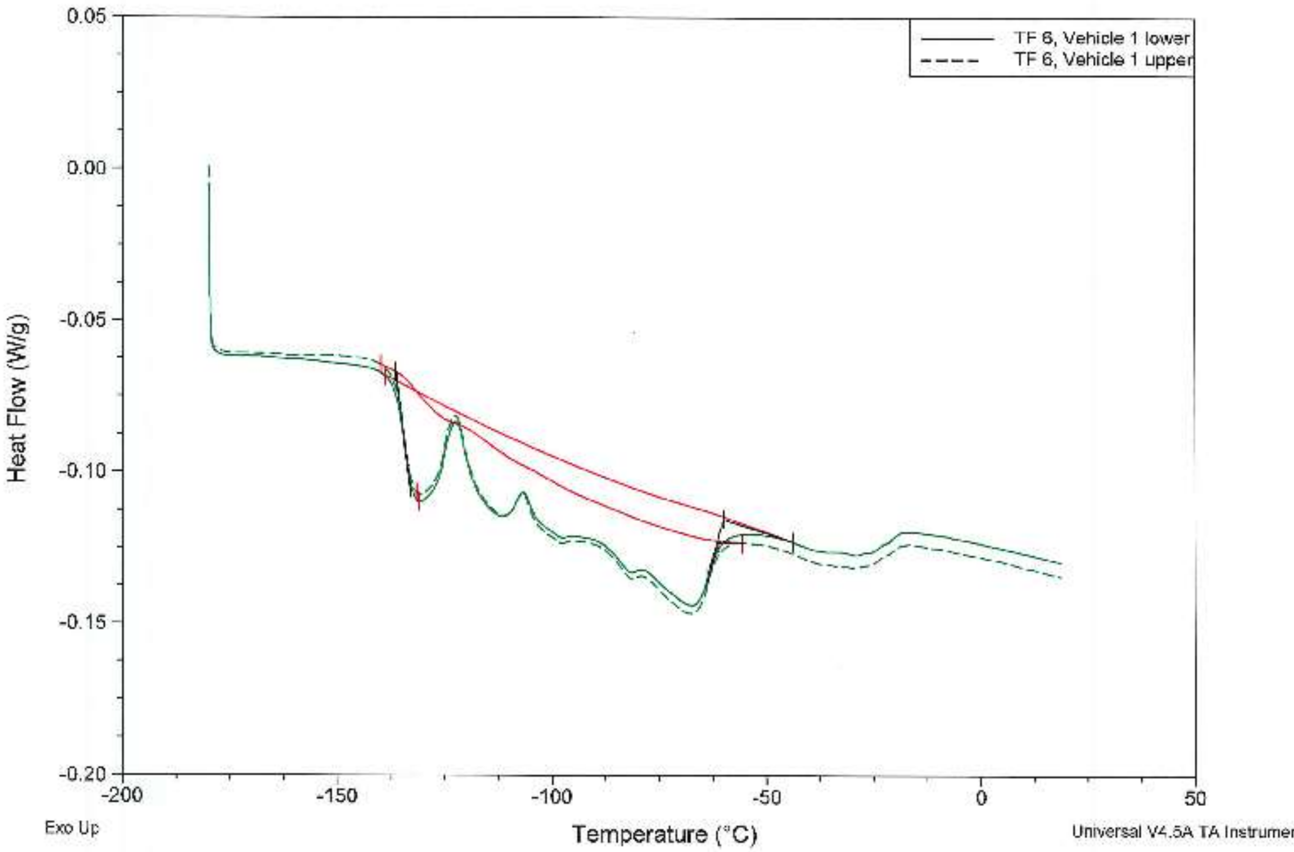


Figure D4: Comparison of upper sample and lower sample of DSC curves taken just before vehicle test cycle. Fuel 6 Vehicle 1

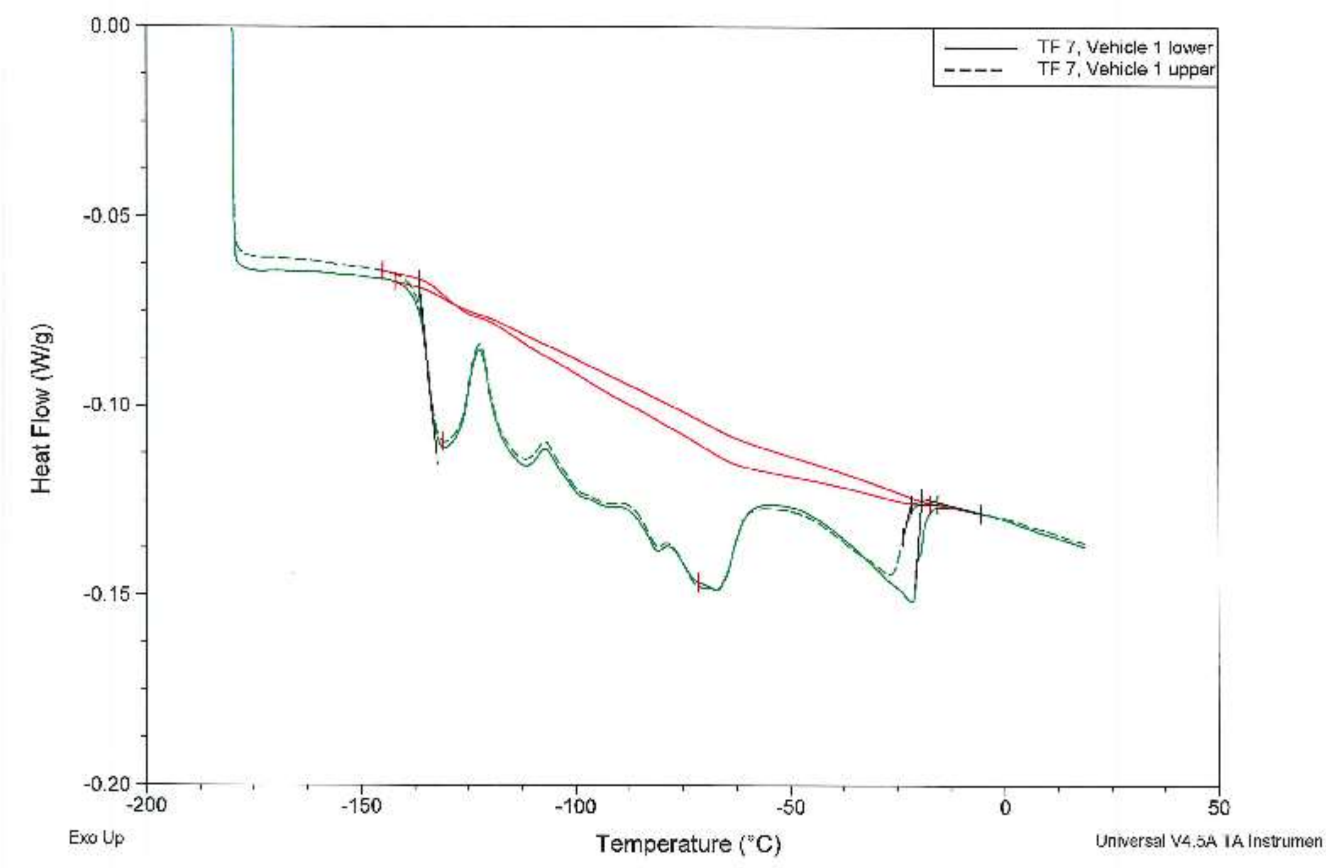


Figure D5: Comparison of upper sample and lower sample of DSC curves taken just before vehicle test cycle. Fuel 7 Vehicle 1

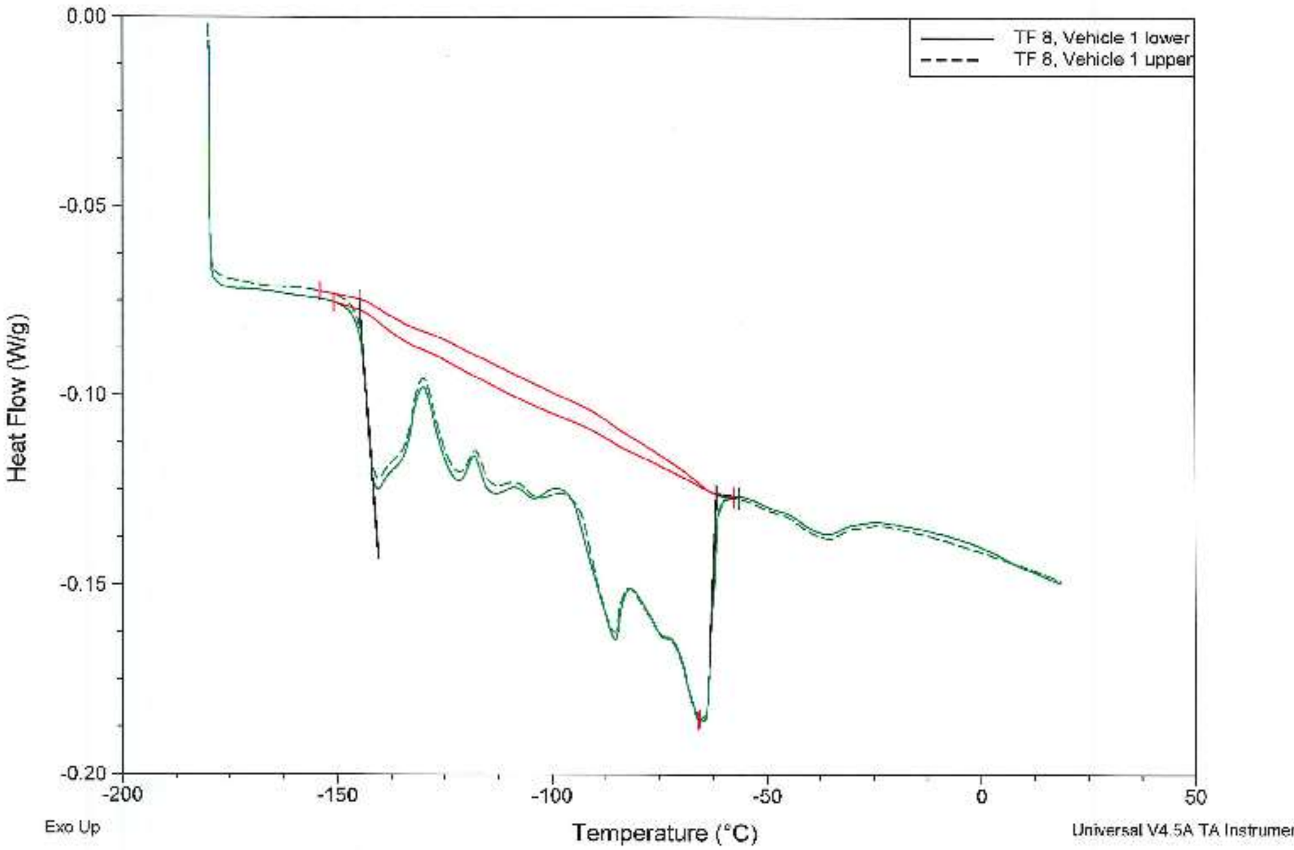


Figure D6: Comparison of upper sample and lower sample of DSC curves taken just before vehicle test cycle. Fuel 8 Vehicle 1

Appendix E: Summary of this cold soak fidelity data and statistics

Table E1. Comparison of actual fuel temperature and start temperature relative to the targeted project temperatures

			Diurnal Cycle Lowest Fuel Tank temperature (°C)		Starting Cycle (°C)		
Vehicle	**Fuel**	**Cloud $Point_{AVG}$ (°C)**	**Lowest Temp Achieved**	**CP ∆ from Lowest Temp Achieved**	**Temp at Start**	**CP ∆ from Target Start Temp**	**Notes**
1	1	-28	-33	-5	-22	6	
1	2	-22	-25	-3	-20	2	
1	3	-22	-26	-4	-21	1	
1	4	-22	-29	-7	-20	2	
1	6	-24	-25	-1	-21	3	
1	7	-21	-25	-4	-19	2	
1	8	-41	-38	3	NA	NA	NO DIURNAL CYCLE, Start-Up Failed
2	1	-28	-33	-5	-25	3	
2	2	-22	-26	-4	-20	2	
2	4	-22	-25	-3	-17	5	
2	7	-21	-14	8	-6	15	
3	1	-28	-34	-6	-26	2	
3	2	-22	No data		-26	-4	NO Cold Soak Data, Cannot Tell if Diurnal Cycle
3	4	-22	-17	5	-12	10	
3	6	-24	-17	7	-16	8	NO DIURNAL CYCLE, Start-Up Attempted
3	7	-21	-22	-1	-16	5	
3	8	-41	-39	2	-37	4	NO DIURNAL CYCLE, Start-Up Attempted

Key:
- (pink) indicates achieved temperature does not meet test criteria
- (yellow) indicates achieved temperature is borderline meeting test criteria
- (green) indicates achieved temperature met test criteria

Statistics: Cold Soak Temperatures

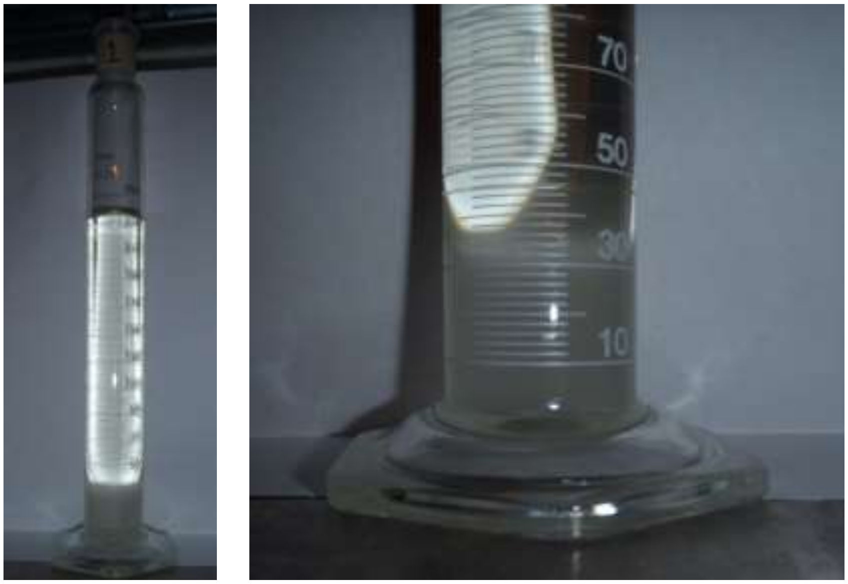
Total Number of Cold Soaks	17	
Number with Diurnal Cycle	13	76.5%
Number where Diurnal Met Temperature Target	0	0.0%

Number with Diurnal Cycle, where fuel temperature at Start-Up met test criteria	6	46.2%

As mentioned previously there were significant test facility problems, which resulted in inability to achieve the desired cold soak temperature profile simulating a 'three-day weekend cold diurnal cycle' needed to induce wax settling. A summary of this cold soak fidelity data and statistics are shown in Table E1. A total of 17 cold soak/vehicle runs were attempted in the program. Of these, 3 cold soak cycles did not feature diurnal simulation, and 1 cycle had no data acquisition of the cold soak -- so it cannot be determined if diurnal simulation was achieved. These four cold soaks are considered failures. Of the 13 cold soak runs that featured diurnal simulation, none met the desired criteria where the minimum temperature achieved must be 10°C below the test fuel cloud point. Therefore every cold soak cycle attempted failed to meet the desired test criteria. Therefore the operability data (fuel filter ΔP, RPM, etc.) acquired in runs where the engine started cannot be considered representative of the program's intent.

Appendix F: Photo graphs of fuels left in the chamber at the end of the diurnal cycle.

The photos below represent fuel that was in the chamber throughout the diurnal cycle. Photos were taken prior to the start cycle.

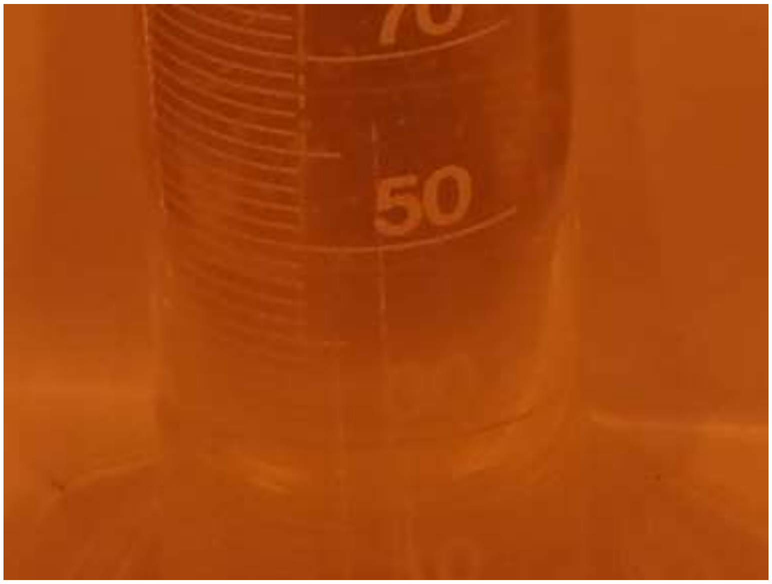


Full view Close up of bottom 25%

Figure F1: Fuel 1

Full view Close up of bottom 25%

Figure F2: Fuel 2



Full view Close up of bottom 30%

Figure F3: Fuel 4

Full view Close up of bottom 25%

Figure F4: Fuel 6

Full view | Close up of bottom 25%

Figure F5: Fuel 7

Full view | Close up of bottom 25%

Figure F6: Fuel 8