CRC Report No. 650

Biodiesel Blend Low-Temperature Performance Validation

CRC Project: DP-2a-07

June 2008

COORDINATING RESEARCH COUNCIL, INC.
3650 MANSELL ROAD·SUITE 140·ALPHARETTA, GA 30022

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Executive Summary

The objective of this effort was to validate that the cold soak filtration test is able to identify biodiesel that, in blends up to B20, shows precipitate formation and fuel filter plugging at temperatures above the cloud point (CP). It is believed that biodiesel blends from B100 with long cold soak filtration time (CSFT) can form precipitates and be visually cloudy after extended cooling (on the order of 16 hours) at temperatures above cloud point; and that these precipitates can cause low-temperature operability problems. A further objective of the study was to evaluate the correlation between bench test results; namely, CP, cold filter plugging point (CFPP), and low temperature flow test (LTFT) and actual heavy-duty (HD) vehicle performance at low temperatures for biodiesel blends. To that end, four B100 materials were tested as B5 and B20 blends in three Class 8 trucks, each believed to represent approximately 30% of the North American Class 8 truck market. The B100 and blends were characterized in detail by multiple laboratories. Three of the B100 exhibited an average CSFT of less than 200 seconds, while the fourth had CSFT >720 seconds. For each fuel/truck combination, we attempted to bracket the low-temperature operability limit and thereby calculate an estimated minimum operating temperature (EMOT). These results were compared to the predicted operability limit based on CP, CFPP, and LTFT.

Cloud point was an accurate or conservative predictor of EMOT in almost all cases. The one exception was TF#5, a B20 blend produced from the high CSFT B100. On average for all fuels combined CP was conservative by about 1°C over the full range of CP examined. CFPP tends to predict an operability limit higher than cloud and may not be protective of the most severe vehicles. LTFT was protective of all vehicles but was very conservative for B20 from high CSFT biodiesel.

Overall, biodiesel blends produced from B100 with CSFT <200 seconds performed as predicted by CP or LTFT measurements. These fuels were also clear and bright above CP and did not cause significant fuel filter blockage and pressure drop above CP. In many cases, these fuels could operate at temperatures 1°C to 2°C below CP.

The B5 blend prepared from the high CSFT B100 appeared cloudy at a test temperature above its CP. In spite of the cloudiness above CP, all three vehicles were able to operate at very near to or below CP on this fuel. Passing tests exhibited continuously increasing fuel filter pressure drop for this fuel in Truck A, even at temperatures slightly above CP. This was not observed for passing tests with other B5 blends. The cloudiness of this test fuel above CP, combined with the increasing pressure drop observed for this fuel throughout the passing test, suggests that it could cause operability problems in situations more severe than those simulated here. The B100 was produced by blending a high CSFT B100 with a second B100 having CSFT <200 seconds. A B100 with a naturally high CSFT may have caused operability problems above the CP at the B5 level.

The B20 blend prepared from the high CSFT B100 was slightly cloudy at -15°C and showed obvious precipitates at -17°C, yet the CP of this fuel was -18°C where the fuel appeared almost milk-white. Failing tests were obtained in Truck A at all temperatures tested, up to 3°C above CP. The truck manufacturer represents roughly 30% of North American Class 8 trucks, implying that B100 with high CSFT may cause unanticipated low-temperature operability problems for B20 blends.

This study showed that high CSFT B100 can cause low-temperature operability problems at temperatures above CP for B20 blends. It is recommended that additional testing of B5 blends from other, potentially more representative, high CSFT B100 be performed.

It was shown that movement of the trucks could cause mixing of the vehicle fuel tank and suspension of materials that had precipitated to the bottom causing a failing test. This may indicate that vehicle dynamometer tests do not capture all failure modes that can be experienced in actual vehicle operation.

Acknowledgements

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Renewable Energy Group
Sanimax Energy
Stepan Company
Southwest Research Institute
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Acronyms and Abbreviations

ADM	Archer Daniels Midland
ASTM	ASTM International, at standards setting organization
AWCD	All-weather chassis dynamometer
Bxx	Biodiesel blend containing xx volume percent biodiesel
BP	BP Corporation
CFPP	Cold filter plugging point
CP	Cloud point
CRC	Coordinating Research Council
CSFT	Cold soak filtration time
EMOT	Estimated minimum operability temperature
EN	European Normalization
FAME	Fatty acid methyl ester
GM	General Motors Corporation
HD	Heavy-duty
IOC	Imperial Oil Corporation
kph	kilometer per hour
LD	Light-duty
LTFT	Low-temperature flow test
PTFE	polytetrafluoroethylene
USDOE	United States Department of Energy

1. Introduction

Like other fuels, diesel fuels must perform under a range of environmental conditions throughout the year. To accomplish this, petroleum-derived diesel is formulated to have a lower CP during colder months with significantly lower CP fuels required in northern tier states and colder areas. Tenth percentile minimum ambient air temperatures are shown in an appendix to the ASTM D975 Standard Specification for Diesel Fuel Oils[1] and are used to estimate low-temperature operability requirements. This is most commonly done by ensuring that fuel CP is below the tenth percentile minimum ambient air temperature. Note that for petroleum-derived diesel fuels flow improver additives allow operability at temperatures significantly below CP.

Biodiesel blend operability may be complicated by the observation that some biodiesel blendstock can form precipitates at temperatures above CP. The objectives for this study were to validate a cold temperature performance test to identify biodiesel that, in blends up to B20, shows precipitate formation and fuel filter plugging at temperatures above the CP. A further objective was to evaluate the correlation between bench test results (CP, CFPP, etc.) and actual HD vehicle performance at low temperatures for biodiesel blends.

[1] 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.

2. Background

A recent CRC study examined the low-temperature performance of a range of petroleum-derived diesel fuels in modern diesel vehicles, and compared the measured operability limit with predictive tests such as CP, CFPP, and LTFT.[2] The results showed large differences in performance among vehicles and fuels. CP was the most conservative estimate of performance and protected all the vehicles; however, considerable operability was observed below CP. LTFT was the next best test for protecting all vehicles. All tests showed significantly more performance give away in the higher CP fuels than in the lower CP fuels.

Some years ago Chandler conducted a study of low-temperature operability of both light-duty (LD) and HD trucks.[3] The focus was on comparing operability limits to laboratory bench tests such as LTFT. The study revealed that LTFT was developed to predict operability for the most severe engine fuel-system from a low-temperature operability standpoint (associated with Cummins engines which were the predominant engines in the HD market at that time). Chandler found that the LD vehicles tested 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.[4] It was shown that changes to filter location, porosity, and to fuel circulation rate can significantly affect low-temperature operability. LTFT continued to be a good predictor of the operability limit, particularly when additives were used.

Up to this time, no similar studies have been done to evaluate the low-temperature performance of biodiesel blends in comparison to results from predictive tests. Biodiesel consists of mono-alkyl esters of fatty acids (typically methyl esters) and is produced by transesterification of lipid feedstocks such as vegetable oil, animal fat, and waste grease in the presence of an alcohol. For legal use in the United States, biodiesel must meet the requirements of ASTM D6751 Standard Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels.[5] Biodiesel CP ranges from around -5ºC to almost 20ºC depending upon the degree of saturation of the feedstock.[6] Biodiesel is commonly used as a blend with petroleum diesel at levels up to 20 volume percent. Blending of

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[2] 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.

[3] 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).

[4] 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).

[5] ASTM D6751 Standard Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels. Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

[6] Kinast, J.A., Production of Biodiesels from Multiple Feedstocks and Properties of Biodiesels and Biodiesel/Diesel Blends. NREL/SR-510-31460, March 2003.

biodiesel into diesel fuel will typically cause an increase in CP, with the magnitude of the increase dependent on both the biodiesel and petroleum diesel 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 unsaponifiable matter. Unsaponifiable matter is non-lipid materials (i.e., materials that cannot be converted to soap) which are mainly plant sterols, tocopherols, and hydrocarbons. A typical soy oil feedstock may contain as much as 1.6 mass% unsaponifiable matter. Van Gerpen and coworkers examined the effect of unsaponifiable matter on CP for B100 and for a B20 blend.[7] At 2 mass% unsaponifiable content, they observed no effect on B100 or blend CP. However, increasing the level to 3% caused a more than 10ºC increase in B100 CP but with no measurable effect on B20 CP. The same study showed that as little as 0.05 mass% saturated monoglyceride could increase B100 CP by 2ºC. Mono- and di-glycerides are limited in D6751 by limiting the allowable level of total glycerin to 0.24 mass%. Total glycerin consists of free glycerin and bound glycerin. Bound glycerin is in the form of mono-, di-, and tri-glycerides and is reported as mass% glycerin. Typical biodiesel (such as those tested here) contains approximately 0.05 to 0.5 mass% monoglycerides, depending on feedstock and processing technology, and from 0.02 to 0.2 mass% saturated monoglycerides. Thus, these compounds can have a significant impact on B100 CP even for high quality samples meeting the specification limits.

Flint Hills Resources has also examined the effect of impurities on biodiesel low-temperature performance measures.[8] They reported significant fuel filter plugging issues for customers during the winter of 2005/2006. During the winter of 2006/2007, a cold soak filtration test (similar to that described in more detail below) was implemented that was believed to identify biodiesel containing one type of impurity that can cause low-temperature operability issues. This produced a dramatic performance improvement. The exact nature of this impurity has not been determined. They also presented evidence that B2.5 dispenser filters could become blocked by saturated monoglycerides as temperatures approached -18ºC (0ºF) for saturated monoglyceride content above about 0.07 mass%, although this evidence was not conclusive.

The cold soak filtration test used currently involves chilling 300 ml of the B100 at 4.4°C (40°F) for 16 hours then allowing the B100 to warm to between 20°C to 22°C. The B100 is then filtered through a 47 mm diameter glass fiber filter having 0.7 micron pore size under 585 torr of vacuum, and the time to filter is recorded. ADM has reported results of a statistically designed study where the effect of several impurities on the CSFT of a distilled soy-biodiesel was evaluated.[9] The impurities examined were sterol

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[7] Van Gerpen, J.H., Hammond, E.G., Yu, L., Monyem, A. "Determining the Influence of Contaminants on Biodiesel Properties" *SAE Technical. Paper No*. 971685 (1997).
[8] Selvidge, C., Blumenshine, S., Campbell, K., Dowell, C., Stolis, J. "Effect of Biodiesel Impurities on Filterability and Phase Separation from Biodiesel and Biodiesel Blends" *Proc. Of the 10th International Conference on Stability, Handling, and Use of Liquid Fuels,* Oct 7-11, 2007; Tucson, AZ. www.iash.net.
[9] Pfalzgraf, L., Lee, I., Foster, J., Poppe, G. "Effect of Minor Components in Soy Biodiesel on Cloud Point and Filterability" *AOCS Inform Special Supplement; Biorenewable Resources No. 4,* September 2007, pp 17.

glucosides (plant sterol components of unsaponifiable matter), monoglycerides, soap, and dissolved water. Pfalzgraf, et al. found that the filtration time was highly sensitive to sterol glucoside and soap content; and that dissolved water could also interact with these impurities to increase filtration time. Monoglycerides had only a small effect on filtration time.

Thus, there remains considerable uncertainty about the impurities that may cause low-temperature operability problems in biodiesel blends. In the absence of detailed chemical identification, many biodiesel marketers have implemented the cold soak filtration tests as a performance metric to identify B100 that may cause operability problems. Additionally, this test is currently the subject of an ASTM ballot as an addition to the D6751 specification for B100. Here we examine the low temperature performance of B5 and B20 blends made from four B100 samples, including one with a long CSFT.

3. Approach

The testing program general concept is as follows:

- Testing of 9 fuels in 3 HD vehicles. Fuels include B5 and B20 blends from four biodiesel plus one base fuel. The four B100 cover a range of CP and include one sample with long cold-soak filtration time.
- Biodiesel was characterized for quality by multiple labs. Base fuel and blends were characterized for CP, CFPP, LTFT and other low temperature operability parameters by multiple labs.
- Vehicles were cooled to test temperature (initial test temperature was the CP) overnight then started and driven on an all-weather dyno. In subsequent tests, temperatures were adjusted to try to define an operability limit.
- Testing protocol was very close to that used in previous studies.[2,3]

Laboratory

The test program was conducted in the All Weather Chassis Dynamometer (AWCD) facility of Imperial Oil Company (IOC) in Sarnia, Ontario. The test temperature range of the AWCD was from 43°C to -40°C. The AWCD has a test cell housing the dynamometers and a pre-soak room that can hold up to three HD trucks. The test cell and the pre-soak room can be controlled at different temperatures at the same time and are capable of overnight soaking and testing three HD trucks per day. Test vehicles can be a mixture of Front Wheel Drive, Rear Wheel Drive, Four Wheel Drive, or in this case, tandem axle trucks. The computerized data acquisition system can record up to 250 channels of data at 10 times per second. The dynamometers have a Road-load Simulation Module that provides realistic loading on the test vehicle. The photographs in Figure 1, Figure 2, and Figure 3 show the test cell, the pre-soak room, and the control room of the facility with the test vehicles on a typical test day.

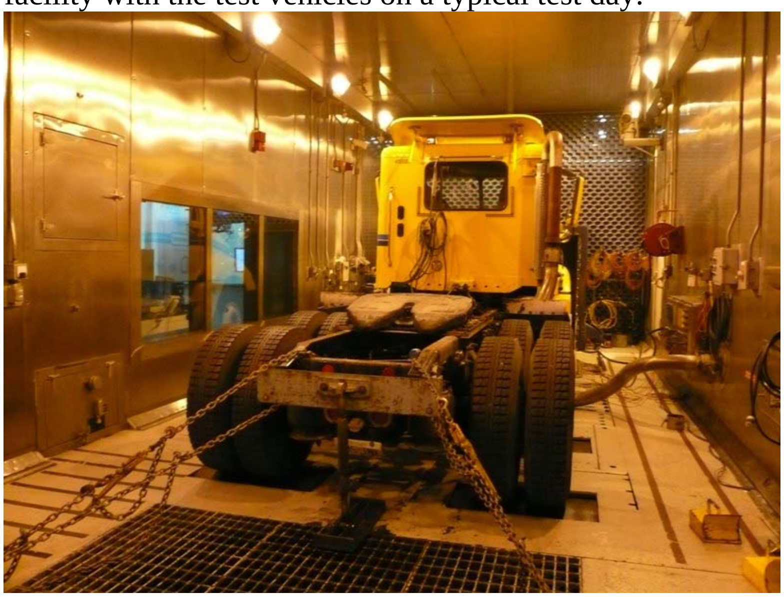


Figure 1. Class 8 truck in the AWCD test cell.

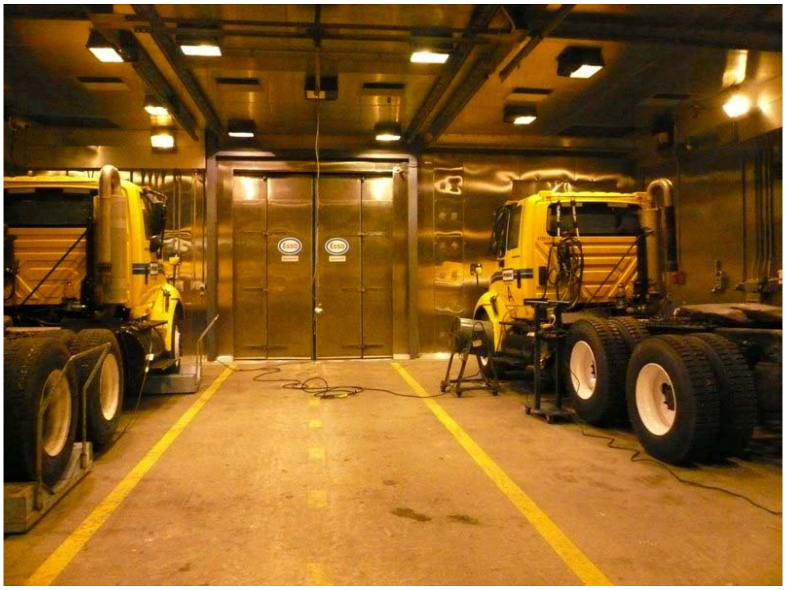


Figure 2. Two trucks in the pre-soak room, AWCD is through the double-doors in center.

Figure 3. Control room for AWCD.

Test Vehicles

Three test vehicle types were selected based on market representation, as shown in Table 1. These engines are believed to be representative of those with the largest market share in North American Class 8 trucks. For example, the Power Systems Research *PartsLink*™ Database indicates that the three engine manufacturers have approximately 75% of the total North American Class 8 market, respectively.[10] One additional vehicle was procured as a stand-by in case of vehicle failure.

[10] Power Systems Research, *PartsLink*™ Database, December (2007).

Table 1. Class 8 trucks procured for this program.

Test Vehicle	Model Year	Engine
International	2003	Caterpillar C-12
International	2005	Cummins ISM
International	2005	Cummins ISM
Freightliner	2003	Detroit Diesel Series 60

Fuel System and Fuel Filter Configurations

The three vehicles with different fuel system configurations were chosen to maximize the applicability of the test results. Fuel system design and fuel filter configurations have a large impact on low-temperature operability limit. Two test vehicle types have the primary fuel filter housing located outside of the engine compartment and behind the cab. The third vehicle type has the primary filter housing within the engine compartment. All primary filters run at negative gauge pressure. Only one engine has a secondary filter, normally run at positive gauge pressure. For Trucks A and C, the return fuel from the engine passes through the filter housing to warm the incoming fuel before being returned to the fuel tank. For Truck D an electric heater drawing approximately 50W is inserted in the fuel filter to warm the incoming fuel. Fuel filter configuration and filter micron size are listed in Table 2. For the Truck C engine, the truck as-received was not equipped with the primary and secondary filters recommended by the engine manufacturer. Table 2 indicates that these filters were changed to the recommended filters. Notably, these are of significantly larger micron size than those on the vehicle as-received. If this practice is common, the true operability limits for this truck will potentially be at significantly higher temperatures than measured in this study. The fuel systems of the vehicles are shown in schematic drawings indicating the locations of pressure and temperature sensors (Figure 4, Figure 5, and Figure 6).

Table 2. Fuel filter configuration and micron size.

Fuel Filters as Found on Rental Trucks

Truck #	Truck Make	Primary Filter Model	Mounting Location	Inlet fuel heated @ filter by:	Primary Filter Micron size	Secondary Filter Model	Secondary Filter Micron size	Mounting Location
A	International	Fleetguard FS19624	External, behind cab	Return fuel	7	N/A	N/A	N/A
B	International	Fleetguard FS19624	External, behind cab	Return fuel	7	N/A	N/A	N/A
C	International	Fleetguard FS19624	External, behind cab	Return fuel	7	Fleetguard FF5319	2	Engine compartment
D	Freightliner	Fleetguard FS19624	Engine compartment	Electric	7	N/A	N/A	N/A

Fuel Filters to be used in Tests

Truck #	Truck Make	Primary Filter Model	Mounting Location	Inlet fuel heated @ filter by:	Primary Filter Micron size	Secondary Filter Model	Secondary Filter Micron size	Mounting Location
A	International	Fleetguard FS19624	External, behind cab	Return fuel	7	N/A	N/A	N/A
B	International	Fleetguard FS19624	External, behind cab	Return fuel	7	N/A	N/A	N/A
C	International	Fleetguard FS19727	External, behind cab	Return fuel	10	Caterpillar 1R0749	4	Engine compartment
D	Freightliner	Fleetguard FS19624	Engine compartment	Electric	7	N/A	N/A	N/A

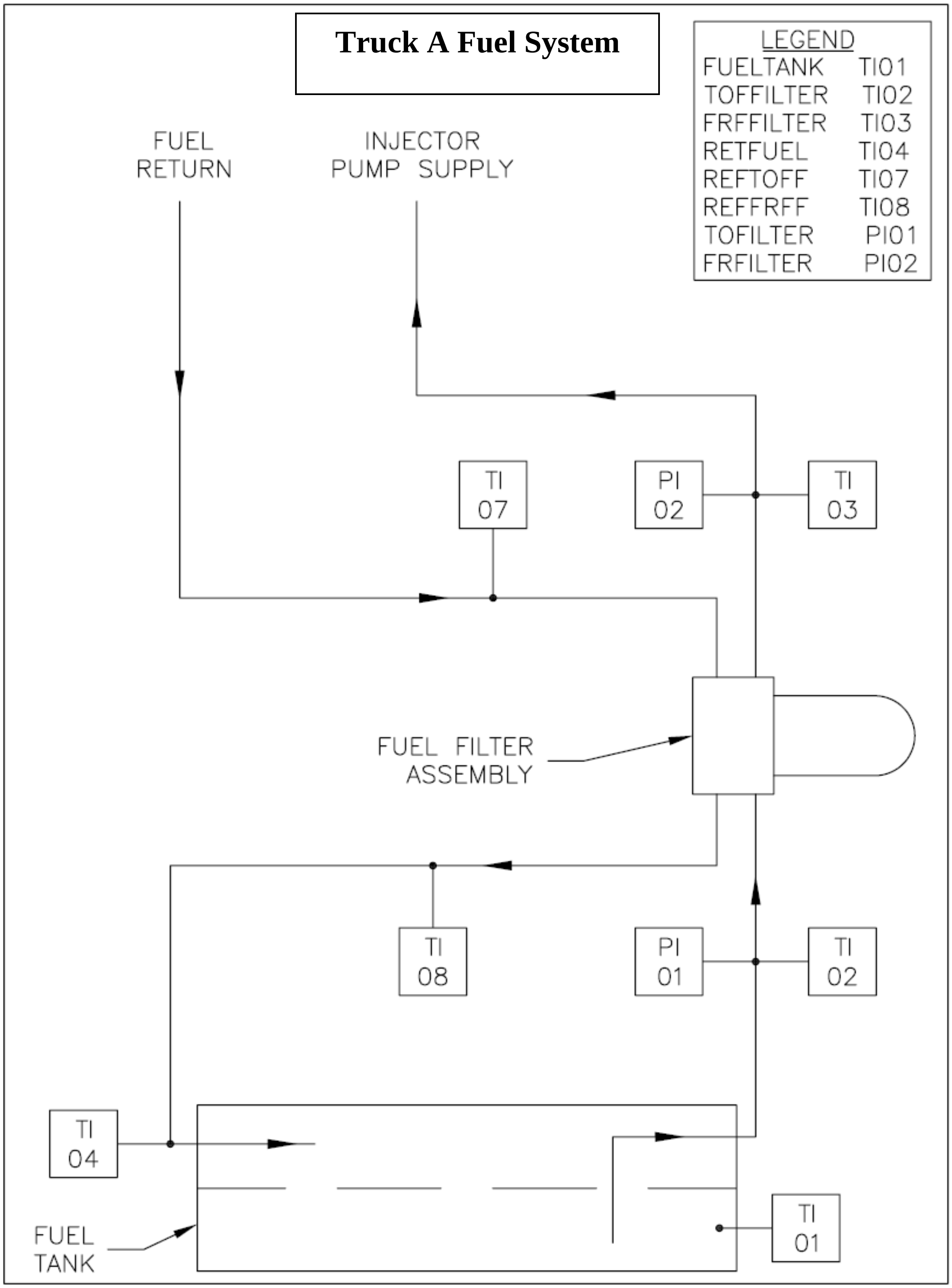


Figure 4. Vehicle fuel system diagram for Truck A.

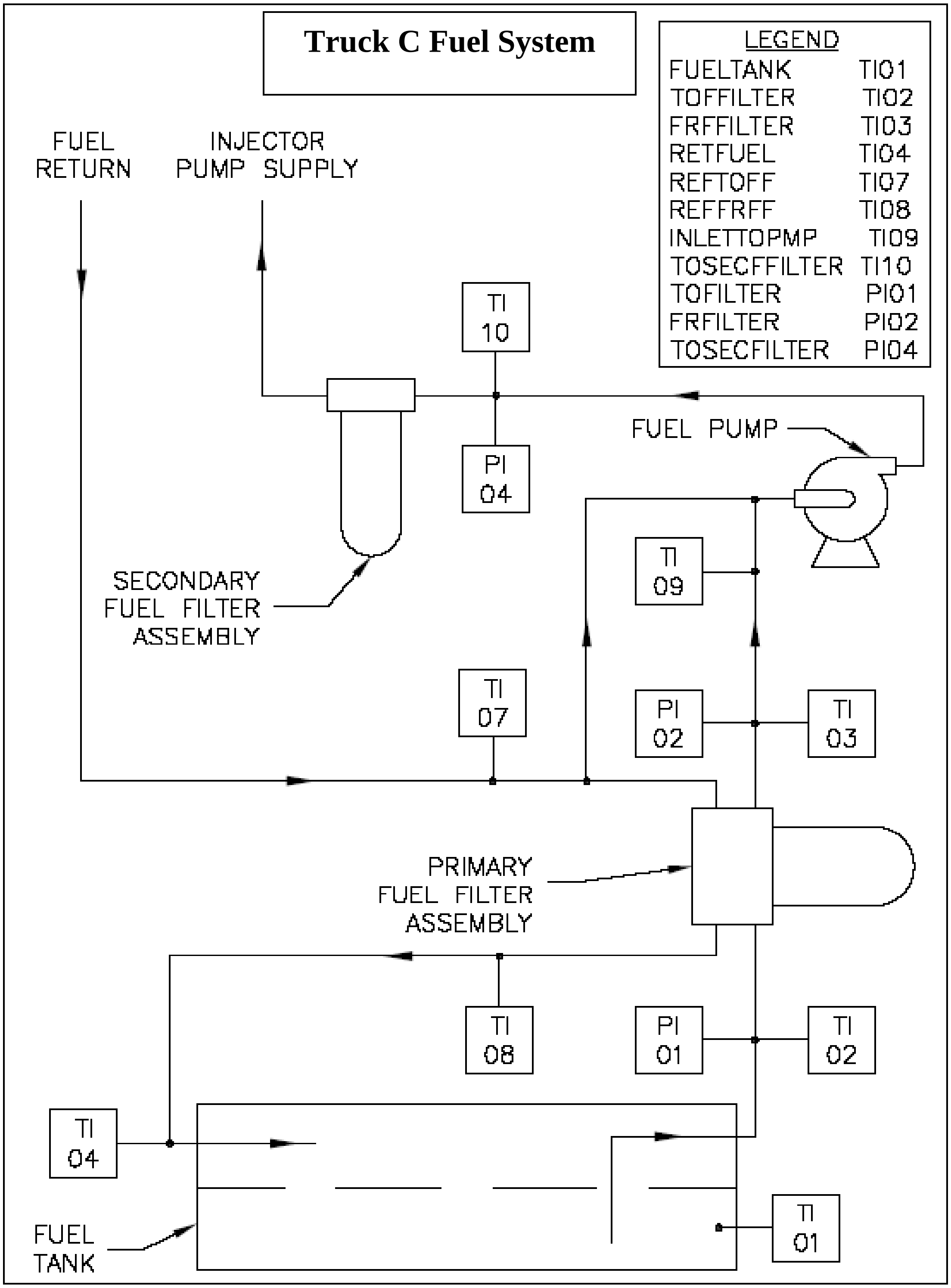


Figure 5. Vehicle fuel system diagram for Truck C.

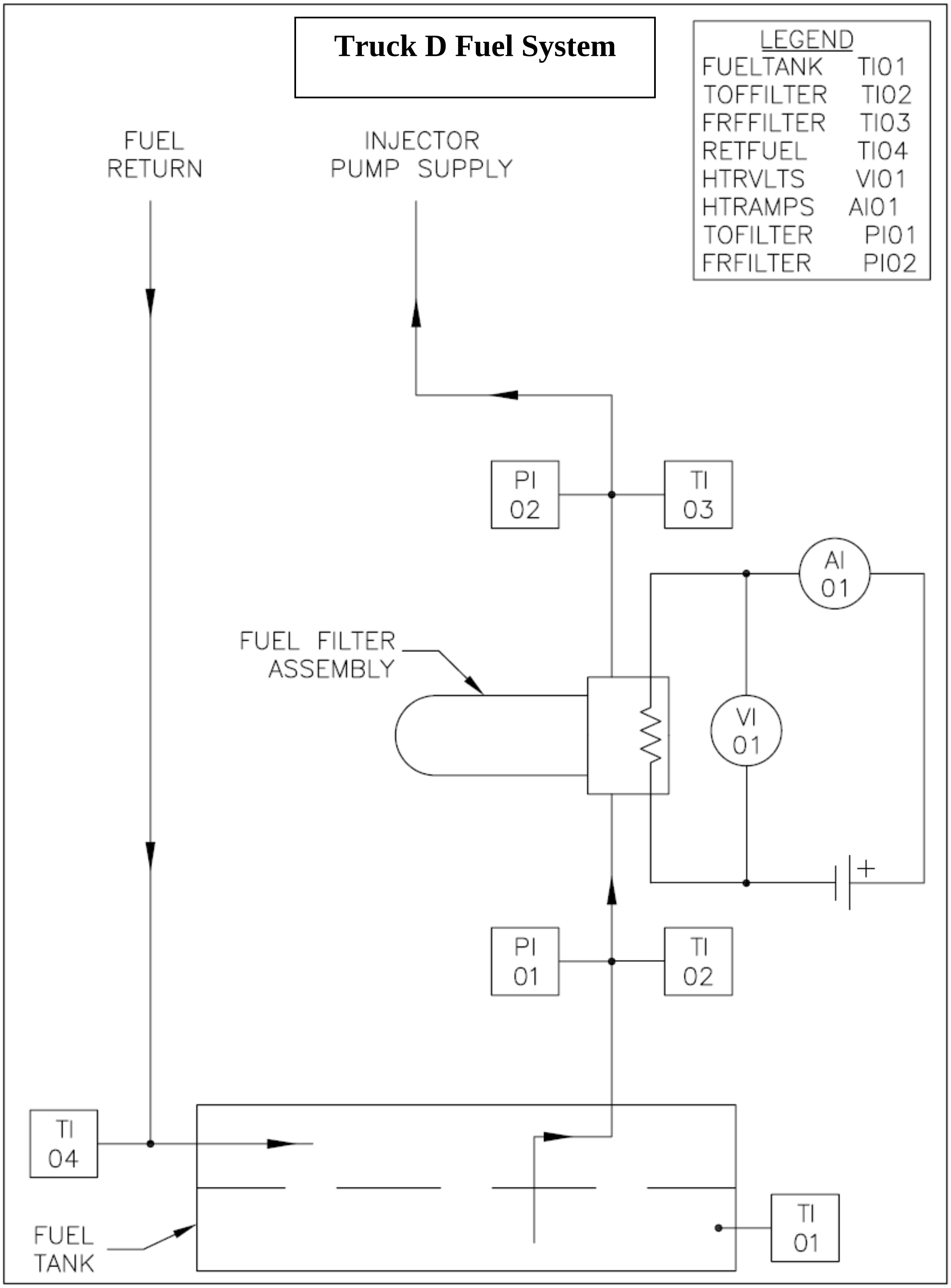


Figure 6. Vehicle fuel system diagram for Truck D.

Test Vehicle Preparation

Sensors were installed on each test vehicle to measure and record temperatures and pressures. The locations of these are noted in the fuel system diagram (Figure 4, Figure 5, Figure 6). Vehicle speed, wind speed, and tractive force were measured by facility sensors. The following are the key parameters:

- Temperatures: ambient, fuel tank, fuel before and after filter, fuel return, engine oil, and coolant.
- Pressure: before and after fuel filter, after secondary filter (if equipped).
- Speed: engine speed (rpm), vehicle speed (kph), wind speed (kph).
- Force: tractive force (N).
- Voltage and current to fuel filter heater in the case of Truck D.

The engine oil in each vehicle was replaced with SAE 0W40 multi-grade oil. This Arctic grade motor oil was selected to ensure that motor oil viscosity was not a factor in starting the engines at cold temperatures. An engine block heater and ether starting-aid system were standard equipment in all of the test vehicles.

Test Fuels

Two diesel fuels were procured representing an Arctic winter diesel (a re-branded Jet A-1) and a regional winter diesel. They are nominally called #1 diesel (#1D) and #2 diesel (#2D), respectively. Four biodiesel (B100) samples were shipped to IOC for blending. They were supplied by four different biofuel manufacturers and designated B100A, B, C, and D. The fuel blends used in this study are listed in Table 3. Detailed characterization results for these fuels and blending components are presented in the Results section.

Table 3. Test fuels for low-temperature operability limit study.

Fuel Code	% #1D	% #2D	% B100A	% B100B	% B100C	% B100D
TF #1		100				
TF #2		95	5			
TF #3		95		5		
TF #4		95			5	
TF #5	40	40	20			
TF #6	40	40		20		
TF #7	40	40			20	
TF #8		80		20		
TF #9		95				5
TF #10	40	40				20
TF #11	80			20		

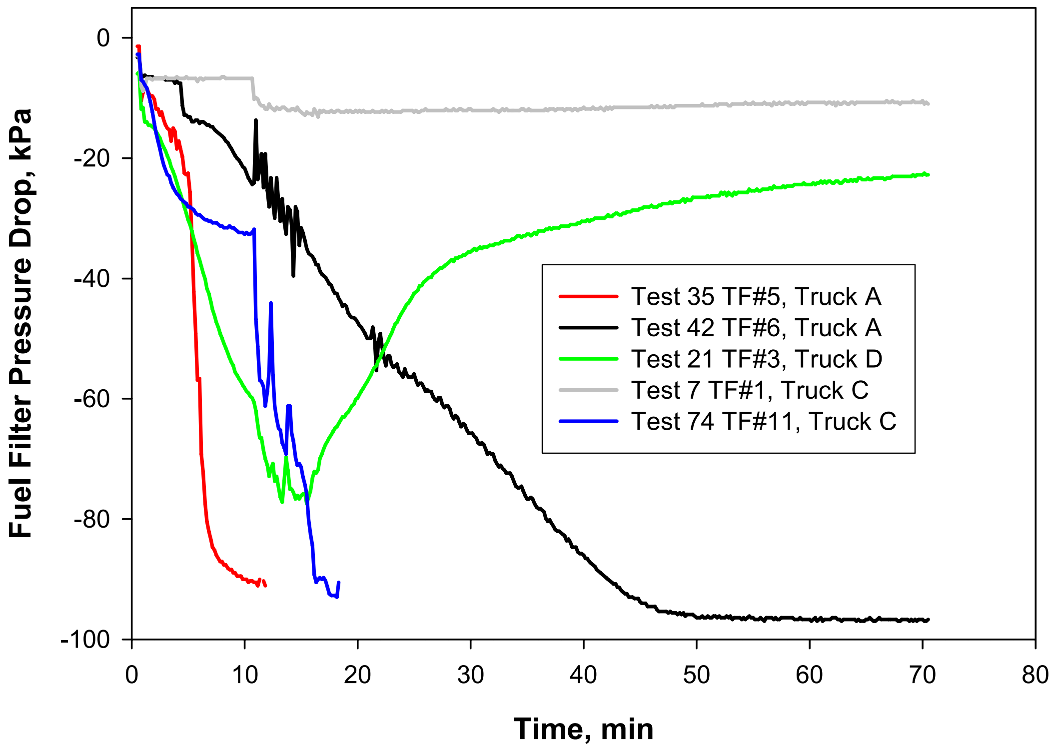
Test Procedures

To prepare the test trucks, one of the two fuel tanks was disconnected from each fuel system. At the start of each test fuel, the previous fuel was drained from the tank and the test fuel flushed into the tank using a double flush procedure. A volume of 200 L of fuel (approximately half of the tank) was used at the start of each test. The trucks were then placed inside the AWCD the night before the test to be conditioned, or "soaked", to the test temperature. A minimum of 12 hours and usually over 14 hours of soaking occurred before tests began. Normally, the air temperature during the soak period was set to about 2 °C below the target test temperature to allow the fuel tank to reach the test temperature within a reasonable time. The air temperature of the test cell was then set to the test temperature before the start of the test. During the overnight soak period, a one-liter sample of the test fuel in a glass bottle was also placed in the facility so that the appearance of the test fuel at the test temperature could be observed, and a photo taken for the record. These photos as well as temperature records for the cool down and soak are part of the data supplied on CD by IOC for this project.

The engine block heaters were plugged in during the overnight soak. Both the ether injection starting aid and warm batteries were used to maximize engine starting probability. Once the engine was started, it was left in idle for 10 minutes before accelerating at a modest rate to 80 km/hr (kph). The truck was then maintained at 80 kph for one hour to complete the drive cycle. The loading of the chassis dynamometer was set to simulate 36,400 kg (~80,000 lb) gross vehicle weight of a class-8 HD truck.

The operability of the test fuel blend can be assessed based on: (i) its ability to start the engine if it is fuel related; (ii) the ability to maintain idle condition for 10 minutes; (iii) the ability to accelerate to 80 kph; and (iv) the ability to maintain a steady speed of 80 kph for one hour. Figure 7 shows fuel filter pressure drop and vehicle speed for several example tests. In this figure, Test 35 shows a vehicle that had completed the 10 minute idle but stalled when the driver tried to accelerate, while in Test 74 the vehicle accelerated to 80 kph but then stalled because of fuel filter plugging. In the other three tests shown, the vehicle successfully completed the test. In Test 21, there was significant fuel filter pressure drop but this abated as the vehicle fuel tank was warmed by the return fuel. Test 42 showed extremely high fuel filter pressure drop that never abated; but nevertheless, the vehicle completed the test.

Figure 8 shows fuel tank temperature traces for the three trucks. These three runs were all for passing tests on the same fuel and illustrate the very different “warm up” behavior of the three vehicle fuel systems. Truck D has the highest fuel return rate, an electric heater on the fuel filter, and the fuel filter located in the engine compartment. For this vehicle, the fuel begins to warm immediately at the start of the test. For Trucks A and C the fuel filter is located outside the vehicle and fuel return rates are much lower; so the fuel tank does not warm during the idle phase and the warming profile is different for the two trucks.



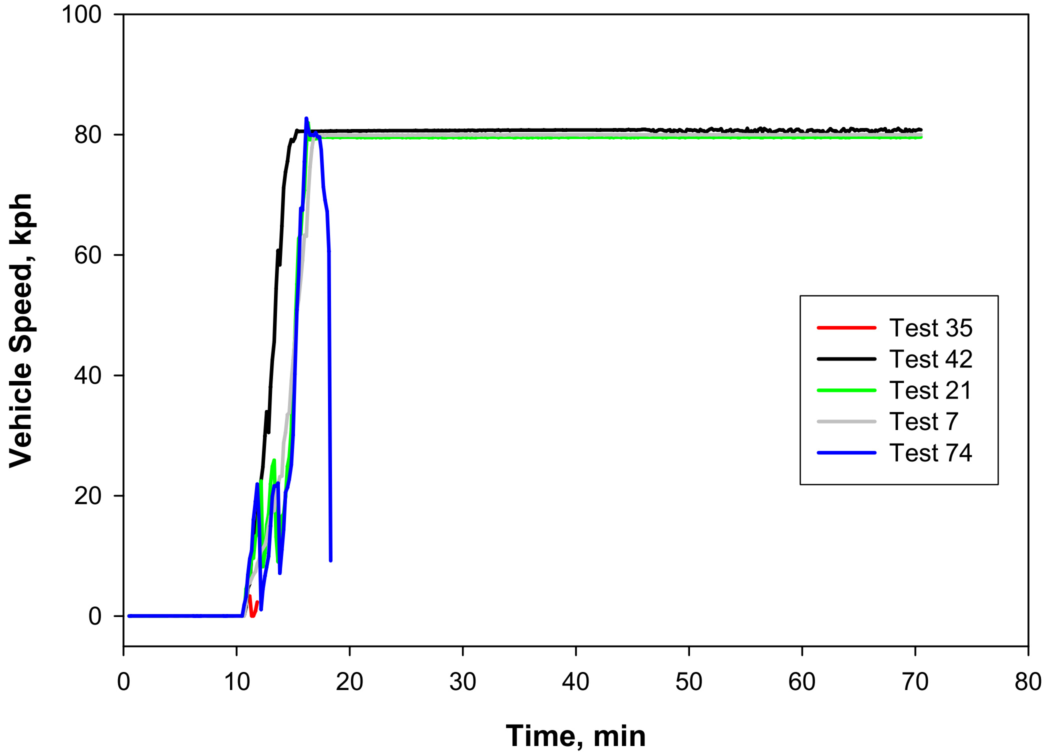


Figure 7. Fuel filter pressure drop traces (top) and vehicle speed (bottom) for example tests.

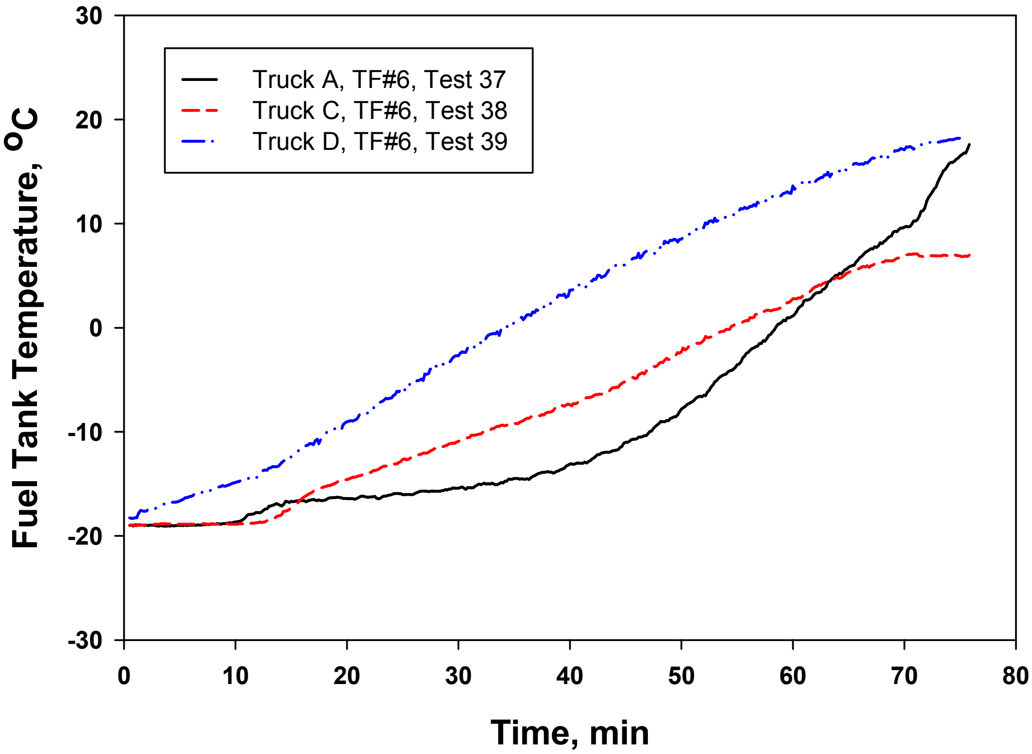


Figure 8. Fuel tank temperature traces for the three trucks on passing tests with the same fuel.

4. Fuel and Blend Component Characterization Results

Base Diesel Characterization

Bench low-temperature operability test results for the No. 1 diesel, No. 2 diesel and their 50/50 blend are shown in Table 4. The 95% confidence interval is reported for the No. 2 diesel CP because this fuel was actually tested in the vehicles. Precision is in line with expectations for this measurement. Neither the No. 1 diesel, nor the 50/50 blend of the two, were tested in the vehicles. The 50/50 blend was used as the petroleum diesel fraction of the B20 blends. Complete D975 characterization and other properties for the No. 1 and No. 2 fuels are shown in Appendix I.

B100 Characterization

The measured properties of B100A, B, C, and D are shown in Table 5 – 8, respectively. All four B100 met D6751 requirements for those properties tested. One lab returned high, out of specification values for free glycerin for B100A, C, and D; however, four other labs showed much lower values. A number of labs reported results as less than a detection limit, and in these cases the average is reported as less than the average of all values, including detection limits, reported.

The cold soak filtration procedure used in this study is listed in Appendix II. B100A exhibits a high CSFT, above 720 seconds. The producer of this material had targeted a CSFT of 400 seconds to 500 seconds to be just above the proposed 360 second limit. Their approach was to blend a high CSFT material with a low (<200 sec) CSFT material. The target value was not attained, and the producer believes this was the result of their lab testing a sample that was not representative of what was sent to IOC. Nevertheless, this material is a blend of low CSFT and high CSFT material, and thus may not be fully representative of a B100 with a naturally high CSFT.

Both B100B and C show CSFT below 200 seconds at all test labs. Three labs found B100D to have a CSFT of less than 200 seconds, while the fourth reported a value of 296 seconds. The possibility that this fourth result was an outlier was investigated. However, the lab returning this result is one of the more experienced labs with this test, and the value could not be rejected using standard statistical-tests.

FAME speciation and other chemical analysis results are reported in Appendix III.

Table 4. Bench low temperature operability test results (ºC) for No. 1 and No. 2 diesel fuels and the 50/50 (volume) blend.

Lab	**CP**				**CFPP**		**LTFT**		
	Method	No. 2	No. 1	50/50 No.2/No.1	Method	No. 2	Method	No. 2	50/50 No.2/No.1
A	D2500	-30	-50						
B	D2500	-29	-50	-36	D6371	-32			
C	D5773	-29.4	-48.1						
D	D5773	-27.4	-47.9				D4539	-27	
G	D5772	-26.5	-41.7		D6371	-25			
I	D5773	-29	-52	-39			D4539	-27	-35
	Average	**-28.6**	**-48.3**	**-37.5**		**-28.5**		**-27.0**	**-35**
	St. Dev.	1.32							
	95% conf	1.06							

Table 5. Characterization and properties of B100A.*

Lab:	Method	A	B	E	F	G	K	L	N	**Average**
Cloud Point, ºC	various	6	2	3.5		4.2	4.9	7		4.6
Flash Point, ºC	D93	174.4		175			>150	178		>150
Acid Value, mg KOH/g	D664	0.06		0.1			0.098	0.07		0.082
Water and Sediment , vol%	D2709	0.01		<0.025			0.02	<0.01		<0.02
Dissolved Water, ppm	various	226	119	184			195.3	170		179
Kinematic viscosity at 40°C, mm^2/s	D445	4.167					4.192			4.180
Copper strip corrosion	D130	1a					1a			1a
Distillation (AET T90), °C	D1160	351								351
Carbon residue, wt%	D4530	0.0067								0.0067
Oxidation Stability, hr	EN14112			5.3			7.55	6.53		6.46
Sulfur, ppm	various	2.2	1.5	1.2			1.8			1.7
Sulfated Ash, wt%	D874	<0.001					0			<0.001
Phosphorus, ppm	D4951	<5					<5			<5
Na+K, ppm	EN14538	<2					0.4			<2
Ca+Mg, ppm	EN14538	<0.2					0.05			<0.2
Soap, ppm	CC1795						8			8
Free glycerin, wt%	D6584	<0.005		<0.005			0.003	0.001	*0.021*	<0.007
Total glycerin, wt%	D6584	0.178		0.190			0.142	0.163	0.148	0.164
Monoglyceride, wt%	D6584	0.604		0.586			0.472	0.532	0.328	0.504
Saturated monoglyceride, wt%	D6584			0.144					0.096	0.120
Cold Soak Filtration Time, sec[a]	[a]				>720	>720	>720			>720

[*]Out of specification or suspect results in italic. [a]Method being balloted into D6751 was requested.

Table 6. Characterization and properties of B100B.[*]

Lab:	Method	A	B	E	F	G	K	L	N	**Average**
Cloud Point, ºC	various	0	-0.5	0.5		1.3	1.2	2		0.8
Flash Point, ºC	D93	130		145			>150	138		>150
Acid Value, mg KOH/g	D664	0.26		0.29			0.296	0.28		0.28
Water and Sediment , vol%	D2709	0.01		<0.025			tr	0.01		<0.025
Dissolved Water, ppm	various	180	119	244			278.2	300		224
Kinematic viscosity at 40°C, mm^2/s	D445	4.105					4.109			4.107
Copper strip corrosion	D130	1a					1a			1a
Distillation (AET T90), °C	D1160	352								352
Carbon residue, wt%	D4530	0.0067								0.0067
Oxidation Stability, hr	EN14112			5.5			6.5	6.33		6.11
Sulfur, ppm	various	1.1	0.5	0.8			0.5			0.7
Sulfated Ash, wt%	D874	<0.001					0			<0.001
Phosphorus, ppm	D4951	<5					<5			<5
Na+K, ppm	EN14538	<2					0.4			<2
Ca+Mg, ppm	EN14538	<0.2					0.05			<0.2
Soap, ppm	CC1795						0			0
Free glycerin, wt%	D6584	<0.005		<0.005			0.002	0.002	0.001	<0.003
Total glycerin, wt%	D6584	0.158		0.151			0.127	0.141	0.140	0.143
Monoglycerides, wt%	D6584	0.550		0.484			0.432	0.472	0.465	0.481
Saturated monoglycerides, wt%	D6584			0.072					0.072	0.072
Cold Soak Filtration Time, sec[a]	[a]				95	92	126	93		102

[*]Out of specification or suspect results in italic. [a]Method being balloted into D6751 was requested.

Table 7. Characterization and properties of B100C.[*]

Lab:	Method	A	B	E	F	G	K	L	N	Average
Cloud Point, ºC	various	8	8.5	8		9.5	9.3	9		8.7
Flash Point, ºC	D93	166.7		167			>150	>150		>150
Acid Value, mg KOH/g	D664	0.4		0.39			0.418	0.4		0.40
Water and Sediment , vol%	D2709	0.01		<0.025			tr	<0.01		<0.025
Dissolved Water, ppm	various	208	178	209			243.9	220		212
Kinematic viscosity at 40°C, mm^2/s	D445	4.442					4.446			4.451
Copper strip corrosion	D130	1a					1a			1a
Distillation (AET T90), °C	D1160	350								
Carbon residue, wt%	D4530	0.0066								
Oxidation Stability, hr	EN14112			>15			27	38		>15
Sulfur, ppm	various	10.8	9.5	8.4			10.4			9.8
Sulfated Ash, wt%	D874	<0.001					0.004			<0.004
Phosphorus, ppm	D4951	<5					<5			<5
Na+K, ppm	EN14538	<3					1.5			<3
Ca+Mg, ppm	EN14538	<0.2					0.03			<0.2
Soap, ppm	CC1795						0			0
Free glycerin, wt%	D6584	<0.005		<0.005			0.001	0.001	*0.029*	<0.008
Total glycerin, wt%	D6584	0.040		0.040			0.038	0.036	0.068	0.044
Monoglycerides, wt%	D6584	0.123		0.132			0.123	0.106	0.114	0.120
Saturated monoglycerides, wt%	D6584			0.045					0.044	0.045
Cold Soak Filtration Time, sec[a]	[a]				179	118	170	105		143

[*]Out of specification or suspect results in italic. [a]Method being balloted into D6751 was requested.

Table 8. Characterization and properties of B100D.*

Lab:	Method	A	B	E	F	G	K	L	N	**Average**
Cloud Point, ºC	various	10	11.5	11.6		12.9	12.3	12		11.7
Flash Point, ºC	D93	156.1		162			>150	164		>150
Acid Value, mg KOH/g	D664	0.16		0.12			0.190	0.14		0.15
Water and Sediment , vol%	D2709	0.01		<0.025			tr	0.01		<0.025
Dissolved Water, ppm	various	105	25	94			107.6	120		90
Kinematic viscosity at 40°C, mm^2/s	D445	4.534					4.449			4.92
Copper strip corrosion	D130	1a					1a			1a
Distillation (AET T90), °C	D1160	352								
Carbon residue, wt%	D4530	0.00674								
Oxidation Stability, hr	EN14112			4.8			8.2	*2.51*		5.17
Sulfur, ppm	various	5.3	2.7	4.5			4.4			4.2
Sulfated Ash, wt%	D874	<0.001					0			<0.001
Phosphorus, ppm	D4951	<5					<5			<5
Na+K, ppm	EN14538	<4					1.8			<4
Ca+Mg, ppm	EN14538	<0.2					0.02			<0.2
Soap, ppm	CC1795						244			244
Free glycerin, wt%	D6584	<0.005		<0.005			0.012	0.006	*0.023*	<0.010
Total glycerin, wt%	D6584	0.026		0.031			0.026	0.028	0.052	0.033
Monoglycerides, wt%	D6584	0.038		0.047			0.023	0.057	0.032	0.039
Saturated monoglycerides, wt%	D6584			0.0186					0.015	0.017
Cold Soak Filtration Time, sec[a]	[a]				185	167	296	112		190

*Out of specification or suspect results in italic. [a]Method being balloted into D6751 was requested.

Preliminary Blend Cloud Point Study

A range of blends of the No. 1 and No. 2 diesel fuels with 20% biodiesel were prepared to determine CP impact. In Figure 9, it can be seen that blending of increasing amounts of No. 1 diesel into the No. 2 fuel caused CP to decrease approximately in proportion to the No. 1 diesel content. However, for the B20 blends, the No. 1 diesel content had little or no impact on CP in this very low temperature operating range. More detailed results presented below and based on measurements from multiple labs indicate that CP is roughly 5°C lower for B20 produced with the No. 1 diesel versus the No. 2 diesel. Because of the lack of a strong effect of No. 1 addition on B20 CP for these fuels, a 50/50 blend of No. 1 and No. 2 diesel was selected for the petroleum fraction of the B20 blends. The B5 blends were prepared using No. 2 diesel.

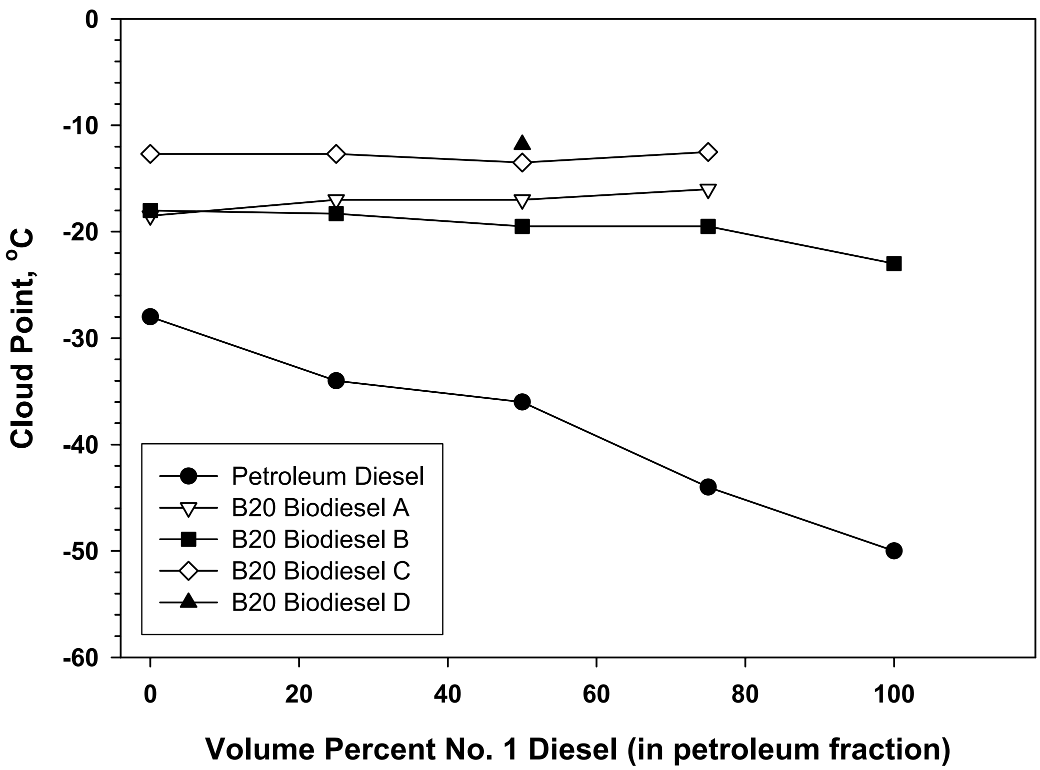


Figure 9. Results of preliminary blend cloud point study.

B5 Blends

B5 blends were prepared from each B100 by blending with the No. 2 diesel fuel. CP and other low temperature operability test results are shown in Table 9. CP is compared with CFPP and LTFT in Figure 10. The B5 results are those clustered between CP of -23°C and -25.3°C; thus, addition of 5% biodiesel caused a 3°C to 5°C increase in CP. CFPP is equal to or slightly below CP, while LTFT is equal to or slightly above CP. This is in line with expectations because the LTFT test was developed to predict performance with the most challenging vehicle fuel systems. Additional characterization results for the B5 blends are shown in Appendix IV.

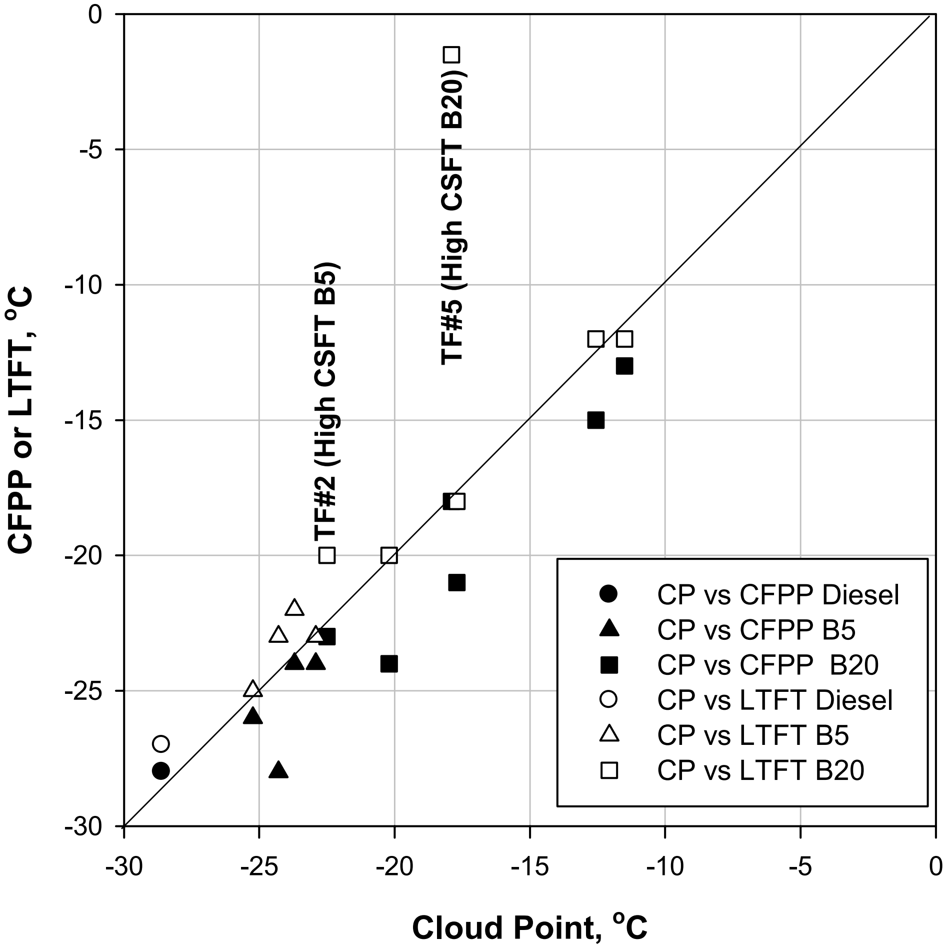


Figure 10. Comparison of CP with CPFF and LTFT for B5 and B20 blends.

B20 Blends

B20 blends were prepared from each B100 by blending with a 50/50 (vol) blend of the No. 1 and No. 2 diesel fuel. Additionally, B100B was blended with both the No. 1 and No. 2 diesel fuels to make two additional B20 blends. Cloud point results for these fuels are shown in Table 10, while CFPP and LTFT are shown in Table 11 and Table 12, respectively. Recall that the CP of the 50/50 No. 1/No. 2 diesel blend was -37.5°C. Blending of 20% biodiesel causes an increase of CP by 12 to 15°C. Figure 10 also shows a comparison of CP with CFPP and LTFT results for the B20 blends. Trends are as expected with the exception of the TF#5, which was prepared from B100A having a high CSFT. For this blend LTFT averages -1.5°C while CP is -17.9°C. Additional characterization results for B20 blends are reported in Appendix IV.

Table 9. Cloud point, CFPP, and LTFT results for B5 blends (°C).

Lab	**Cloud point**	**TF#2**	**TF#3**	**TF#4**	**TF#9**	**CFPP**					**LTFT (Lowest Pass Temp)**				
	Method	**B5A**	**B5B**	**B5C**	**B5D**	Method	**B5A**	**B5B**	**B5C**	**B5D**	Method	**B5A**	**B5B**	**B5C**	**B5D**
A	D2500	-20	-25	-25	-25										
B	D2500	-26.5	-27	-25	-25	D6371	-29	-28	-28	-23					
C	D5773	-24.4	-24.6	-22.4	-21.5										
D	D5773	-24.7	-24.7	-23	-24.5						D4539	-22	-25	-22	-21
G	D5772	-22.1	-22.7	-20	--	D6371	-26	-23	-22	--	D4539	-20	-24	-20	--
H	D5771	-25.6	-26.5	--	-25	D6371	-29	-26	--	-25					
I	D5773	-25	-26	-23	-23						D4539	-24	-25	-24	-22
J	D5773	-26	-26	-23	-22	D6371	-29	-25	-23	-23	D4539	-25	-25	-24	-24
Average		**-24.3**	**-25.3**	**-23.0**	**-23.7**		**-28**	**-26**	**-24**	**-24**		**-23**	**-25**	**-23**	**-22**
Standard Dev		2.18	1.36	1.61	1.53										
95% conf.		1.51	0.94	1.20	1.13										

Table 10. Cloud point results for B20 blends (°C).

Lab	Cloud point	TF#5	TF#6	TF#7	TF#10	TF#8	TF#11
	Method	**B20A**	**B20B**	**B20C**	**B20D**	**B/No. 2**	**B/No. 1**
A	D2500	-14.5	-17.0	-13.0	-11.2	-16.0	-21.0
B	D2500	-19.0	-21.0	-13.0	-12.5	-20.0	--
C	D5773	-19.1	-21.4	-12.4	-11.2	-18.0	-22.4
D	D5773	--	-20.4	-12.1	-11.3	-18.1	-22.8
G	D5772	-18.3	-20.8	-11.0	-10.0	-16.0	-23.6
H	D5771	-17.6	--	-13.5	--	--	--
I	D5773	-18	-20	-13	-12	-18	-22
J	D5773	-19	-21	-12	-12	-18	-23
Average		**-17.9**	**-20.2**	**-12.5**	**-11.5**	**-17.7**	**-22.5**
Standard Dev.		1.62	1.49	0.79	0.81	1.38	0.90
95%conf		1.20	1.11	0.55	0.60	1.03	0.72

Table 11. CFPP results for B20 blends (°C).

Lab	CFPP	TF#5	TF#6	TF#7	TF#10	TF#8	TF#11
	Method	**B20A**	**B20B**	**B20C**	**B20D**	**B/No. 2**	**B/No. 1**
B	D6371	-20	-26	-18	-14	-23	--
G	D6371	-15	-23	-12	-11	-19	-22
H	D6371	-20		-15	--	--	--
J	D6371	-18	-24	-14	-13	-22	-24
Average		**-18**	**-24**	**-15**	**-13**	**-21**	**-23**

Table 12. LTFT results for B20 blends (°C).

Lab	LTFT	TF#5	TF#6	TF#7	TF#10	TF#8	TF#11
	Method	**B20A**	**B20B**	**B20C**	**B20D**	**B/No. 2**	**B/No. 1**
D	D4539	0	-20	-12	-11	-19	-22
G	D4539	-8	-20	-10	-12	-16	-16
I	D4539	1	-19	-12	-11		-19
J	D4539	1	-21	-13	-12	-19	-23
Average		**-1.5**	**-20.0**	**-11.8**	**-11.5**	**-18.0**	**-20.0**

5. Vehicle Low-Temperature Operability Results

A listing of all vehicle tests performed along with the basic results (test temperature, maximum fuel filter pressure drop, and pass/fail rating) is shown in Table 13. For a test to be rated as failing, at some point in the test the vehicle failed to operate by:

- Not starting
- Not completing the 10minute idle
- Not accelerating to 80 kph
- Not being able to maintain 80 kph for the required time.

Table 13. Basic vehicle testing results.

Test Date	Test No.	Vehicle	Fuel	Test Temperature, °C	Maximum Primary Filter ΔP, kPa	Rating
13/12/2007	1	Truck D	TF #1	-33.6	-77.7	F
	2	Truck A	TF #1	-34.5	-99.0	F
	3	Truck C	TF #1	-33.8	-88.1	F
14/12/2007	4	Truck C	TF #1	-30.6	-90.3	F
	5	Truck D	TF #1	-30.2	-96.1	F
	6	Truck A	TF #1	-30.2	-77.2	F
17/12/2007	7	Truck C	TF #1	-26.4	-10.5	P
	8	Truck A	TF #1	-26.0	-8.1	P
	9	Truck D	TF #1	-25.7	-22.0	P
18/12/2007	10	Truck C	TF #2	-24.0	-32.6	P
	11	Truck A	TF #2	-24.0	-37.4	P
	12	Truck D	TF #2	-23.6	-30.2	P
19/12/2007	13	Truck D	TF #2	-25.9	-67.3	P
	14	Truck C	TF #2	-25.8	-74.3	P
	15	Truck A	TF #2	-26.1	-82.8	F
20/12/2007	16	Truck D	TF #3	-24.3	-35.3	P
	17	Truck C	TF #3	-24.5	-19.3	P
	18	Truck A	TF #3	-24.6	-27.3	P
21/12/2007	19	Truck A	TF #3	-26.5	-93.5	F
	20	Truck C	TF #3	-25.9	-94.3	F
	21	Truck D	TF #3	-26.0	-77.2	P
07/01/2008	22	Truck A	TF #4	-24.7	-86.3	P
	23	Truck C	TF #4	-23.9	-71.1	P
	24	Truck D	TF #4	-23.7	-41.7	P
08/01/2008	25	Truck D	TF #4	-26.1	-71.5	F
	26	Truck A	TF #4	-25.5	-86.5	F
	27	Truck C	TF #4	-26.2	-90.5	F
09/01/2008	28	Truck A	TF #5	-18.0	-78.8	F
	29	Truck C	TF #5	-19.6	-90.8	F
	30	Truck D	TF #5	-18.4	-96.1	F
10/01/2008	31	Truck C	TF #5	-15.5	-68.1	P
	32	Truck A	TF #5	-15.4	-100.2	F
	33	Truck D	TF #5	-14.9	-63.3	P
11/01/2008	34	Truck D	TF #5	-16.9	-60.6	P
	35	Truck A	TF #5	-17.0	-91.1	F
	36	Truck C	TF #5	-17.2	-62.5	P

Table 13. Continued.

Test Date	Test No.	Vehicle	Fuel	Test Temperature, °C	Maximum Primary Filter ΔP, kPa	Rating
14/01/2008	37	Truck A	TF #6	-19.0	-76.4	P
	38	Truck C	TF #6	-19.0	-43.0	P
	39	Truck D	TF #6	-18.3	-17.2	P
15/01/2008	40	Truck D	TF #6	-21.4	-53.2	P
	41	Truck C	TF #6	-21.3	-67.3	P
	42	Truck A	TF #6	-21.4	-96.4	P
16/01/2008	43	Truck A	TF #6	-24.3	-88.9	F
	44	Truck C	TF #6	-24.3	-95.1	F
	45	Truck D	TF #6	-24.1	-96.1	F
17/01/2008	46	Truck D	TF #7	-13.3	-11.8	P
	47	Truck C	TF #7	-13.4	-60.4	P
	48	Truck A	TF#7	-12.1	-91.9	F
18/01/2008	49	Truck A	TF #7	-13.4	-13.1	P
	50	Truck C	TF #7	-14.9	-92.7	F
	51	Truck D	TF #7	-15.0	-88.1	F
21/01/2008	52	Truck D	TF #8	-20.7	-96.4	P
	53	Truck C	TF #8	-21.3	-96.4	F
	54	Truck A	TF #8	-20.7	-84.4	F
22/01/2008	55	Truck D	TF #8	-20.6	-96.1	P
	56	Truck C	TF #8	-19.6	-68.7	P
	57	Truck A	TF #8	-19.8	-95.6	F
23/01/2008	58	Truck A	TF #9	-22.1	-15.4	P
	59	Truck C	TF #9	-22.1	-13.7	P
	60	Truck D	TF #9	-22.3	-40.4	P
24/01/2008	61	Truck D	TF #9	-24.2	-76.9	P
	62	Truck C	TF #9	-24.3	-94.3	F
	63	Truck A	TF #9	-24.1	-90.8	F
25/01/2008	64	Truck A	TF #10	-11.8	-20.8	P
	65	Truck C	TF #10	-11.7	-93.2	F
	66	Truck D	TF #10	-11.7	-46.4	P
28/01/2008	67	Truck C	TF #10	-12.1	-50.2	P
	68	Truck A	TF #10	-13.0	-84.7	F
	69	Truck D	TF #10	-13.7	-93.4	F
29/01/2008	70	Truck D	TF #11	-23.3	-40.6	P
	71	Truck C	TF #11	-22.9	-44.4	P
	72	Truck A	TF #11	-23.1	-77.7	P
30/01/2008	73	Truck A	TF #11	-24.8	-94.0	F
	74	Truck C	TF #11	-24.7	-93.0	F
	75	Truck D	TF #11	-24.8	-85.2	P

Operability Results for Each Truck

Results for Truck A are shown in Figure 11. There were 15 failing tests, 10 passing tests, and the operability limit was bracketed for 9 of the 11 fuels in this vehicle. Notably, test fuel #5 did not produce a passing test even at temperatures several degrees above CP. Test fuel #5 is the B20 blend from B100A, the high CSFT B100; this result is also discussed in detail below. For test fuel #7 in Truck A, a failing test was actually observed at a higher temperature than a passing test. For the failing test (test number 48) the fuel in the vehicle fuel tank was stirred prior to the test. The purpose of doing this was to determine if stirring caused by movement of the vehicle on to the chassis dynamometer might cause an otherwise passing condition to produce a fail by suspending material that had precipitated to the bottom of the tank. Apparently, this can occur since a subsequent test of this fuel/vehicle combination at a lower temperature (below CP) produced passing results. The effects found for stirring of the fuel tank may indicate that vehicle dynamometer tests do not capture all failure modes that can be experienced in actual vehicle operation.

Results for Truck C are shown in Figure 12. There were 11 failing tests, 15 passing tests, and operability limit was bracketed for 10 of the 11 fuels. For the single fuel where operability was not bracketed (TF#2), a passing result was obtained below CP. For test fuel #10, passing and failing tests were obtained at nearly the same temperature, and right at the CP. For the passing test, the vehicle was actually cooled overnight on the dynamometer; while for the failing test, the vehicle was moved from the cold soak room onto the dyno. This test may be a case where precipitated materials at the bottom of the fuel tank were suspended by moving the vehicle.

Results for Truck D are shown in Figure 13. There were 7 failing tests, 18 passing tests, and operability limit was bracketed for 7 of the 11 fuels. However, for the remaining 4 fuels, operability below CP was demonstrated. The operability limit was demonstrated to be below at or CP for 9 of the 11 fuels. Because of the electrical heating of the primary fuel filter and a higher fuel return rate, this truck was the least challenging from a low-temperature operability standpoint.

Primary fuel filter pressure traces for each truck operating on B5 blends are shown in Figure 14, Figure 15, and Figure 16; truck speed traces are found in Appendix V. In all three figures, failing tests were those where the pressure trace does not extend to 70 minutes. For TF#2 (high CSFT B100) in Truck A, the passing test exhibited continuously increasing pressure drop, even though test temperature is slightly above CP. For all other passing fuel/truck combinations, the pressure trace had stabilized or begun to increase by the end of the test.

Primary fuel filter pressure traces for each truck operating on the B20 blends are shown in Figure 17, Figure 18, and Figure 19; truck speed traces are found in Appendix V. As before, failing tests were those where the pressure trace does not extend to 70 minutes. In addition to these, in test number 32 (Truck A) the vehicle completed 70 minutes of operation but was unable to maintain 80 kph for the final 20 minutes. Notably the fuel used in this test (TF#5) was blended from B100A, the sample with high CSFT. There are several passing tests in Truck A, where fuel filter pressure drop increased continuously throughout the test. For test numbers 42 and 72 the test temperature was below CP. However, for test 37 the test temperature was roughly 1°C above CP. In Trucks C and D, all passing tests show stable or increasing fuel filter pressure drop by the end of the test.

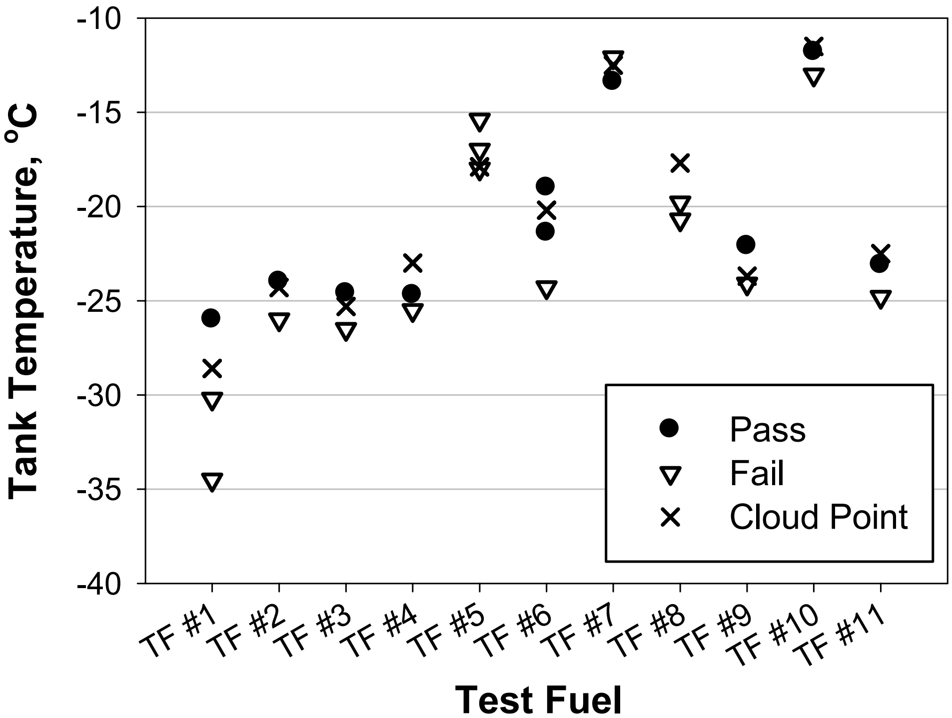


Figure 11. Operability test results for Truck A.

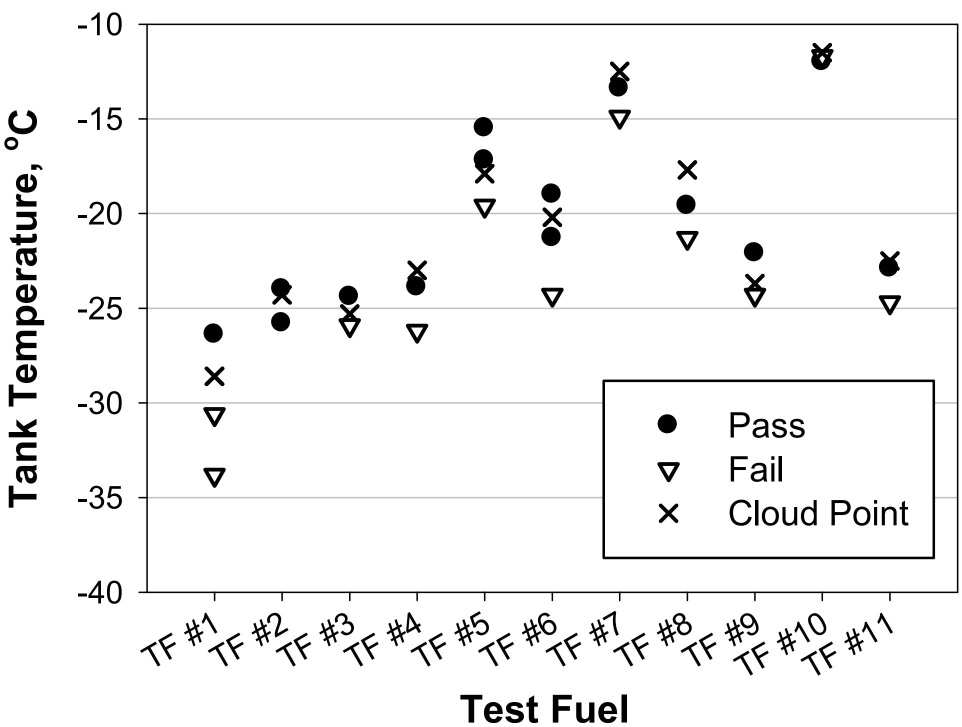


Figure 12. Operability test results for Truck C.

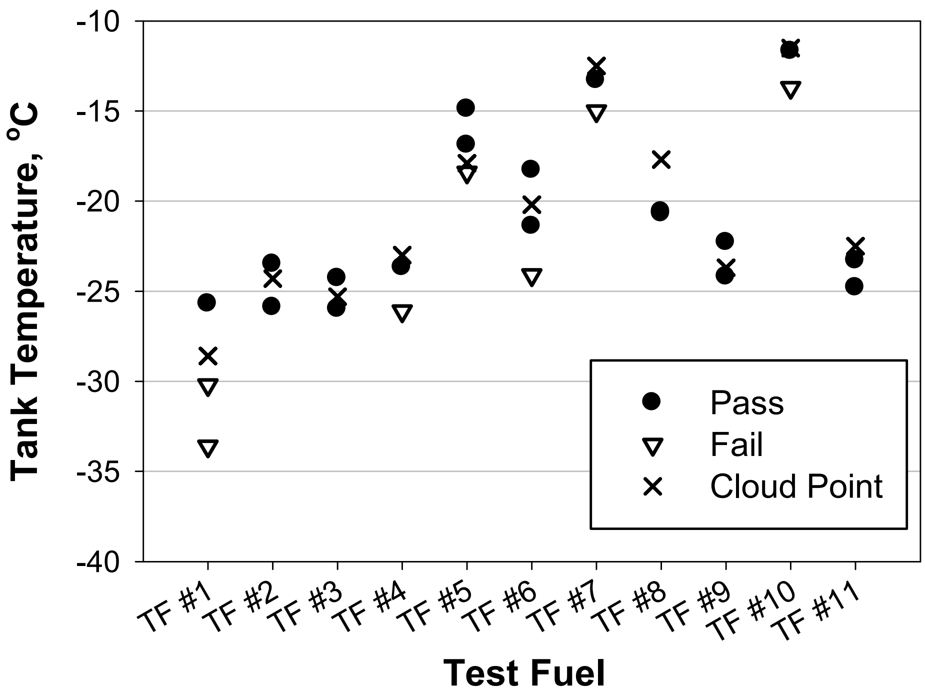


Figure 13. Operability test results for Truck D.

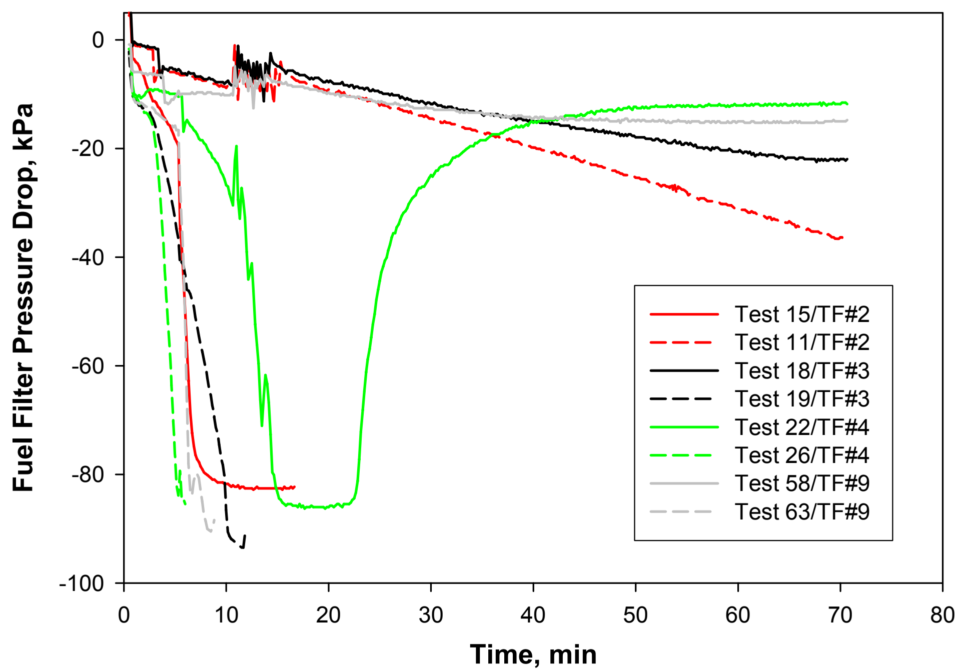


Figure 14. Primary fuel filter pressure drop for Truck A operating on B5 blends.

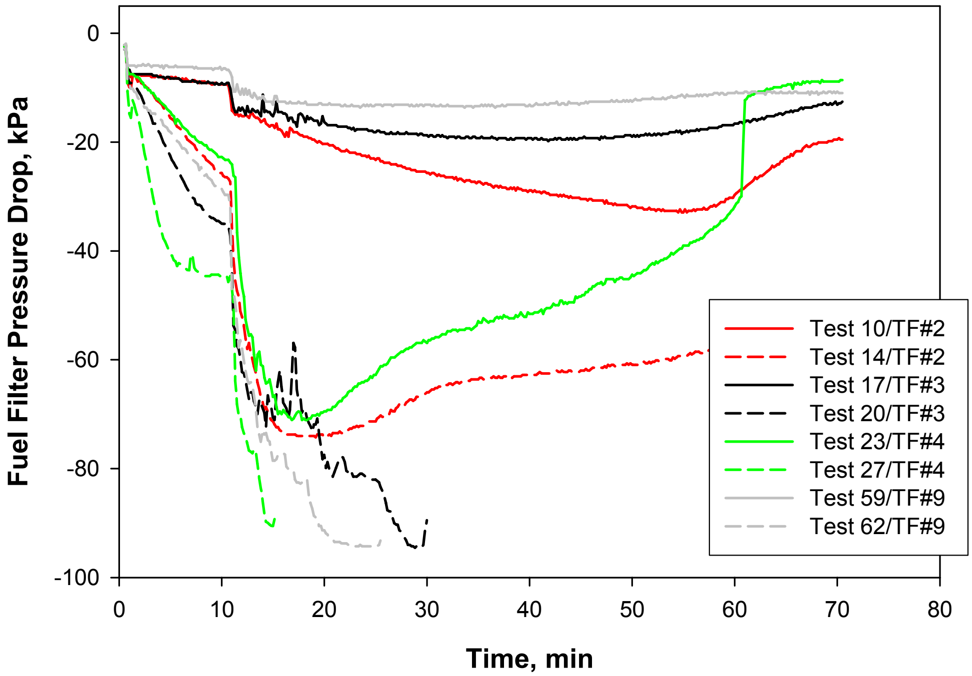


Figure 15. Primary fuel filter pressure drop for Truck C operating on B5 blends.

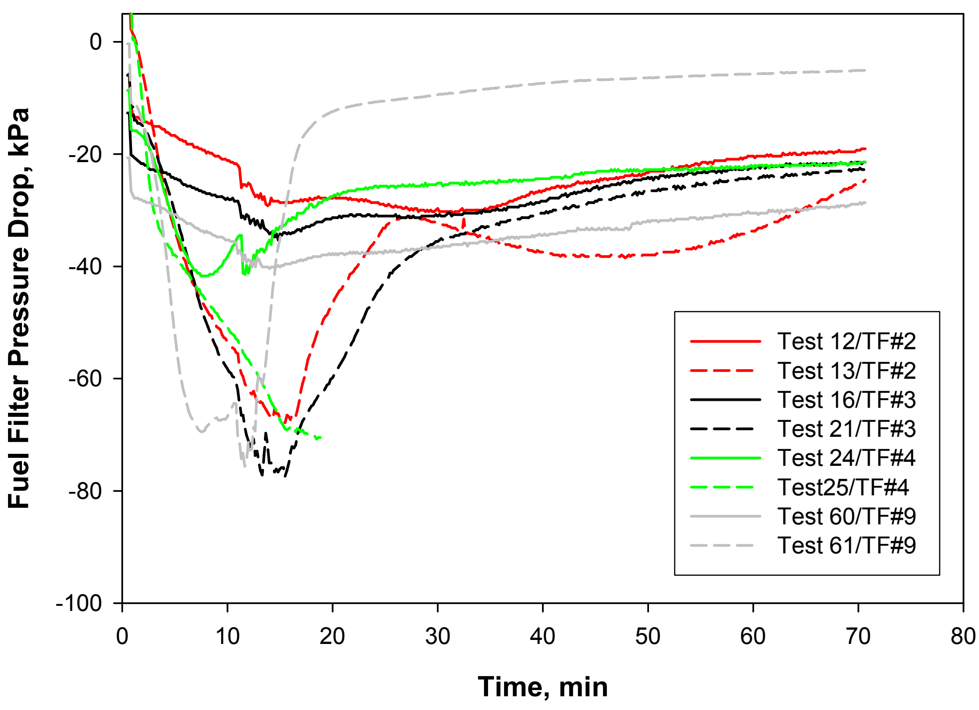


Figure 16. Primary fuel filter pressure drop for Truck D operating on B5 blends.

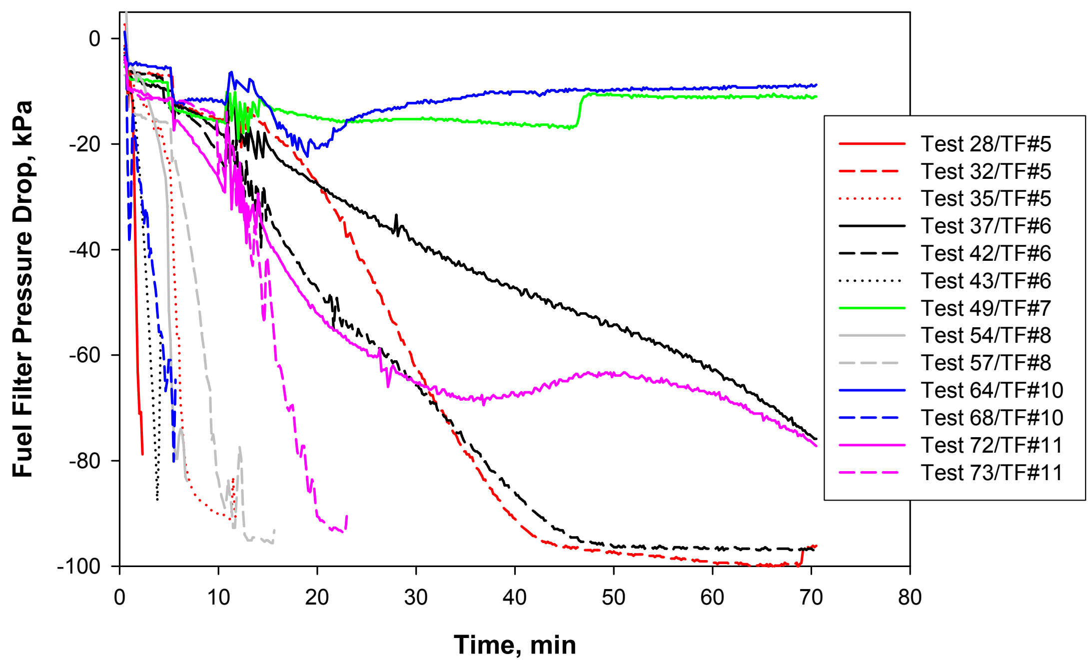


Figure 17. Primary fuel filter pressure drop for Truck A operating on B20 blends.

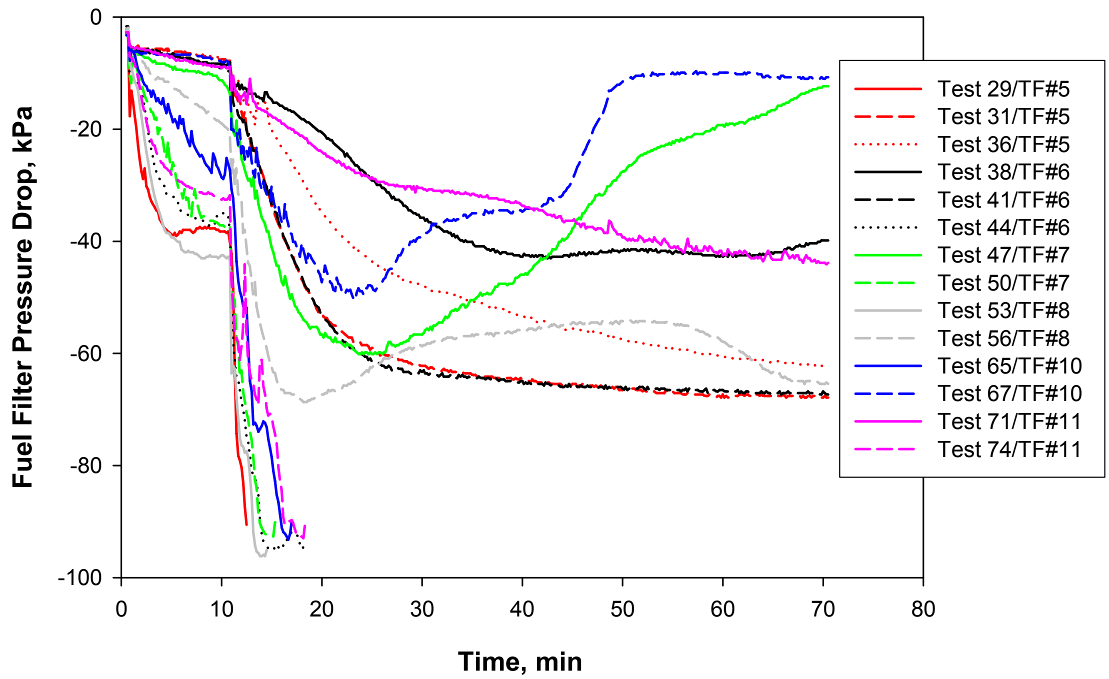


Figure 18. Primary fuel filter pressure drop for Truck C operating on B20 blends.

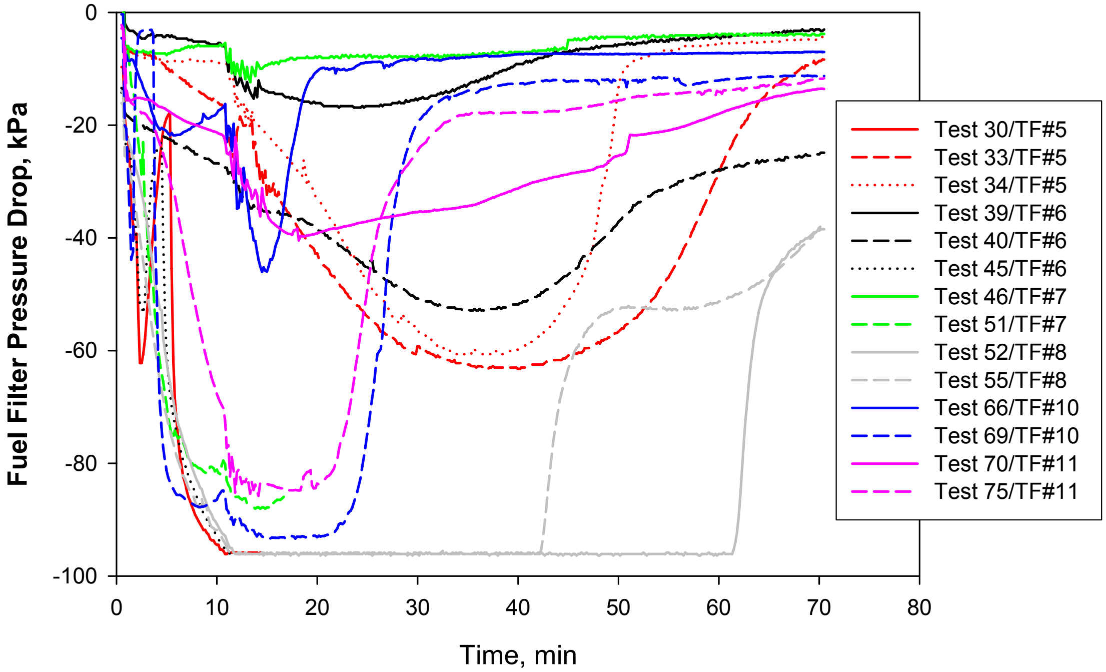


Figure 19. Primary fuel filter pressure drop for Truck D operating on B20 blends.

Operability Results for Each Fuel

No. 2 Diesel

Low-temperature operability results for the No. 2 diesel fuel (TF #1) in all three trucks are shown in Figure 20. Photographs of the fuel at the three test temperatures are also shown in the figure. At -26°C where passing tests were obtained, the fuel is clear and bright, while at temperatures below CP the fuel is obviously cloudy. Because conventional diesel fuel operability is not the focus of this study, operability for this fuel was only defined within an 8°C window.

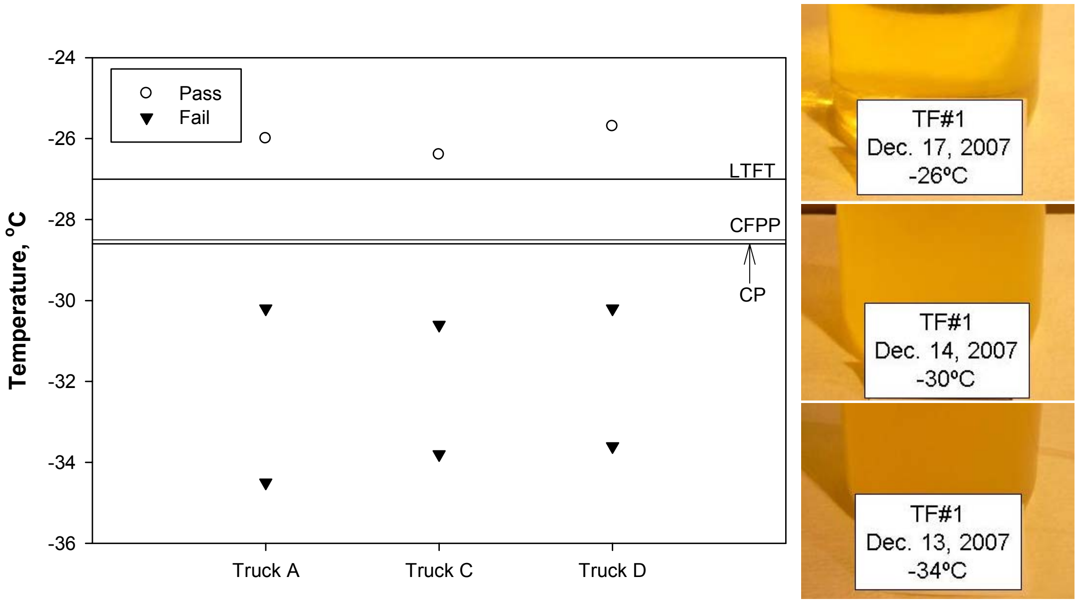


Figure 20. Low temperature operability results for No. 2 diesel fuel (TF#1).

B5 Blends

Low-temperature operability results for test fuels #2, #3, #4, and #9 are shown in Figure 21 – 24, along with photographs of the fuels at the various test temperatures. Notably, TF#2 appears cloudy at a test temperature above its CP. TF#2 was blended from B100A which had a high CSFT. In spite of the cloudiness above CP, all three vehicles were able to operate at very near to or below CP on this fuel. TF#3 and TF#9 are clear and bright at test temperatures above CP. TF#4 is cloudy at the highest test temperature, but this is below CP, and all three vehicles were able to operate on this fuel at this temperature. The cloudiness of TF#2 above CP, combined with the increasing pressure drop observed for this fuel thoughout the passing test, suggests that this fuel could cause operability problems in situations more severe than those simulated here. As noted, the B100 used in making this blend was produced by blending a high CSFT material with a low CSFT material. A B100 with a naturally high CSFT may have caused operability problems above the CP.

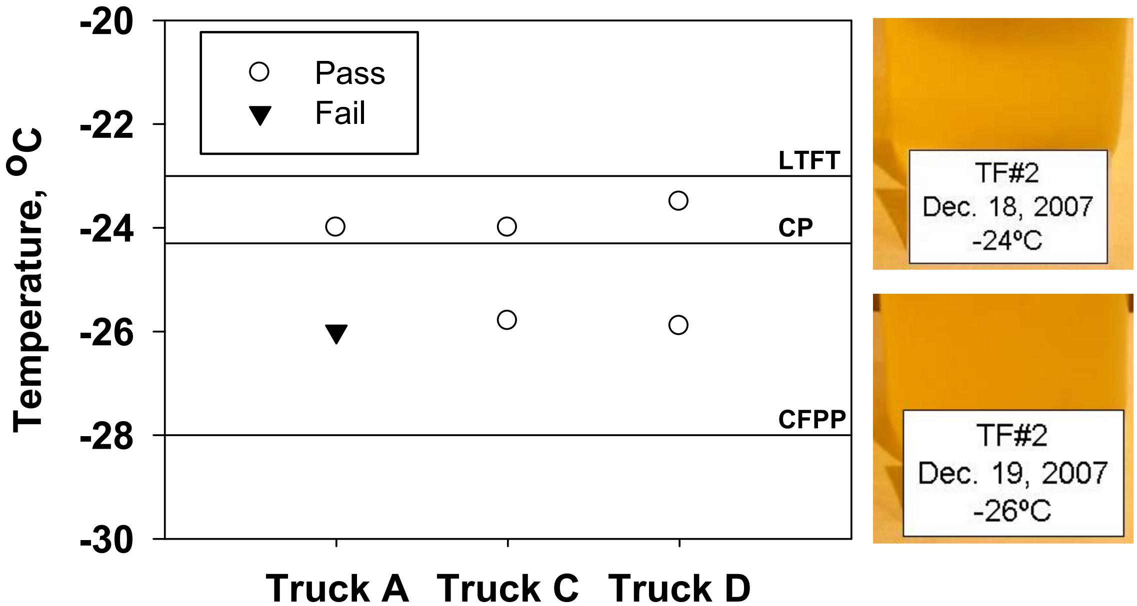


Figure 21. Low temperature operability results for TF #2 (B5 blend with B100A).

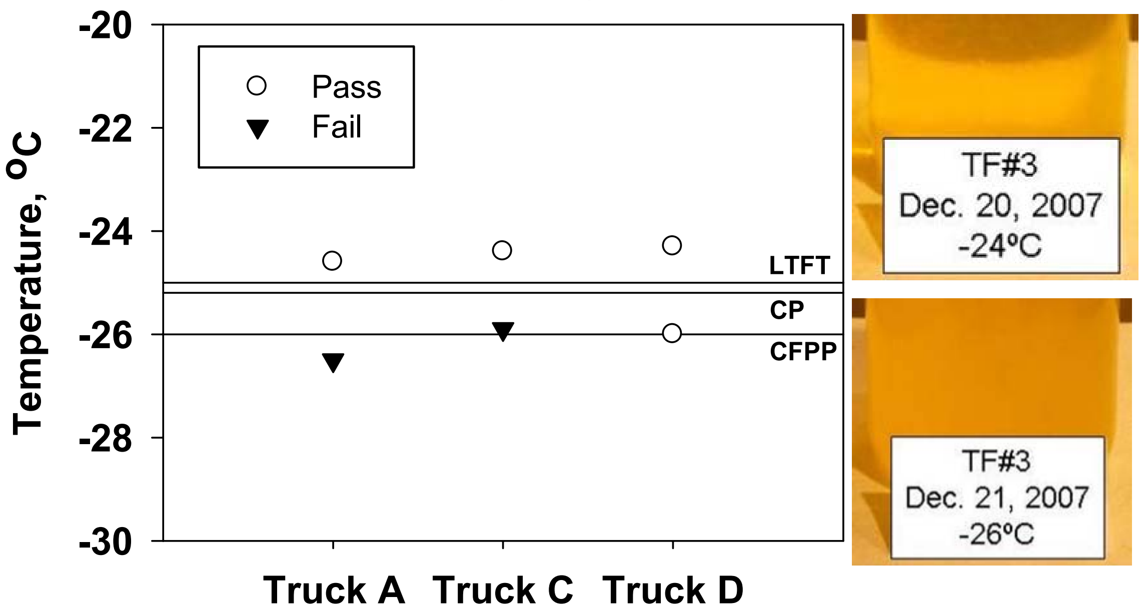


Figure 22. Low temperature operability results for TF #3 (B5 blend with B100B).

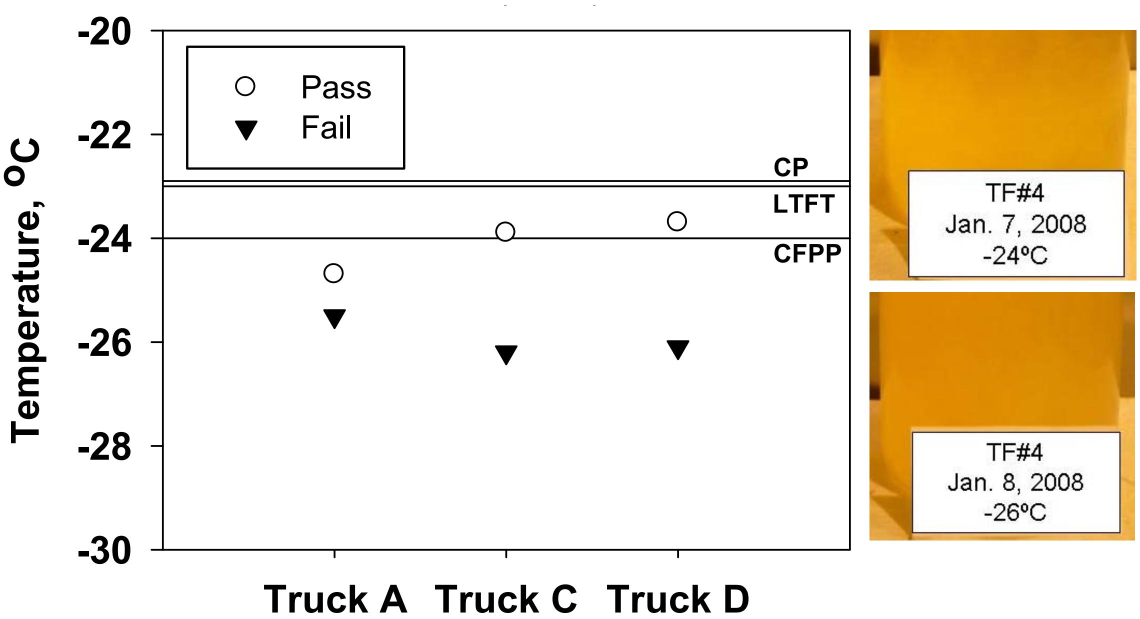


Figure 23. Low temperature operability results for TF #4 (B5 blend with B100C).

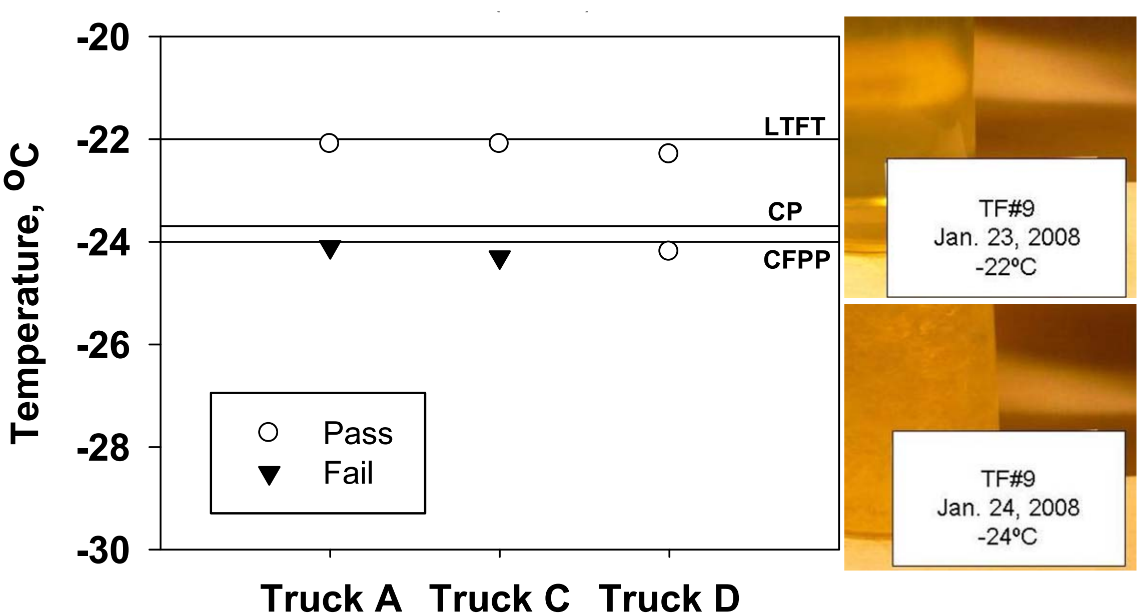


Figure 24. Low temperature operability results for TF #9 (B5 blend with B100D).

B20 Blends

Low-temperature operability results for test fuels #5, #6, #7, #10, #8, and #11 are shown in Figure 25 – 30, respectively, along with photographs of the fuels at test temperature. Test fuel #5 (high CSFT biodiesel) is slightly cloudy at -15°C and shows obvious precipitates at -17°C; the CP of this fuel is -18°C where the fuel appears almost milk-white. Failing tests were obtained in Truck A at all temperatures tested. In hindsight, testing this fuel at significantly higher temperature would have been interesting in order to determine if LTFT was a good measure of operability for this fuel in Truck A.

However, because passing tests were obtained at the higher two temperatures in Trucks C and D, and because Truck A very nearly successfully completed the test at the highest temperature of -15°C, it is also possible that the operability limit is only slightly higher. Nevertheless, Truck A failed to operate on this fuel at temperatures significantly above CP.

For TF#6, clear and bright fuels correspond to passing tests while a cloudy fuel produced failing results. For TF#7 and TF#10, a precipitate has formed for both tests, supporting the idea described above that stirring of the tank could suspend the precipitate and lead to filter plugging and test failure.

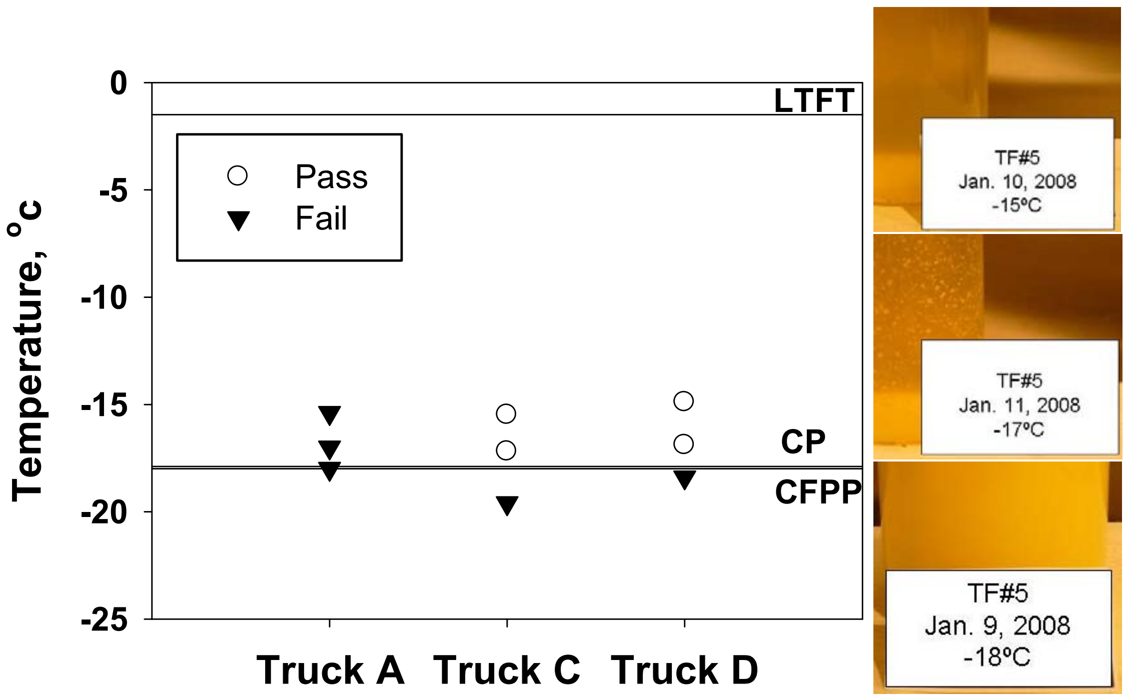


Figure 25. Low temperature operability results for TF #5 (B20 blend with B100A).

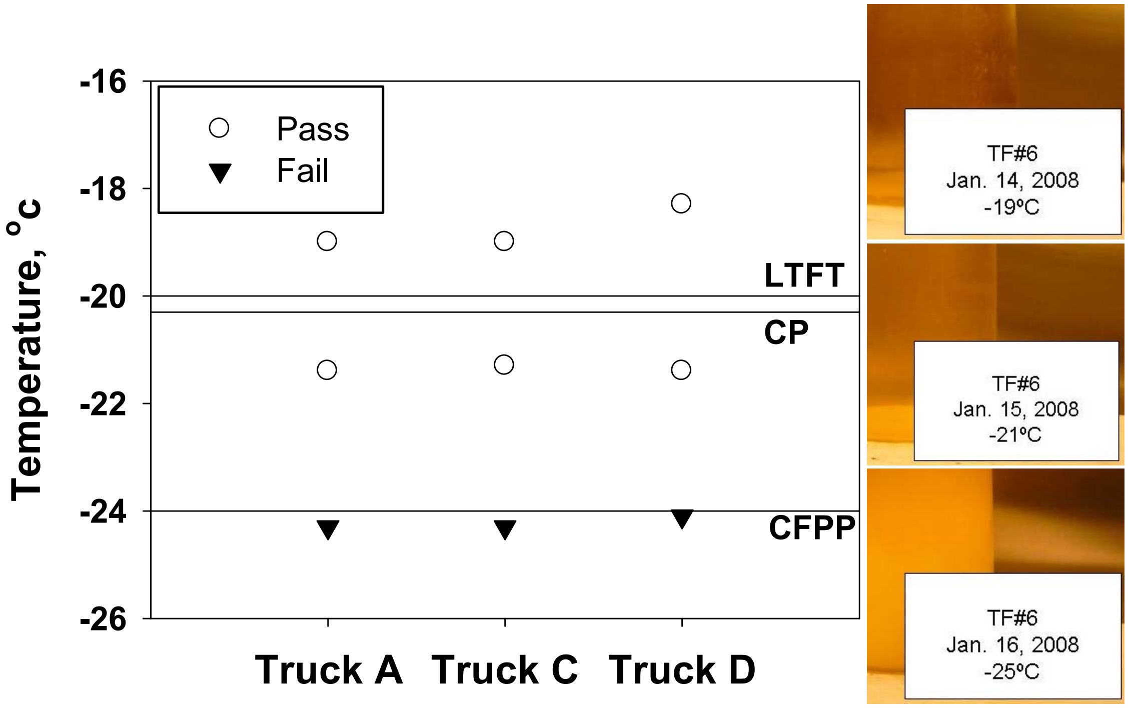


Figure 26. Low temperature operability results for TF #6 (B20 blend with B100B).

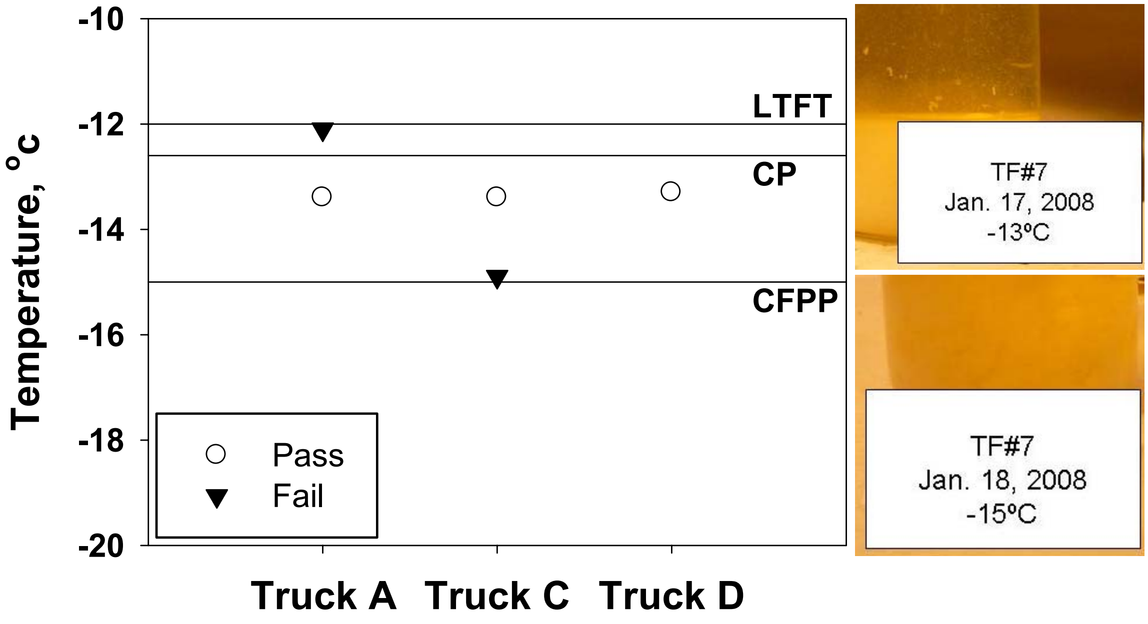


Figure 27. Low temperature operability results for TF #7 (B20 blend with B100C).

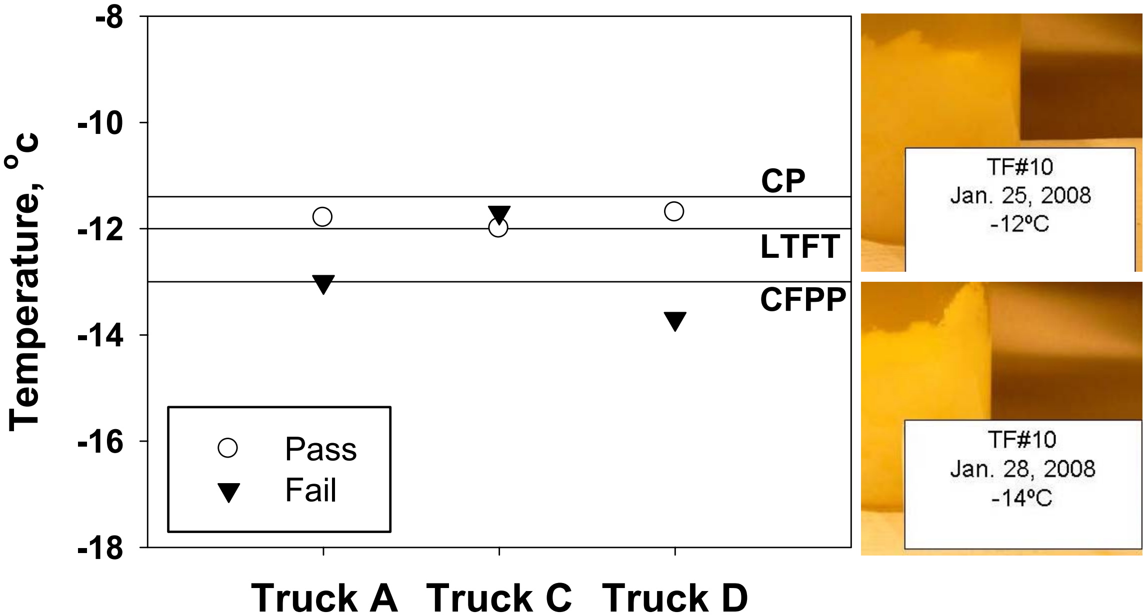


Figure 28. Low temperature operability results for TF #10 (B20 blend with B100D).

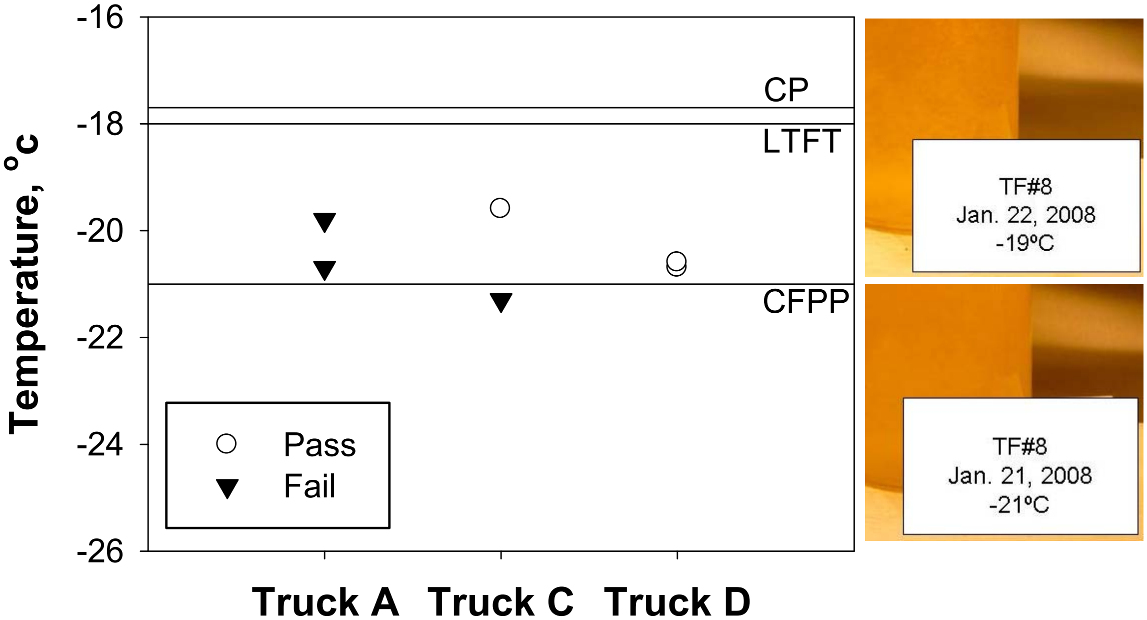


Figure 29. Low temperature operability results for TF #8 (No. 2 Diesel B20 blend with B100B).

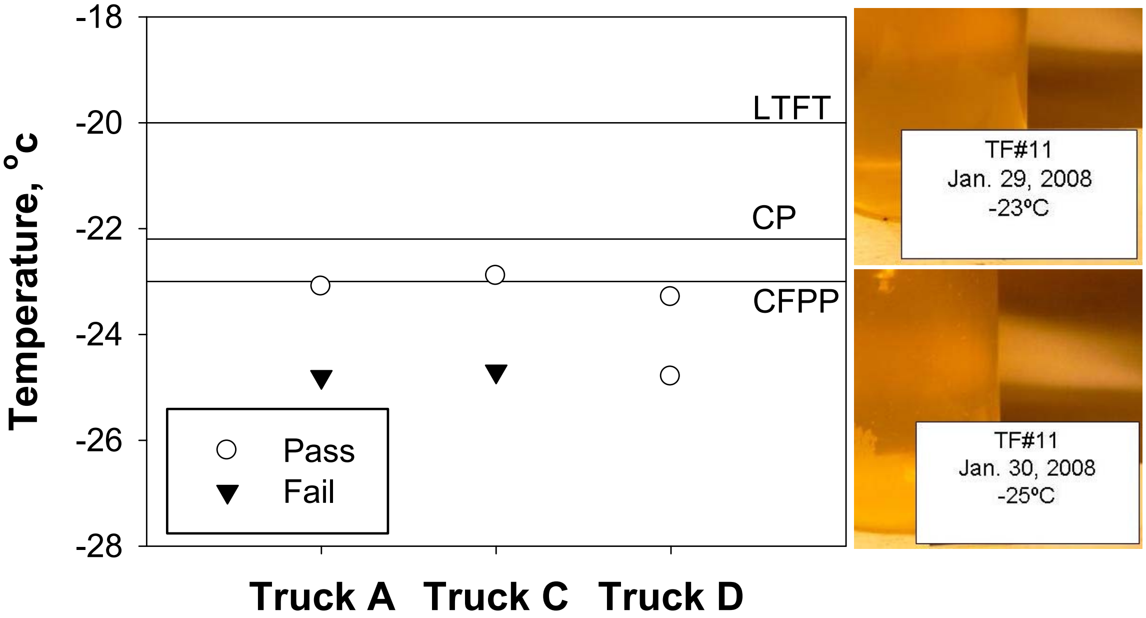


Figure 30. Low temperature operability results for TF #11 (No. 1 Diesel B20 blend with B100B).

Data Analysis

Based on the data presented in the previous section, it is possible to define an EMOT. To be consistent with previous work, the difference between the lowest pass temperature and the highest fail temperature was calculated and the EMOT was arbitrarily decided to be one-third of the temperature difference above the maximum fail temperature or two-thirds below the lowest passing test. Results of this estimation are shown in Table 14. In cases where the result is listed as "greater than," no passing test was obtained. In cases where the result is listed as "less than," no failing test was obtained; in general, these results are at temperatures below the fuel CP.

Table 14. Estimated minimum operating temperature, °C

	Truck A	Truck C	Truck D
TF #1	-28.8	-29.2	-28.7
TF #2	-25.4	<-25.8	<-25.9
TF #3	-25.9	-25.4	<-26
TF #4	-25.2	-25.4	-25.3
TF #5	>-15.4	-18.8	-17.9
TF #6	-23.3	-23.3	-23.2
TF #7	<-13.4	-14.4	-14.4
TF #8	>-19.8	-20.7	<-20.7
TF #9	-23.4	-23.6	<-24.2
TF #10	-12.6	-11.9	-13.0
TF #11	-24.2	-24.1	<-24.8

Figure 31 shows a comparison of EMOT and CP. In this and the subsequent figures, the color (white, black, or grey) of a symbol indentifies the truck. If the symbol is a circle, then an operability limit was determined. If the symbol is a triangle pointing up, then no passing test was obtained and the operability limit is greater than this value, and similarly for a triangle pointing down. Cloud point is an accurate or conservative predictor of EMOT in almost all cases. The one exception is TF#5, the B20 blend produced from the high CSFT B100A which produced failing results at 3°C above CP. A regression line suggests that on average CP was conservative by about 1°C over the full range of CP.

Figure 32 compares EMOT to CFPP and Figure 33 compares EMOT to LTFT. For these vehicles and fuels, CFPP tends to predict an operability limit higher than cloud and may not be protective of the most severe vehicles. LTFT was protective of all vehicles but was very conservative for TF#5 in Trucks C and D.

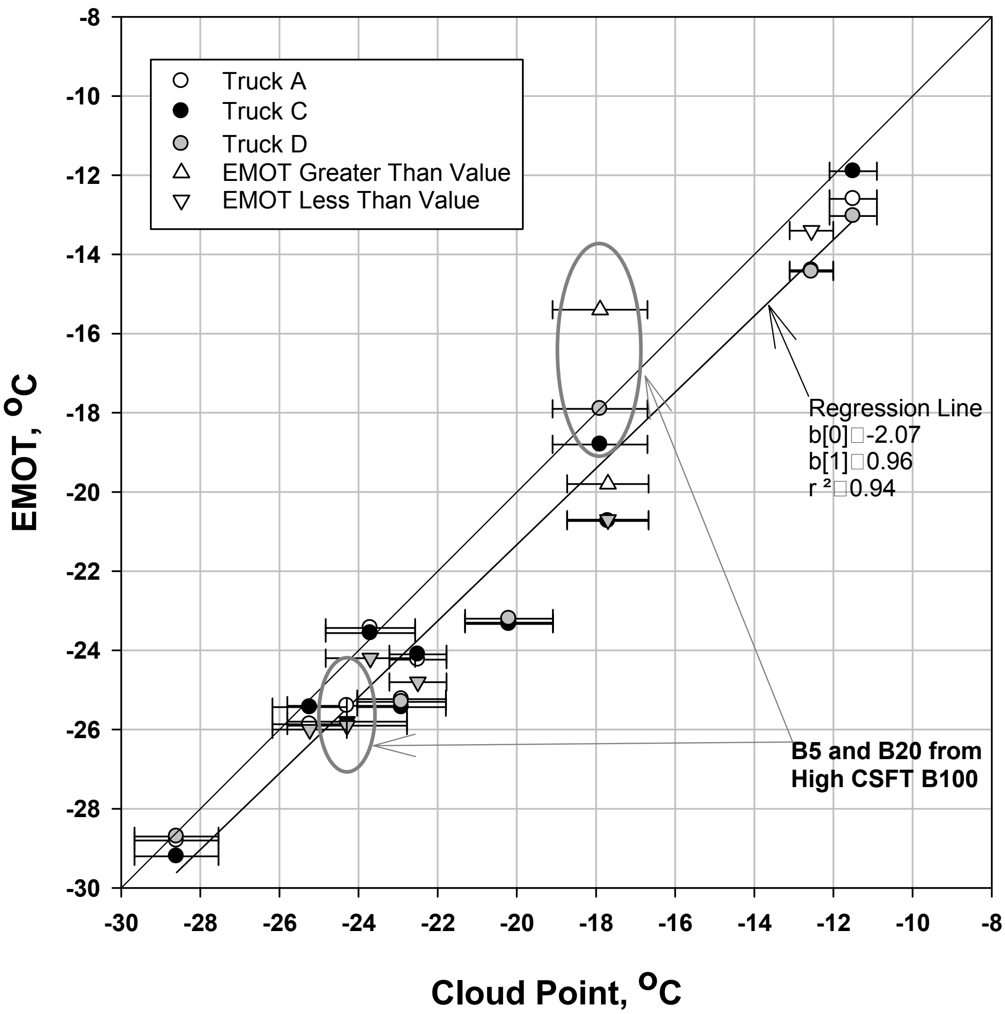


Figure 31. EMOT as a function of CP for all fuels tested (95% confidence interval shown for CP).

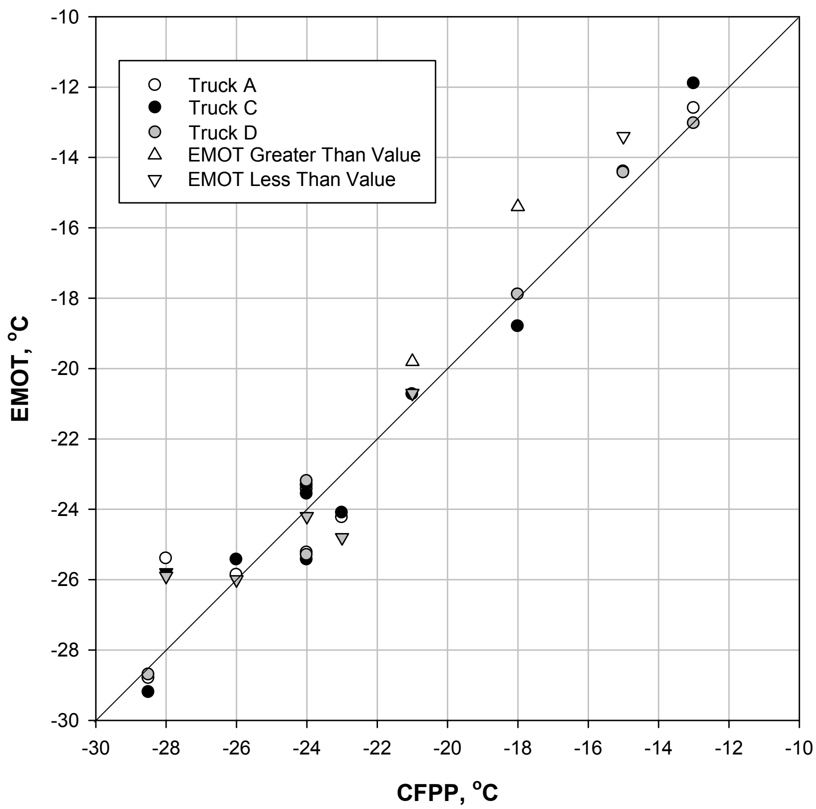


Figure 32. EMOT as a function of CFPP for all fuels tested.

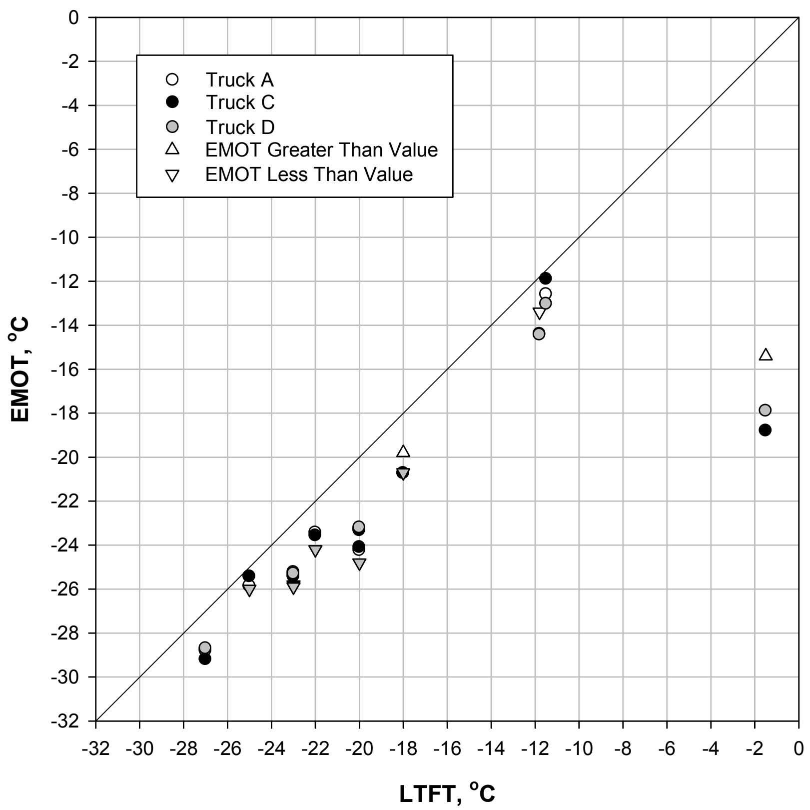


Figure 33. EMOT as a function of LTFT for all fuels tested.

6. Conclusions

1. Cloud point was an accurate or conservative predictor of EMOT in almost all cases. The one exception was TF#5, the B20 blend produced from the high CSFT B100. On average for all fuels combined CP was conservative by about 1°C over the full range of CP. CFPP tends to predict an operability limit higher than cloud and may not be protective of the most severe vehicles. LTFT was protective of all vehicles but was very conservative for TF#5 in Trucks C and D.

2. Overall, biodiesel blends produced from B100 with CSFT of less than 200 seconds performed as predicted by CP or LTFT measurements. These fuels were also clear and bright above CP and did not cause significant fuel filter blockage and pressure drop above CP. In many cases these fuels could operate at temperatures 1°C to 2°C below CP.

3. The B5 blend prepared from the high CSFT B100 appeared cloudy at a test temperature 1°C above its CP. In spite of the cloudiness above CP, all three vehicles were able to operate at very near to or below CP on this fuel. Passing tests exhibited continuously increasing fuel filter pressure drop for this fuel in Truck A, even at temperatures slightly above CP. This was not observed for passing tests with other B5 blends. The cloudiness of this test fuel above CP, combined with the increasing pressure drop observed for this fuel throughout the passing test, suggests that it could cause operability problems in situations more severe than those simulated here. Also, the B100 was produced by blending a high CSFT B100 with a second B100 having CSFT <200 seconds. A B100 with a naturally high CSFT may have caused operability problems above CP at the B5 level.

4. The B20 blend prepared from the high CSFT B100 was slightly cloudy at -15°C and showed obvious precipitates at -17°C, yet the CP of this fuel was -18°C where the fuel appears almost milk-white. Failing tests were obtained in Truck A at all temperatures tested, up to 3°C above CP The truck manufacturer represents roughly 30% of the North American Class 8 truck market, implying that B100 with high CSFT may cause unanticipated low-temperature operability problems.

5. This study showed that high CSFT B100 can cause low-temperature operability problems at temperatures above CP for B20 blends. It is recommended that additional testing of B5 blends from other, potentially more representative, high CSFT B100 be performed.

6. It was shown that movement of the trucks could result in mixing of the vehicle fuel tank and suspension of materials that had precipitated to the bottom causing a failing test. The effects found for stirring of the fuel tank may indicate that vehicle dynamometer tests do not capture all failure modes that can be experienced in actual vehicle operation.

Appendix I: Base Diesel Characterization – Details

Table 15. ASTM D975 characterization for No. 2 diesel fuel.

	Lab D	**Lab A**	**Units**	**Method**
Kinematic Viscosity at 40 C	2.11	2.126	mm2/s	D445
Flash Pt, Pensky-Martens Closed	66.5	58.3	°C	D93
Carbon Residue by MCRT	<0.0001		mass%	D4530
Sulfur by Xray	0.0005	<0.001	mass%	D2622
Ash from Petroleum Products	<0.001	<0.001	mass%	D482
Corrosion of Cu	1A	1A		D130
Cloud Point/Phase Tech	-27.4	-30	°C	D5773
Distillation				D86
Initial Boiling Point	175.3	176.3	°C	
5 Vol Percent Recovered	195.7	194.4	°C	
10 Vol Percent Recovered	204.9	205.1	°C	
20 Vol Percent Recovered	216.7	216.7	°C	
30 Vol Percent Recovered	226.3	225.7	°C	
40 Vol Percent Recovered	235.4	234.0	°C	
50 Vol Percent Recovered	243.2	242.5	°C	
60 Vol Percent Recovered	252.3	251.4	°C	
70 Vol Percent Recovered	263.1	261.7	°C	
80 Vol Percent Recovered	275.8	273.9	°C	
90 Vol Percent Recovered	294.1	291.2	°C	
95 Vol Percent Recovered	310	305.6	°C	
Final Boiling Point	323.7	322.2	°C	
% Recovered	96.4	98.2	Vol%	
% Residue	1.5	0.5	Vol%	
% Loss	2.1	1.3	Vol%	
Barometric Pressure	760		Torr	
Derived Cetane Number (IQT)	39.73			D6890
Hydrocarbon Types by FIA				D1319
Total Aromatics	26.7	27.9	Vol%	
Total Olefins	2.3	1.1	Vol%	
Total Saturates	71	71	Vol%	
HFRR (Wear Scar Average)	441	435	micron	D6079
Density of Liquids, 60°F	0.8134		g/mL	D4052
Water and sediment		0.01	Vol%	D2709
Pour point (Lab G)	-36		°C	D5950
CFPP (Lab G)	-25		°C	D6371
LTFT (Lab G)	-27		°C	D4539

Table 16. ASTM D975 characterization for No. 1 diesel fuel.

	Lab D	Lab A	Units	Method
Kinematic Viscosity at 40 C	1.379	1.385	mm2/s	D445
Flash Pt, Pensky-Martens Closed	50.5	43.9	°C	D93
Carbon Residue by MCRT	<0.0001		mass%	D4530
Sulfur by Xray	0.0005	<0.001	mass%	D2622
Ash from Petroleum Products	0.001	<0.001	mass%	D482
Corrosion of Cu	1A	1A		D130
Cloud Point/Phase Tech	-47.9	-51	°C	D5773
Distillation				D86
Initial Boiling Point	149.4	155.1	°C	
5 Vol Percent Recovered	166.5	166.7	°C	
10 Vol Percent Recovered	169.9	172.8	°C	
20 Vol Percent Recovered	176.9	176.6	°C	
30 Vol Percent Recovered	185.2	184.9	°C	
40 Vol Percent Recovered	195	194.6	°C	
50 Vol Percent Recovered	205.6	204.7	°C	
60 Vol Percent Recovered	217	216.1	°C	
70 Vol Percent Recovered	229.7	228.2	°C	
80 Vol Percent Recovered	243.9	242.1	°C	
90 Vol Percent Recovered	261.8	259.7	°C	
95 Vol Percent Recovered	275.3	272.1	°C	
Final Boiling Point	289.3	287.7	°C	
% Recovered	96.8	98.6	Vol%	
% Residue	1.4	0.5	Vol%	
% Loss	1.8	0.9	Vol%	
Barometric Pressure	760		Torr	
Derived Cetane Number (IQT)	38.46			D6890
Hydrocarbon Types by FIA				D1319
Total Aromatics	20.4	22.9	Vol%	
Total Olefins	3.2	0.8	Vol%	
Total Saturates	76.4	76.3	Vol%	
HFRR (Wear scar average)	385	345	micron	D6079
Density of Liquids at 60°F	0.7827		g/mL	D4052
Pour point (Lab G)	-51		°C	D5950

Appendix II: Cold Soak Filtration Procedure

Determination of Fuel Filter Blocking Potential of Biodiesel (B100) Blend Stock by Cold Soak Laboratory Filtration

A1.1 Scope

A1.1.1 This test method covers the determination by filtration time after cold soak of the suitability for a Biodiesel (B100) Blend Stock for blending with middle distillates to provide adequate low temperature operability performance to at least the cloud point of the finished blend.
A1.1.2 The interim precision of this test method has been determined.
A1.1.3 The values stated in SI units are to be regarded as the standard.
A1.1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

A1.2 Referenced Documents

A1.2.1 *ASTM Standards*[2]*:*
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems
D5452 Test Method for Particulate Contamination in Aviation Fuels by Laboratory Filtration
Research Report D02: RR-XXXX, Interlaboratory Study to Establish Precision Statements for ASTM Dxxxx-xx, Standard Test Method for Cold Soak Filtration of Biodiesel B100 Blend Stock

A1.3 Terminology

A1.3.1 *Definitions:*
A1.3.1.1 *bond*, *v*—to connect two parts of a system electrically by means of a conductive wire to eliminate voltage differences.
A1.3.1.2 *ground*, *v*—to connect electrically with earth.
A1.3.1.3 *Biodiesel*, *n*—a fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats, designated B100
A1.3.2 *Definitions of Terms Specific to This Standard:*
A1.3.2.1 *glass fiber filter*, *n*—the 0.7 micron glass fiber filters used in this test method.
A1.3.2.2 *filtered flushing fluids*, *n*—either of two solvents, heptane or 2,2,4-trimethylpentane, filtered through a nominal 0.45 μm glass fiber filter.
A1.3.3 Abbreviations:
A1.3.3.1 CSFT – cold soak filtration test

A1.4 Summary of Test Method

A1.4.1.1 300 mL of biodiesel (B100) is stored at 4.4°C (40°F) for 16 hours, allowed to warm to 20-22°C (68-72°F), and vacuum filtered through a single 0.7 μm glass fiber filter.
A1.4.1.2 The filtration time in seconds is reported.

A1.5 Significance and Use

A1.5.1 Some substances that are soluble or appear to be soluble in biodiesel at room temperature will, upon cooling or standing at room temperature for extended periods, come out of solution. These substances can cause filter plugging. This method provides an accelerated means of assessing the propensity for these substances to plug filters.
A1.5.1.1 Fuels that give short filtration times are expected to give satisfactory operation down to the cloud point of biodiesel blends.
A1.5.2 The test method can be used in specifications as a means of controlling levels of minor filter plugging components in biodiesel and biodiesel blends.

A1.6 Apparatus

A1.6.1 *Filtration System*—Arrange the following components as shown in Fig. 1.
A1.6.1.1 *Funnel and Funnel Base*, with a stainless steel filter support for a 47 mm diameter glass fiber filter, and locking ring or spring action clip, capable of receiving 300 mL.
NOTE 1-Sintered glass supports were found to give much higher filtration times during initial studies and should not be used.
A1.6.1.2 *Ground/Bond Wire*, 0.912-2.59 mm (No. 10 through No. 19) bare stranded flexible, stainless steel or copper installed in the flasks and grounded as shown in Fig. 1.
NOTE 2—The electrical bonding apparatus described in Test Method D 5452 or other suitable means of electrical

grounding which ensure safe operation of the filtration apparatus and flask can be used. If the filtrate is to be subsequently tested for stability it is advisable not to use copper as copper ions catalyze gum formation during the stability test.

A1.6.1.3 *Receiving Flask*, 1 L borosilicate glass vacuum filter flask, which the filtration apparatus fits into, equipped with a sidearm to connect to the safety flask.

A1.6.1.4 *Safety Flask*, 1 L borosilicate glass vacuum filter flask equipped with a sidearm to connect the vacuum system. A fuel and solvent resistant rubber hose through which the grounding wire passes shall connect the sidearm of the receiving flask to the tube passing through the rubber stopper in the top of the safety flask.

A1.6.1.5 *Vacuum System*, a vacuum system capable of producing a vacuum of 70 to 100 kPa below atmospheric pressure when measured at the receiving flask. A mechanical vacuum pump may be used if it has this capability.

NOTE 3—Water aspirated vacuum will not provide relative vacuum within the prescribed range.

A1.6.2 *Other Apparatus*:

A1.6.2.1 *Forceps*, approximately 12 cm long, flat-bladed, with non-serrated, non-pointed tips.

A1.6.2.2 *Graduated Cylinders*, to contain at least 0.5 L of fluid and marked at 10 mL intervals. 100 mL graduated cylinders may be required for samples which filter slowly.

A1.6.2.3 *Petri Dishes*, approximately 12.5 cm in diameter, with removable glass supports for glass fiber filters.

NOTE 4—Small watch glasses, approximately 5 to 7 cm in diameter, have also been found suitable to support the glass fiber filters.

NOTE 5 – B100 will dissolve some plastics. This can cause the filters to adhere to the plastic.

A1.6.2.4 *Glass fiber Filters***,** plain, 47-mm diameter, nominal pore size 0.7-μm.

A1.6.2.5 Protective Cover, polyethylene film or clean aluminum foil.

A1.6.2.6 *Liquid or air bath or chamber* capable of sustaining a temperature of 4.4°C +/-1.1°C (40°F +/-2°F) for 16 hours.

A1.6.2.7 *Timer* capable of displaying elapsed times of at least 900 seconds to the nearest 0.1 second.

A1.7 Reagents and Materials

A1.7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

A1.7.2 *Flushing Fluids*: Flushing fluids are not required for the test as the filter is not weighed. However, heptane or isooctane may be used to wash the apparatus after filtration to remove any residue. Alternatively soap and water may be used per A1.7.3.

A1.7.2.1 *Heptane*, (**Warning**—Flammable.)

A1.7.2.2 *2,2,4-trimethylpentane (isooctane)* , (**Warning**—Flammable.)

A1.7.3 *Liquid or Powder Detergent*, water-soluble, for cleaning glassware.

A1.8 Preparation of Apparatus and Sample Containers

A1.8.1 Clean all components of the filtration apparatus using the reagents described in A1.7.2 and A1.7.3.

A1.8.1.1 Remove any labels, tags, and so forth.

A1.9 Sampling

A1.9.1 The sample container should be 500 mL (±15 mL) in volume and have a screw on cap with an inert liner. Glass containers are preferred to facilitate a visual inspection of the contents and the container before and after filling. Glass containers also allow for visual inspection of the container, after the sample is emptied, to confirm complete emptying of the container. Epoxy lined sample cans, polytetrafluoroethylene (PTFE) bottles, and high density linear polyethylene bottles have also been found suitable as sample containers but are less desirable since visual inspection of the interior of the container is more difficult.

A1.9.2 Precautions to avoid sample contamination shall include selection of an appropriate sampling point. Samples should preferentially be obtained dynamically from a sampling loop in a distribution line, or from the flushing line of a field sampling kit. Ensure that the line to be sampled is flushed with fuel before collecting the sample.

A1.9.2.1 Use clean sample containers.

A1.9.2.2 Keep a clean protective cover over the top of the sample container until the cap is installed. Similarly protect the funnel opening of the assembled filtration apparatus with a clean protective cover until ready for use.
A1.9.2.3 Where it is desirable or only possible to obtain samples from static storage, follow the procedures given in Practice D 4057 or equivalent, taking precautions for cleanliness of all equipment used. The sample should pass through a minimum number of intermediate containers prior to placement in the prepared container.
A1.9.2.4 Samples obtained from static storage can give results which are not representative of the bulk contents of the tank because of particulate matter settling. Where possible, the contents of the tank should be circulated or agitated before sampling, or the sampling performed shortly after a tank has been filled.
A1.9.3 Visually inspect the sample container before taking the samples to verify that there are no visible particles present inside the container. Fill the sample container to contain 300 mL. Protect the fuel sample from prolonged exposure to light by wrapping the container in aluminum foil or storing it in the dark to reduce the possibility of particulate formation by light-promoted reactions. Do not transfer the fuel sample from its original sample container into an intermediate storage container. If the original sample container is damaged or leaking, then a new sample shall be obtained.
A1.9.3.1 If a 500 mL bottle is not available, or the sample has already been received in a container not suitable for this test, follow A1.9.5.
A1.9.4 Analyze fuel samples as soon as possible after sampling.
A1.9.4.1 Upon receipt of a Biodiesel Blend Stock (B100) sample, the entire sample shall be heated to 40°C for at least 3 hours under an inert atmosphere to erase any thermal history and to dissolve any solids that might have precipitated during transit unless it is known that the sample has never been cooled below 20°C. If the sample has never been exposed to temperatures below 20°C then proceed to A1.9.5.
A1.9.4.2 After heating for the required time, allow the sample to sit for 24 hours at a temperature no lower than 20°C.
A1.9.5 Shake the sample vigorously for 1 minute and transfer 300 mL to a clean fresh 500 mL (±15 mL) bottle.

A1.10 Preparation of Glass Fiber Filter

A1.10.1 Each filtration uses one filter. The glass fiber filter used for each individual test shall be identified by marking the petri dishes used to hold and transport the filters.
A1.10.2 Clean all glassware used in preparation of glass fiber filter as described in A1.8.1.
A1.10.3 Using forceps place the filters on clean glass support rods, or watch glasses, in petri dish.
A1.10.4 Place the petri dish with its lid slightly ajar, in a drying oven at 90 ± 5°C and leave it for 30 min.
A1.10.5 Remove the petri dish from the drying oven. Keep the petri dish cover ajar, such that the filter is protected from contamination from the atmosphere. Allow 30 min for the filter to come to equilibrium with room air temperature and humidity.
A1.10.6 Using clean forceps, place the filter centrally on the filter support of the filtration apparatus (see Fig. A1.1). Install the funnel and secure with locking ring or spring clip. Do not remove the plastic film from the funnel opening until ready to start filtration.

A1.11. Procedure

A1.11.1Place 300 mL of sample in a glass 500 mL bottle and set in a liquid or air bath or chamber at 4.4°C +/- 1.1°C (40°F ±2°F) for 16 ± 0.5 hours.
A1.11.2 After the 16 hour cold soak is completed, allow the sample to come back to room temperature (20 – 22°C / 68 – 72°F/) on its own without external heating. The sample shall be completely liquid before filtration. The sample shall be filtered within 1 hour after reaching 20-22°C (68-72°F).
A1.11.3 Complete assembly of the receiving flask, 0.7 micron glass fiber filter and funnel as a unit (see Fig. 1) before swirling the sample. To minimize operator exposure to fumes, the filtering procedure should be performed in a fume hood.
A1.11.4 Start the vacuum system. Record the pressure in the system after one minute of filtration. The vacuum shall be between 71.1 and 84.7 kPa (21 and 25 inches of Hg) below atmospheric pressure. If the vacuum is not within the specified range, make adjustments to the vacuum system.
A1.11.5 Thoroughly clean the outside of the sample container in the region of the cap by wiping it with a damp, lint-free cloth. Swirl the container vigorously for about 2-3 seconds to dislodge any particles that may have adhered to the walls of the container.
A1.11.6 Immediately after swirling, pour the entire contents of the sample container into the filtration

funnel and simultaneously start the timer. The entire contents of the sample container shall be filtered through the glass fiber filter to ensure a correct measure of the contamination in the sample.

NOTE 6 – Care must be taken not to shake the sample vigorously as this could cause some of the solids to go back into solution.

A1.11.7 If the filtration is not complete when 720 seconds (12 minutes) has elapsed, turn off the vacuum system and record the duration of the filtration to the nearest second. Record the pressure in the system and the volume filtered just before the termination of the filtration.

A1.12 Reporting

A1.12.1 Report the time for the 300 mL B100 to be completely filtered as B100 filtration time in seconds.

A1.12.2 If the filtration of the 300 mL failed to be completed after 720 seconds, report the volume which was filtered after 720 seconds.

A1.13 Precision and Bias

A1.13.1.1 *Precision*—The precision of this test method for B100 filtration has not yet been determined.

A1.13.1.1.1 *Repeatability*— The difference between successive test results, obtained by the same operator using the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of this test method, exceed 0.1689(X + 1.2018) time (s) only in one case in twenty.

A1.13.1.1.2 *Reproducibility*— The difference between the two single and independent results obtained by different operators working in different laboratories on identical test material for B100 filtration has not yet been determined.

A1.13.1.2 *Interim Precision*-Repeatability and reproducibility determinations were made using data from the ASTM Biodiesel Low Temperature Operability Task Force. The analysis of the data is the subject of Research Report D02: RR-XXXX. The report is an attempt to supply such an analysis based on well-established methodologies. Subsequent to test method publication a more thorough round robin is planned.

	200 sec	360 sec
Repeatability	34.0	61.0
Reproducibility	115.9	208.1

NOTE 7 – *Interim Precision*- repeatability and reproducibility determinations were made using data from the ASTM Operability Task Force. *The degree of freedom associated with the repeatability estimate from this round robin study is 25 for repeatability which is below 30 but acceptable. The degree of freedom associated with the reproducibility estimate from this round robin study is 10 and below acceptable limits. For that reason only the repeatability is included in section A1.13.1 Since the minimum requirement of 30 (per practice D 6300) is not met, users are cautioned that the actual repeatability/reproducibility may be significantly different than these estimates.*

An ASTM ILS will be conducted in future.[3]

A1.13.2 *Bias*—The procedure given for the determination of B100 filtration time has no bias because the value of the filtration time is defined in terms of this test method.

A1.14. Keywords

A1.14.1 biodiesel; diesel fuel; glass fiber filter; filter blocking potential, cold soak filtration test, CSFT, biodiesel blend; laboratory filtration; glass fiber filter; low temperature operability, middle distillate fuel.

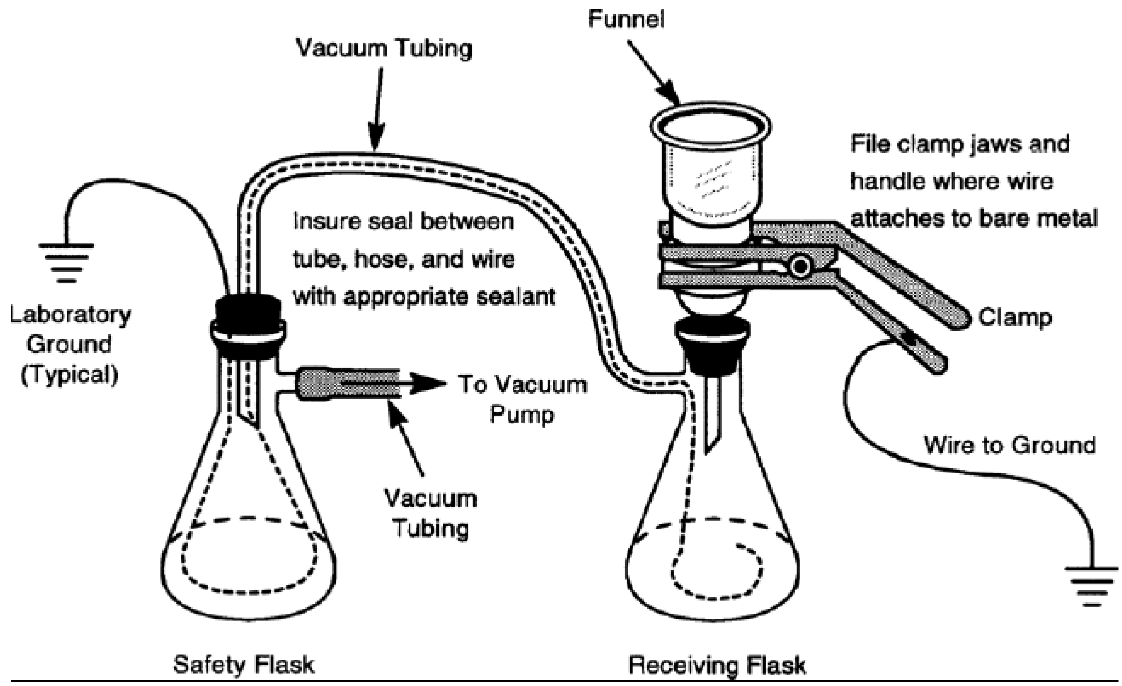


FIG. A1.1 Schematic of Filtration System

Appendix III: B100 Characterization–Additional Results

Table 17. FAME speciation for B100 samples (wt%, normalized to 100%).

Lab	F	K	O	P	Q
FAME	B100A				
12:0					
14:0		0.6		0.7	
16:0	21.3	21.9	22.1	21.6	22.7
16:1		0.5		0.5	
17:0					
18:0	1.4	3.0	3	2.9	3.1
18:1 cis	17.8	16.7	16.9	16.5	17.4
18:1 trans				0.9	
18:2	59.4	55.6	56.6	55.6	56.8
20:0		0.2			
18:3	0.1	1.2	1.3	1.2	
20:1		0.2			
Lab	F	K	O	P	Q
FAME	B100B				
12:0					
14:0		0.1			
16:0	9.3	10.9	11	10.6	11.0
16:1		0.1			
17:0					
18:0	3.4	4.8	4.7	4.8	4.9
18:1 cis	25.6	23.7	23.7	23.3	24.2
18:1 trans				1.6	
18:2	55.3	53.0	53.5	52.3	52.7
20:0		0.4		0.5	
18:3	6.4	6.9	7.2	6.9	7.2
20:1		0.1			
Lab	F	K	O	P	Q
FAME	B100C				
12:0					
14:0		1.4		1.4	1.4
16:0	23.1	24.0	24.8	22.5	24.4
16:1	1.8	2.4		2.3	2.5
17:0				0.4	
18:0	10.9	12.0	12.2	11.3	12.3
18:1 cis	46.4	40.4	43.1	38.4	41.3
18:1 trans				4.1	
18:2	17.7	17.8	18.8	17.2	18.0
20:0		0.2			
18:3		1.1	1.1	1.1	
20:1		0.8		0.8	

Table 17. Continued.

Lab	F	K	O	P	Q
FAME	B100D				
12:0					
14:0	1.1	2.7		2.5	2.3
16:0	26.6	27.8	28.4	25.5	24.7
16:1	2.2	3.0		3.2	3.1
17:0				0.9	
18:0	15.0	16.1	16.2	14.9	14.4
18:1 cis	45.4	38.7	43.3	36.5	34
18:1 trans				5.5	3.4
18:2	9.8	10.4	11.3	9.7	17.7
20:0		0.2			
18:3		0.7	0.3	0.8	
20:1		0.4			

Table 18. Specific gravity of B100 samples.

	Specific Gravity at 60˚F
Lab	K
B100A	0.8833
B100B	0.8847
B100C	0.8775
B100D	0.8755

Appendix IV: Additional Blend Characterization Results

Table 19. Pour point results for B5 and B20 blends (°C).

Lab	Pour	TF#2	TF#3	TF#4	TF#9	TF#5	TF#6	TF#7	TF#10	TF#8	TF#11
	Method	B5A	B5B	B5C	B5D	B20A	B20B	B20C	B20D	B/No. 2	B/No. 1
B	D97	-34	-42	-38	-31	-21	-27	-17	-15.5	-26	
G	D5950	-30	-33	-30		-21	-27	-15	-15	-24	-48
H	D5950	-30	-30		-30	-18		-15			
J	D5949	-33	-36	-33	-30	-21	-24	-15	-15	-21	-27
Average		**-31.8**	**-35.3**	**-33.7**	**-30.3**	**-20.3**	**-26.0**	**-15.5**	**-15.2**	**-23.7**	**-37.5**

Table 20. Oxidation stability by EN14112 for B5 and B20 blends (hours).

Lab	IP	TF#2	TF#3	TF#4	TF#9	TF#5	TF#6	TF#7	TF#10	TF#8	TF#11
		B5A	B5B	B5C	B5D	B20A	B20B	B20C	B20D	B/No. 2	B/No. 1
J	EN14112	15.1	15.3	14.8	30	8.7	12.7	6.7	11.6	13	11.6
M	EN14112	>20	>20	>20	17.7	13.1	11	>20	12.7		

Table 21. Biodiesel content of B5 and B20 blends.

Lab		TF#2	TF#3	TF#4	TF#9	TF#5	TF#6	TF#7	TF#10	TF#8	TF#11
		B5A	B5B	B5C	B5D	B20A	B20B	B20C	B20D	B/No. 2	B/No. 1
J	EN14078, wt%	5.43	5.33	5.39	5.49	23.13	22.78	23.39	23.42	22.86	21.37
	Vol%*	5.00	4.90	5.00	5.10	20.90	20.55	21.27	21.35	21.02	18.91

*Estimated from wt% and specific gravity of blending components.

Table 22. Viscosity at low temperature measured for several biodiesel blends.

	D445 Viscosity, cSt		
	0C	-18C	-40C
TF#2 (B5A)	5.50	10.41	Solid
TF#3 (B5B)	5.50	10.38	Solid
TF#9 (B5D)	5.48	10.38	Solid
TF#6 (B20A)	4.70	wax crystals	Solid
TF#10 (B20D)	4.86	Solid	Solid

Appendix V: Truck Speed Traces

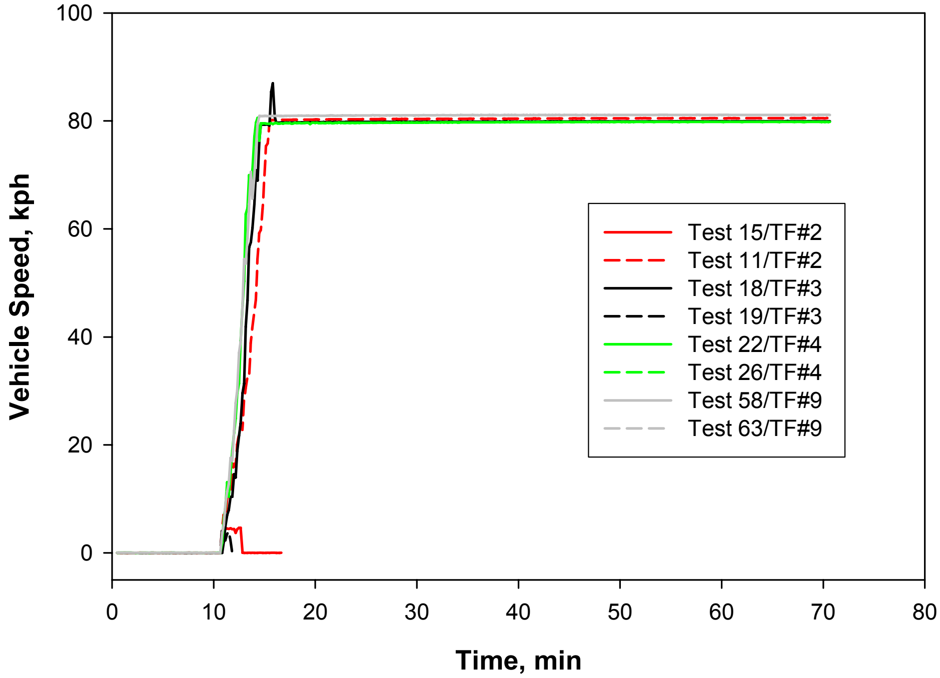


Figure 34, Vehicle speed traces for B5 blends tested in Truck A.

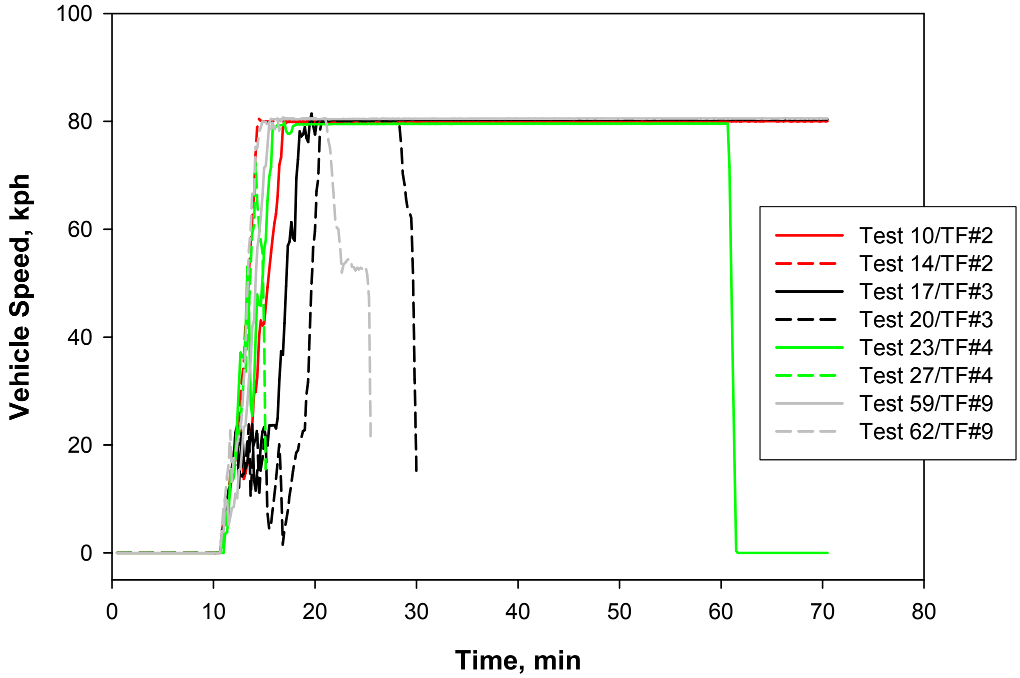


Figure 35. Vehicle speed traces for B5 blends tested in Truck C.

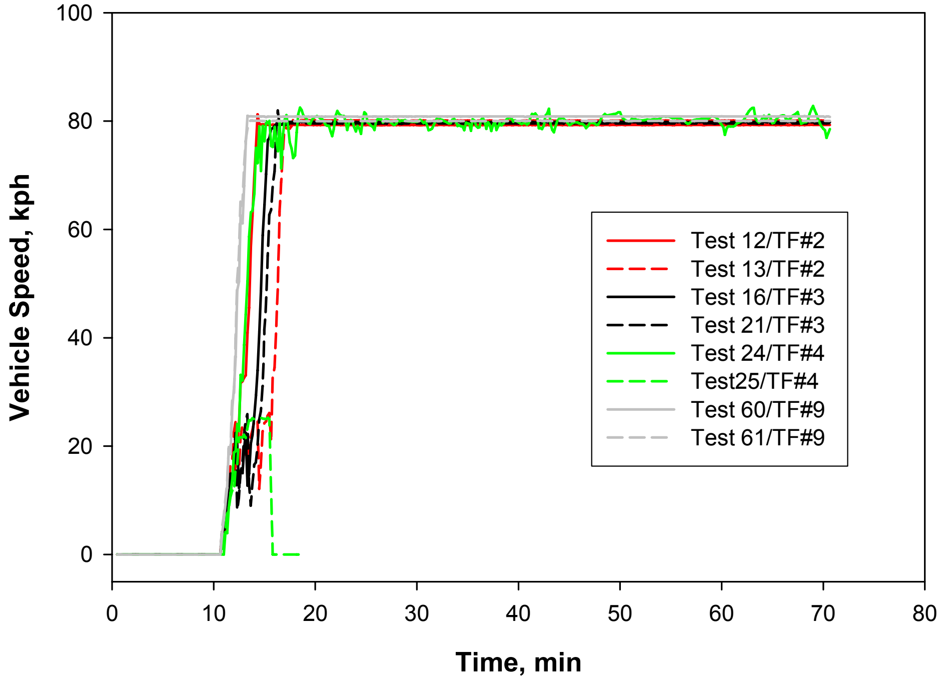


Figure 36. Vehicle speed traces for B5 blends tested in Truck D.

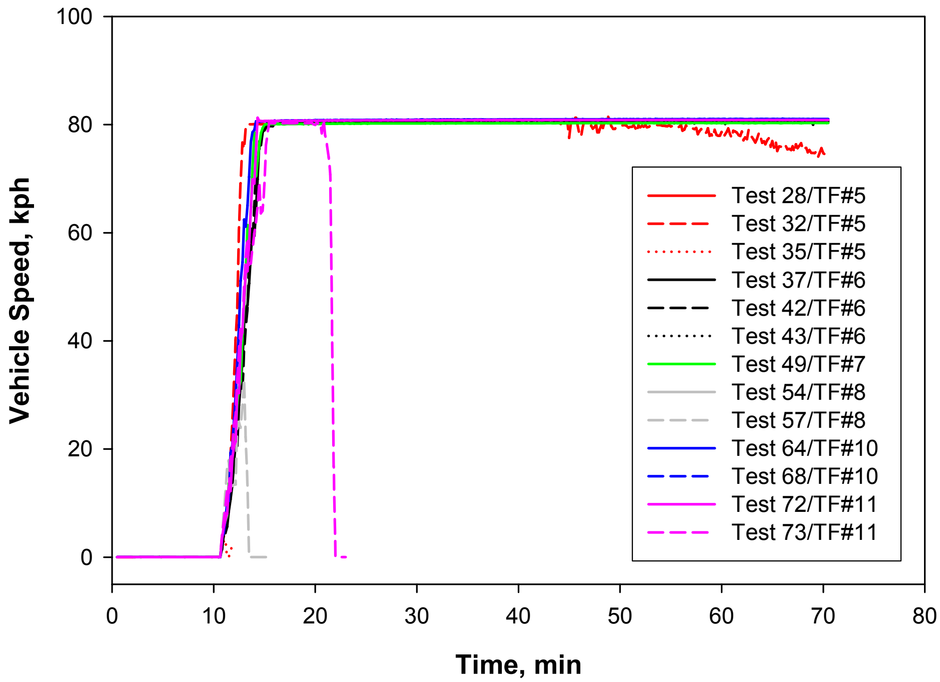


Figure 37. Vehicle speed traces for B20 blends tested in Truck A.

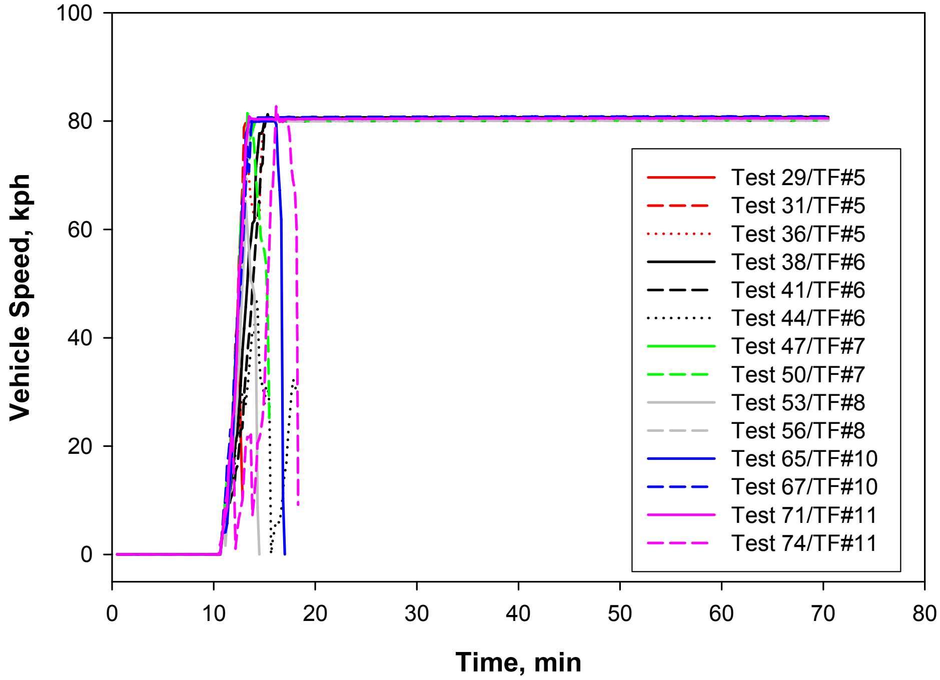


Figure 38. Vehicle speed traces for B20 blends tested in Truck C.

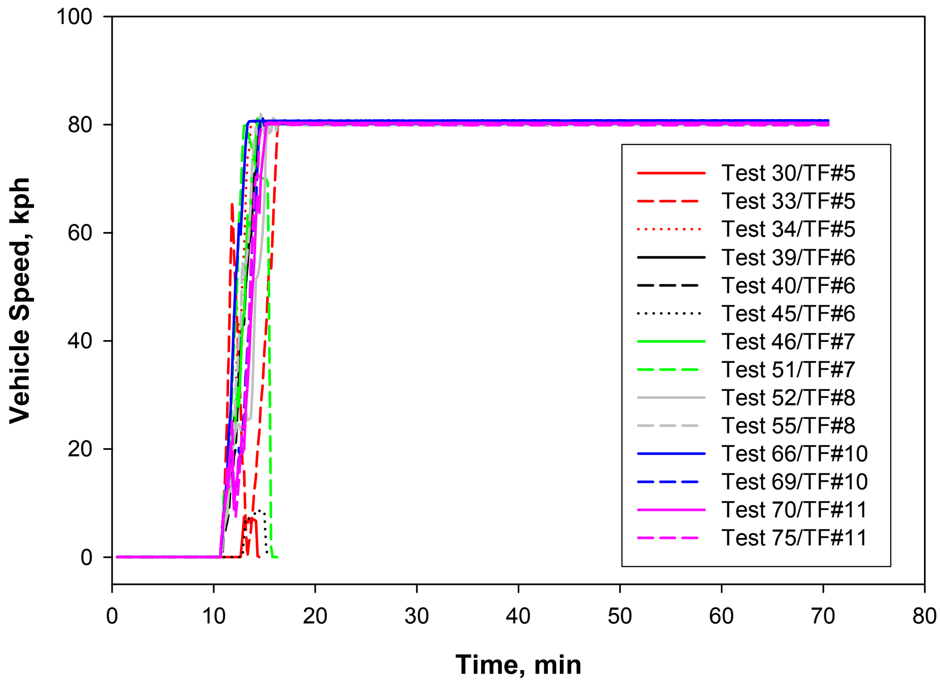


Figure 39. Vehicle speed traces for B20 blends tested in Truck D.