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Diesel Fuel Low Temperature Operability Guide

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DIESEL FUEL LOW TEMPERATURE OPERABILITY GUIDE

INTRODUCTION

Diesel Fuel Low Temperature operability is about changes in fuel performance parameters that prevent the required fuel flow for engine operation in cold environments. It is among the top concerns related to diesel fuel among fuel suppliers, engine and vehicle manufacturers, as well as the vehicle/equipment users.

Users in warm regions are not affected. Other regions are faced with this issue for only two or three months of the year. However, in colder regions, equipment operators can be affected for a significant portion of the year. Most think of Low Temperature issues only as wax precipitation at lower temperatures. Other factors such as freezing water in the fuel should also be considered.

Specifying low temperature operability temperatures for different regions can be developed in many ways. None of them will completely eliminate the possibility of vehicle failures since they are based on historical temperature data and because there is always the chance that abnormally low temperatures will occur. Operability temperature recommendations are usually set to minimize low temperature issues, not to eliminate them. Users need to be aware of potential fuel concerns and be prepared to take additional steps when temperatures are lower than historic values.

Diesel fuel composition plays an extremely important role in low temperature operability. The blending of waxy streams or biodiesel on their own may result in low temperature issues or may cause additives not to work as designed. The presence of impurities or improper additization may result in interactions that can cause filter plugging or other low temperature issues.

The purpose of this document is to discuss these topics and to provide a guide for the end user, fuel producer/distributor, and vehicle/equipment manufacturer, and to help them understand the challenges of operating diesel engines in a low temperature environment. With proper knowledge of the issues, low temperature operability problems can be minimized. Many of the low temperature handling and performance principles discussed in this document may also apply to other distillate fuel applications such as burner, gas turbine, and marine fuels.

Changes in new engine fuel systems and fuels may affect the approach in the future. Therefore, this document will be updated periodically.

DIESEL FUEL

Fuel used in a compression-ignition engine is typically called diesel fuel. Diesel fuel is not a single molecule but a blend of many components. There are a number of fuel specifications in various regions of the world. Many have requirements or guidelines for low temperature operations but they are not universally the same. One such fuel specification used in the U.S., ASTM Specification D975, was originally approved in 1948 when the diesel engine and its fuel had been in use for many years. At that time diesel fuel oil was produced from petroleum crude oil using fairly simple refining techniques, usually just atmospheric distillation of the required cut. As properties required by engines and the diesel fuel distribution system changed, D975 also changed. But the underlying technology of fuel production remained; it was produced from petroleum crude oil using common refining techniques. These techniques have expanded to include hydrotreating, hydrocracking, non-hydrogen cracking processes and several chemical processes for removing deleterious materials. This type of diesel fuel is sometimes called conventional diesel fuel.

In recent years, fuels made from raw materials other than petroleum crude oil have been developed. These fuels are frequently produced from those other raw materials via processes that are substantially different from petroleum refining processes. These types of fuels are identified by a variety of designations such as biodiesel, renewable diesel, hydrotreated vegetable oil (HVO), gas-to-liquids (GTL), biomass-to-liquids (BTL), water-diesel emulsions, and alcohol-diesel emulsions. They all have one thing in common; they are intended to power compression-ignition (or diesel) engines and as such in a general sense they all are diesel fuel. However, in some cases these fuels do not comply with the regional required specification such as ASTM D975 and/or may not be accepted by diesel engine and equipment manufacturers or suitable for a universal trouble-free operation in compression-ignition engines. In each case stakeholders through their standard setting organization (such as CEN, CGSB or ASTM) process will evaluate and determine if a newly introduced form of diesel fuel is acceptable, meets the requirements, requires a new standard specification, or if is not acceptable for use in a compression-ignition engine. So just because something is generally described as "diesel fuel," it does not mean that it works well in all diesel engines and diesel powered equipment.

FUEL CHARACTERIZATION

The introduction of non-conventional streams such as biodiesel, GTL, and BTL into diesel fuel blends has complicated fuel characterization. Biodiesel introduces narrow distillation cuts of mono-methyl fatty acid esters as well as impurities that come along with them. GTL and BTL are narrow distillation cuts produced from raw material sources like natural gas and biomass, respectively, which can contain high concentrations of specific n-paraffins. All of these blend streams can have a significant impact on diesel fuel characterization and low temperature properties. However, the basics for fuel characterization are the same as for conventional diesel fuel analysis.

There are several analytical measurements that can provide information for estimating a diesel fuel's low temperature performance. Distillation, Cloud Point (CP), Pour Point (PP), viscosity, n-paraffin content and distribution are all very useful diesel fuel characteristics from a low

temperature standpoint. Table 2 provides examples of these properties for a variety of S15 (fuels that contain less than 15 mg/kg of sulfur) Grades No. 1-D and No. 2-D diesel fuels from the USA.

Distillation: A diesel fuel has an initial boiling point (IBP) and a final boiling point (FBP). The most common method for measuring the distillation is the ASTM D86 [1] test method, but there are several other distillation methods that can be used to collect similar information. The low and high temperature limits of the distillation and the breadth of the distillation indicates the kinds of n-paraffins present. In general, with a higher FBP there will be larger n-paraffin molecules present which can translate into a higher CP fuel. The wider or broader the difference between IBP and FBP is, the greater the tendency to naturally produce smaller crystals which are beneficial to lower temperature operability. Broader distillations are generally more responsive to cold flow additives which will be discussed further in a later section of this guide.

The addition of biodiesel at up to 20% in a fuel blend still permits the use of ASTM D86 distillation to establish limits for most specifications. The addition of biodiesel, GTL, and BTL may change the shape of the distillation curve causing changes to the (FBP-90) and/or (90-20) differences but the overall distillation range will be similar.

Cloud Point (CP): Defined as the first appearance of haze in a diesel fuel due to wax crystal formation under prescribed cooling conditions. CP is measured manually according to ASTM Method D2500 [2], but there are a number of automated methods that correlate to D2500. Originally this test method was part of ASTM D97 [3], which was then called Standard Test Method for Cloud and Pour Point. The differences in the CP and PP test methods, as well as the importance of each of the tests, resulted in their separation into individual test methods in the 1950s.

Most specifications include a seasonal CP requirement or some reference to it because CP indicates the earliest appearance of wax precipitation that might result in plugging of fuel filters or fuel lines. CP is considered to be the most conservative estimate of low temperature vehicle operability. ASTM D2500 is a very fast cool, manual test and as such does not simulate real world cooling conditions. In the manual method, the fuel sample is cooled in progressively colder baths and examined every 1°C as the fuel sample cools. A diagram of the manual CP apparatus may be found in Figure 1. (The ASTM D2500 CP test method has a precision of 2°C repeatability and 4°C reproducibility.)



NOTE 1-All dimensions are in milllimetres.

Figure 1 - Schematic of Manual CP Apparatus [2]

In order to increase precision in the CP determination, several automated analytical methods were developed. The automatic CP determination methods specify different cooling rates – some require stepped cooling while others require linear/constant cooling rates. The precision of these methods vary. However, in every case, the automatic instruments provide greater precision than the manual method. All automatic test methods have a relative bias versus the manual method (positive or negative bias). In some methods this bias is very small and statistically insignificant, while in others the bias is statistically significant but too small to be considered of any practical significance, whilst for some methods the bias must be added / subtracted in order to report the result as equivalent to the manual method.

ASTM D5773 [4] is the most prevalent automated CP test method. The method operates by cooling a diesel sample at a constant rate and monitoring the intensity of light transmitted through it. When the first wax crystals have formed, the light transmittance decreases. The corresponding temperature is the CP. The repeatability of test method ASTM D5773 is 1.3°C for distillates, and reproducibility is 2.5°C [4].

In very low CP, No. 1-D diesel fuels that are sharply fractionated and contain low percentages of very waxy components (such as streams from a catalytic dewaxing unit), the CP test may indicate a colder vehicle minimum operability temperature than can actually be achieved. In these rare situations the low temperature flow test (LTFT, described later in the document) may be a better predictor of actual vehicle operability performance [5]. The CP test method cooling rate is so fast (1°C to 5°C/minute) that it may overshoot the wax appearance temperature observed under real world slow cool conditions (1°C to 8°C/hour). Therefore, it potentially can mislead the end user into thinking the vehicle operability limit is lower than it actually is. This phenomenon is normally not a problem in fuels with CPs above -35°C.

Distillation combined with CP can tell a lot about a fuel's responsiveness to cold flow additives. For example, between two fuels with similar distillations but having different CPs, the higher CP fuel is waxier and the lower CP fuel should be more responsive to cold flow additives. As with many standardized tests, CP is designed to provide the best precision by controlling as many variables as possible, but the downside to this is that the controls may actually disturb or remove conditions or materials that may be important to low temperature performance. In the case of CP test method there is a step in the test procedure to filter and remove water and particulates, parameters that if present, may actually contribute to poor low temperature operability. Other tests for water and particulate analysis may be necessary to provide relevant information on low temperature operability and handling.

Biodiesel addition to fuel blends typically increases the blend CP. Vehicle testing to date [6] has shown that CP still provides a conservative estimate of low temperature operability limits for blends containing up to 20% biodiesel. GTL and BTL can also increase the CP of the fuel depending on the degree of isomerization of the stream. No isomerization or low levels of isomerization will result in a narrow cut of *n*-paraffins in the GTL and BTL and will show greater increases in CP and be more difficult to treat with cold flow improvers.

Pour Point (PP): The PP test, ASTM D97, like CP is a manual, fast cool test method. The ASTM D97 PP test method has a precision of 3°C repeatability and 9°C reproducibility [3]. PP is defined as 3°C above the fuel specimen gel temperature under cooling conditions specified in ASTM D97. A schematic of the ASTM D97 test equipment is shown in Figure 2 below.



Note-Dimensions are in millimetres (not to scale).

Figure 2 - Schematic of Manual PP Apparatus [3]

The PP measurement for a diesel fuel sample is a directional indicator of the diesel fuel's low temperature handling (bulk movement and storage of fuel) performance. Unfortunately, PP is often incorrectly mentioned in the same discussion with CP, cold filter plugging point (CFPP) and LTFT which are measures of vehicle low temperature operability. PP does not correlate to low temperature operability or to fuel filterability at low temperatures. Under the right circumstances PP can explain failures for diesel fuel to move through the vehicles fuel delivery system. The fuel lines and filter on a vehicle fuel delivery system are much smaller than the fuel tank and they can cool faster to below the fuel's PP during overnight cooling, while the fuel in the fuel tank does not. Therefore, it is possible for the fuel to gel in fuel lines or filter yet still to be fluid in the fuel tank.

A fuel with a -30°C PP would be expected to have better low temperature handling than a -15°C PP fuel, but it would be unrealistic to assume that either fuel would remain liquid if held at their respective PP temperature for extended periods. However, PP can also provide useful information when used in combination with CP.

If you know a fuel has no cold flow additive in it and the untreated CP - PP delta is 10°C or more, then the fuel is probably going to be fairly responsive to cold flow additives. If the

difference between CP and PP is less than 5°C, then the fuel will most likely be more difficult to treat with cold flow additives to improve vehicle operability performance.

It is possible for the PP to be higher than the CP. For example, if a fuel sample has a CP of -2°C and the temperature at which the fuel sample fails to pour is -3°C then the PP for that fuel would be reported by the PP test method as 3°C above that temperature or 0°C. In cases like this the fuel is generally extremely unresponsive to cold flow improvers (CFI) designed for better low temperature vehicle operability and the distillation range is usually quite narrow. However, the fuel may still be reasonably responsive to pour point depressants (PPD). To reduce PP significantly one only needs to change the shape of the wax crystals while to improve operability the size (smaller being better) and the shape of the wax crystals also need to be modified.

In order to increase precision in the PP determination, several automated analytical methods were developed for PP determination. ASTM D5949 [7] and ASTM D5950 [8] are the two most prevalent PP test methods. In these methods, the fuel sample is cooled according to a prescribed cooling rate and surface movement is measured by various automated methods.

n-Paraffins: n-Paraffin analysis is done by gas chromatography (GC) where the specific nparaffins are identified thermally and their amounts are determined from calibration with pure compounds. This analysis is particularly useful when fuels do not respond to cold flow additives as expected. A very narrow n-paraffin distribution, like a narrow distillation, suggests a propensity to naturally produce larger crystals and thereby poorer response to cold flow improvers.

The n-paraffin distribution in combination with the non-crystalizing liquid portion of the fuel dictates what the CP of the diesel fuel will be. The longer the n-paraffin chain the higher the molecule's melting point and its crystallization temperature, so a C₃₀ n-paraffin will crystalize and come out of solution at a higher temperature than a C₂₇ n-paraffin at the same concentration. The C₂₇ n-paraffin may stay in solution at lower temperatures at the same concentration but at higher concentrations it may come out at the same temperature as a C₃₀ at a lower concentration resulting in two fuels with the same CP but having very different wax crystallization characteristics. (Figure 3) For diesel fuel operability above -40°C, the n-paraffins of C₁₉ and longer chain lengths play the major role in wax crystal formation. (Figure 4) The composition of the liquid or non-crystallizing phase will impact the precipitation of the wax. The non-polar n-paraffins will be less soluble in aromatic or polar liquids, so the wax crystals will precipitate at higher temperatures. In a highly non-polar liquid the n-paraffins will stay in solution longer and crystallize at lower temperatures.



Figure 3: Examples of n-Paraffin Distributions in Seven Diesel Fuels (identified by letter)



Figure 4: n-Paraffin Distributions for C19+ Chain Lengths in Seven Diesel Fuels (identified by letter)

Biodiesel is composed primarily of mono-methyl esters of fatty acids. The saturated forms of these esters make up the waxes produced upon cooling biodiesel. These saturates can be analyzed by GC. The result is a much narrower distribution of wax producing species since they are composed primarily of C_{14} , C_{16} , and C_{18} fatty acid mono-methyl esters. Biodiesel saturates have GC elution times similar to C_{21} n-paraffin. One would see a spike in the chromatogram at about that point if biodiesel were present in a fuel blend.

GTL and BTL as mentioned previously may introduce narrow cuts of n-paraffins depending on the level of isomerization of the stream. Adding 5% or more of a non-isomerized stream may make the fuel blend untreatable by typical cold flow improvers.

Kinematic Viscosity: The Kinematic Viscosity range of diesel fuel is limited to a maximum value in most specifications, so the effect of fuel viscosity on low temperature vehicle operability is

usually not a factor. However, there are occasions when the high end of the fuel viscosity range may contribute to vehicle failures. It is also important to keep in mind that laboratory tests, such as CFPP and LTFT, used to estimate the low temperature operability of a diesel fuel are also limited by viscosity because the chilled test fuel flows through filter screens. In the case where a CFPP or LTFT failure is observed, but the test fuel is clear and bright – with no wax out of solution, high fuel viscosity at the test temperature may be the cause. In this case, the "minimum pass" temperature from the lab test may not accurately estimate actual vehicle operability – since the vehicle's fuel system may not be viscosity limited at the same level as the lab test. This is a more common phenomenon with concentrated fuel additives, where the additive is solid but completely clear 3°C below the Pour Point temperature, than in diesel fuels. Viscosity limits should be evaluated for new fuel types or when new diesel fuel blending streams are introduced to minimize any field problems attributable to high viscosity.

Biodiesel generally has a higher viscosity than conventional diesel fuels, so care must be taken when blending biodiesel with diesel fuel to make sure viscosity limits are not exceeded. GTL and BTL streams have viscosities in the same range as conventional diesel fuel Grades No. 1 and No. 2, so they have less impact on fuel blend viscosities.

LABORATORY FILTERABILITY TESTS FOR ESTIMATING FUEL OPERABILITY LIMITS

It is important to understand that neither CFPP nor LTFT laboratory operability tests were developed to simulate performance in vehicles. Each test evaluates fuels using a consistent time, temperature, and filter to characterize one fuel's performance in comparison to another fuel's performance. The tests correlate rather than simulate low temperature performance in vehicles.

Further to this discussion to simulate is defined as 'to assume or have the appearance or characteristics of' something [9]. The something in this case is the operation of a diesel engine under low temperature conditions. A correlation by definition establishes an orderly connection between two things, such as tests. In this discussion the tests are either CFPP or LTFT and the low temperature performance of a diesel fuel in a diesel vehicle. As you read about the tests and the operation of the fuel delivery system and the engine in more detail, it should become clear that these tests do not replicate any operating conditions of real diesel powered equipment.

Cold Filter Plugging Point (CFPP): Introduced in 1965 in Europe to estimate low temperature operability performance in light duty vehicles when cold flow additives are used. The CFPP (ASTM D6371) [10] is a fast cool test method where a small portion of diesel fuel is rapidly cooled and repeatedly drawn through a 45 μ m opening screen using a 2 kPa vacuum every 1°C as the sample is cooled. The temperature at which the sample fails to be drawn through the screen in 60 seconds or less is designated as the CFPP temperature or the estimated operability limit for that diesel fuel. The CFPP, therefore is a fast test, typically producing a result in 45 minutes or less. A schematic of the ASTM D6371 test equipment is shown in Figure 5 below.



NOTE 1—All dimensions are in millimetres, and the comma (,) is used as the decimal point.

Figure 5 - Schematic of a CFPP Apparatus [10]

A field trial in terms of cold operability failure temperatures was conducted in Sweden in winter 1964/65 with 15 different diesel vehicles and 25 diesel fuels. [11] The fuels differed in their CPs - some of the fuels contained cold flow improver additives, while others did not. After the field testing was finished a new laboratory test method was developed in consideration of the following requirements:

- Filtration testing to reflect the field failure mechanism
- Rapid test to satisfy needs of refinery laboratories
- Use of standard test facilities (cooling bath, glassware)
- Test parameter selection to provide protection for the most sensitive vehicles.

This work resulted in the development of the CFPP test apparatus and procedure. The fuel test results generated with the new CFPP method showed a good correlation to the cold operability data generated in the 1964/65 field trial (Figure 6).



Figure 6 - Correlation of the Swedish field operability data 1964/65 vs. CFPP [12]

CFPP is used throughout the world today where cold flow additives are in use. The precision for CFPP is 1°C repeatability and the reproducibility is an equation, 0.102 (25 - X)°C, where X is the average of two results from different instruments or different laboratories [10]. The reproducibility increases as the temperature decreases. The Table below provides examples of the reproducibility one can expect from CFPP.

Table 1

Repeatability, °C	Average Temp., °C	Reproducibility, °C
1	-10	3.57
1	-20	4.59
1	-30	5.61
1	-35	6.12

Precision for the CFPP Test (ASTM D6371-2005(2010))

In North America where the diesel fuel market is predominantly heavy duty (HD) trucks, CFPP has found limited and successful use in the United States, but it is not an accepted test method in Canada. Some of the features of the CFPP test that have been criticized include disturbing of the fuel every °C on cooling preventing the formation of a gel matrix which could impact low temperature performance. The cooling rate is also a deviation from vehicle operation because it is very fast, roughly 40 times as fast as real world cooling rates which impacts wax crystal size and shape and thereby real-world performance. The screen used in CFPP testing has been a point of contention due to its uniform openings and pore size (45 μ m) compared to the paper filters in vehicles which are the tortuous path type and have a porosity in the range of 20 μ m to 2 μ m. However, the developers of the CFPP test remind us that this is a correlation to vehicle performance and the above factors were taken into account through test modifications to correlate to the extensive vehicle database. Even with the above criticism regarding the CFPP test it is still the most widely used filterability test method for estimating vehicle operability in LD vehicles. A big advantage for CFPP is that it is a very fast test providing a result in 45 minutes or less.

Using CFPP results that are more than 10°C below CP has shown to reduce the test method's capability of estimating light-duty (LD) vehicle low temperature performance. (Figure 7)

Operability vs. CFPP European Data Supporting CP-CFPP <10

European testing has shown that CFPP best predicts performance if the Cloud-CFPP delta is $\leq 10^{\circ}C(18^{\circ}F)$



Numbers represent CP-CFPP°C delta

Figure 7: European Vehicle Data Comparing CFPP to Lowest Vehicle Operability Temperature [13]

Biodiesel like many other heavier or waxier streams may actually improve CFPP results with up to about 5% by volume in a fuel blend, but greater volumes have been found to reverse this performance. The increase or decrease in CFPP response may or may not correlate to vehicle low temperature operability. GTL and BTL, depending on the range and amounts of n-paraffins, will impact the CFPP for the base fuel and the ability to treat with cold flow improvers. Narrow distributions with high concentrations of n-paraffins will decrease the base fuel CP-CFPP delta and make the fuel more difficult to treat.

Low Temperature Flow Test (LTFT): The LTFT, ASTM D4539 [14], was developed and introduced in the 1980s to provide better protection for the heavy-duty (HD) truck designs found in North America. In the development of the LTFT, HD vehicle performance was the target and specifically the most severe fuel delivery design at the time of the LTFT development. Statistically this is an important factor since the test was developed fitting the results to the most severe vehicle fuel delivery design. By definition the less severe fuel delivery systems would always be overprotected. The CFPP development approach was to statistically fit all the vehicle data.

The LTFT is similar to the CFPP, however, a larger volume of fuel is slowly cooled (1°C/hr.) and drawn through a less porous metal screen (17 μ m), only once under a higher vacuum (20 kPa).

A fresh fuel sample is filtered at every 1°C cooled until a sample fails to filter within 60 seconds. The LTFT limit is defined by the lowest passing temperature.



A schematic of the ASTM D4539 test equipment is shown in Figure 8 below.

Figure 8 - Schematic of Manual LTFT Apparatus [14]

Like the CFPP test, the LTFT is a correlation to low temperature vehicle performance. The LTFT test better simulates the fuel system parameters with respect to HD trucks during overnight cooling at winter ambient temperatures. For example, the LTFT uses a slower cooling rate (1°C/hr.), which is more like the cooling rate of the large volume fuel tanks found on HD trucks. The LTFT uses a less porous (17 μ m) tortuous path screen and a larger volume of fuel per test. The LTFT only tests each sample once, and incorporates a stirring step just before filtering. The stirring step was incorporated during development because it significantly improved precision. The LTFT has a precision of 2°C repeatability and 4°C reproducibility.

A disadvantage of the LTFT is that it takes long to run, due to the slow cooling rate, making it an overnight test.

Biodiesel blending at up to 20% has shown LTFT still to be a good estimate of HD low temperature vehicle operability for diesel fuel without cold flow improvers [6]. Biodiesel generally makes the fuel more difficult to treat with cold flow improvers or may require different cold flow improvers. The impact of GTL and BTL is the same as described for CFPP above.

A 2005 CRC program using North American Light Duty Vehicles found 4 of the 5 vehicles tested, with several fuels -- both cold flow additive improved and untreated, were adequately protected by CFPP; however, one vehicle was not [15]. The one vehicle exception had a fuel delivery system design similar to typical North American Heavy Duty trucks.

Fuel Characterization Examples: Table 2 provides examples of fuel properties for a number of typical Grade No. 1-D and Grade No. 2-D S15 Diesel Fuels from the USA including B5, B11 and B20.

Table 2	2
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							Fuel					
		Test		_	-	- ()	_	_				
		Method	A	В	C	D(B20)	E	F	G(B5)	H(B11)	1	J
AutoClou	ıd,°C	D5772	-12.3	-19.5	-8.4	-10.9	-10	-45.8	-8.2	-11.5	-46	-6.2
AutoPou	r,°C	D5950	-18	-33	-10	-33	-12	<-60	-24	-48	<-45	-9
CFPP, °C		D6371	-17	-18	-15	-16	-18		-13	-20	<-45	-6
Density,	15°C, g/ml	D4052	0.8370	0.8346	0.8292	0.8340	0.8254	0.8262	0.8411	0.8490	0.8129	0.8247
S, wt%		D5453	<0.001	<0.001	<0.001	<0.001	<0.001	0.00005	<0.001	<0.001	<0.001	<0.001
KV@40°0	C, cSt	D445	2.43	1.74	2.3	2.42	2.1	1.761	3.09	2.56	1.56	3.75
D86, °C		D86										
	IBP		174	170	177	170	175	158	196	174	170	133
	T20		232	193	220	209	212	188	235	220	196	272
	Т50		261	220	254	256	244	221	271	264	218	294
	Т90		312	322	321	311	315	283	326	327	259	334
	FBP		352	356	349	339	351	315	349	347	283	350
	(T90-T20)		80	129	101	102	103	96	101	107	63	62
	(FBP-T90)		40	34	28	28	36	32	23	20	24	16

Examples of USA S-15 Grades No. 1-D and No. 2-D Diesel Fuels

DIESEL FUEL BLENDING

As stated in the Introduction diesel fuel was historically made as a single stream often referred to as a straight run distillation cut. Originally, the best distillation range for diesel fuel was 150° to 400°C, which still generally holds for today's fuel blends. In ASTM terms, a Grade No. 2-D diesel fuel would have higher CP, 90% distillation temperature, and viscosity than a Grade No. 1-D. Further discussion is provided in the specifications section.

In today's more complex production and supply schemes, the final product is a blend of a number of streams and components. Optimization of refinery yields, reduced cost and improved physical and chemical properties are some of the reasons. Refinery units were developed to convert the heavy bottoms from straight and vacuum distillation to lighter streams that can be blended into gasoline and the middle distillate fuels (jet, diesel fuel, fuel oil, kerosene, etc.). These processes had significant benefits in yield to the point where on a volume basis a barrel of crude becomes 1.3 barrels or more of finished products.

Some blend streams are also made from non-crude oil based sources. Biodiesel, natural gas-toliquid, and biomass-to-liquid are representative examples. The streams, like biodiesel, are often blended at terminals because they are not manufactured at the refinery, often are not allowed to be shipped on multi-product pipelines because of potential for water contamination or contamination of jet fuel with biodiesel, and it sometimes makes more sense to use local biodiesel production and such blending methods may also be legally required.

Streams are combined at the refinery and at terminals and sometimes at both locations to meet the low temperature requirements of the region where they are sold. Diesel fuel may also be additized to meet low temperature operability requirements of the region in which it is sold. With so many streams there are numerous ways in which to blend them to meet the low temperature requirements. This can result in improved or directionally poorer low temperature performance with regard to response to cold flow additives. The potential for variability in fuel composition can make it more difficult for the end user to choose the correct fuel for their needs. Other specifications not directly related to low temperature performance restrict the way diesel fuels can be blended and may improve the uniformity of fuels produced in any region. The volume blended and the wax characteristics of the streams may also impact responsiveness to cold flow additives and ultimately low temperature performance in handling and in vehicles. Minimum cetane number, specific distillation maximums, viscosity minimums and maximums, density limits, aromatic content limits, storage stability and cleanliness limits, as well as government instituted limits for sulfur and blending of biodiesel may all indirectly impact low temperature operability.

Biodiesel presents a unique challenge for meeting low temperature requirements because of solubility limitations. Biodiesel used in blends with diesel fuel are typically in the -5°C to +15°C CP range depending on the feedstock. There are lower CP biodiesels as indicated in Table 3, but the lower CP biodiesels are less commercially available or are not economically viable. As a point of reference the CPs of commonly used biodiesels (soy, canola, palm and tallow) fall in the range of CPs for heavy refinery streams normally used in diesel fuel blending. The CP of finished winter diesel fuel varies depending on the region of the world but most locations

require fuels to operate much lower than -5°C in winter. To offset the increase in CP caused by biodiesel and other heavy or higher CP streams, the refiner or terminal operator can add lower CP fuels such as Grade No. 1-D diesel fuel or kerosene or jet to the blend. These streams are all similar and in some cases identical and are usually lower polarity streams with less aromatic content. Unfortunately, biodiesel is more compatible with polar or aromatic streams. In Minnesota, which has a biodiesel mandate, a waiver was granted allowing users of straight Grade No. 1-D to add no biodiesel in the winter because biodiesel has poor solubility in Grade No. 1-D at low temperatures.

Table 3

Biodiesel(B100)	Cloud Point, °C	Biodiesel(B100)	Cloud Point, °C
Algae 1	-5.2	Hepar, Low IV	6.7
Algae 2	3.9	Jatropha	2.7
Babassu	4.0	Lesquerella fendleri	-11.6
Beef Tallow	16.0	Linseed	-3.8
Borage	-1.3	Moringa oleifera	13.3
Camelina	1.5	Mustard	3.2
Canola	-3.3	Neem	14.4
Castor	-13.4	Palm	13.0
Choice White Grease	7.0	Perilla Seed	-8.5
Coconut	0.0	Poultry Fat	6.1
Coffee	0.2	Rice Bran	0.3
Corn, Distiller's	-2.8	Soybean	0.9
Evening Primrose	-7.5	Sunflower	3.4
Fish	3.2	Tung	-10.0
Нетр	-1.3	Used Cooking Oil	2.4
Hepar, High IV	16.0	Yellow Grease	6.0

Typical B100 Biodiesel Cloud Points [16]

This solubility difference causes a low temperature issue when higher levels of biodiesel are desired or required by law since the normal procedure to lower a diesel fuel CP is to blend in more Grade No. 1-D. However, in this case the more Grade No. 1-D fraction reduces the soluble content of biodiesel at low temperatures. The use of cold flow additives can be beneficial in these cases by reducing the amount of Grade No. 1-D needed.

Biodiesel, because it is usually blended at fuel terminals, may present blending problems especially at lower temperatures and the reader is recommended to examine the studies on biodiesel blending found on the National Biodiesel Board's website [17].

The other approach that some legislators have taken to maximize biodiesel and other renewable blending stream usage is to require the diesel supplier to volume average a specific renewable content for the year and allow them to choose how much biodiesel to blend as long as at the end of the year they have met the volume average requirement. In Canada which has very low temperatures in the winter months, industry practice is to blend very little or no

biodiesel into the diesel fuel during the colder months and increase the level of biodiesel used in the warmer months.

Table 4 and Figure 9 describes examples of blending streams that can be combined to produce a finished diesel fuel. The stream's general impact on the CP of the diesel fuel blend is described, however, this response can vary significantly depending on catalysts and distillation cuts used by specific refineries. Biodiesel and other renewable streams can also vary significantly, so the information in Table 4 should be taken as general trends.

Refineries often have unique names for the streams they produce, but some of the more common names and abbreviation are also mentioned in Table 4.

Table 4

LAGO	Light Atmospheric Gas Oil	CP neutral – generally a base component in the blend to which higher and lower CP streams are added to meet volume demand
LGO	Light Gas Oil	CP neutral – similar or identical to LAGO but may come from different processing
HAGO	Heavy Atmospheric Gas Oil	CP个 at even low percentages
HGO	Heavy Gas Oil	CP个 at even low percentages – similar to HAGO but may be from different processes
LCO	Light Cycle Oil	CP neutral unless at very high levels – narrow wax distribution
LVGO	Light Vacuum Gas Oil	CP个 at even low percentages
VGO	Vacuum Gas Oil or Vis-breaker Gas Oil	CP个 at even low percentages
KERO	Kerosene	CP↓ narrow wax distribution but short n- paraffins
HCN	Heavy Cat Naphtha	$CP \downarrow$ narrow wax distribution but low levels of short n-paraffins
HNN	Heavy Natural Naphtha	CP ↓ narrow wax distribution but short n- paraffins
CLHO or CCLGO	Cat Light Heating Oil or Cat Cracker Light Gas Oil	CP neutral unless at very high levels – narrow wax distribution

Common Diesel Fuel Blend Streams and their impact on CP

FCCU HGO or CCHGO	Fluid Cat Cracker Unit Heavy Gas Oil or Cat Cracker Heavy Gas Oil	CP个 at even low percentages – similar to HAGO but from the cat cracker
DHT Rundown or Product	The stream from a Distillate Hydrotreater	Increases the CP of the stream being treated
HCHGO	Hydrocracked Heavy Gas Oil	CP↑ at even low percentages – similar to HAGO but from hydrocracking
HCLGO	Hydrocracked Light Gas Oil	CP neutral – similar to LAGO but from hydrocracking
Biodiesel or FAAE	Fatty Acid Alkyl Ester	CP个 similar to HAGO but solubility in blend is different - Fatty Acid Methyl Ester is the more common form
HVO	Hydrotreated Vegetable Oil	CP↑ if non-isomerized; CP↓ if severely isomerized; or anywhere in between



Figure 9: n-Paraffin distributions for some common diesel fuel blending streams [12]

FUEL DISTRIBUTION

There are many varieties and configurations of fuel distribution systems, therefore, the ability to have fuel flow in cold temperatures does not follow a single and simple guideline. Fuel distribution can include movements from refinery to terminal, terminal to pipeline, pipeline to terminal, terminal to delivery truck, and tankage to service station pump (Figure 10). There are also specialized applications such as transfer from tankage to railroad engine fuel tanks.

Fuel distribution covers very large systems moving thousands of barrels of fuel daily to very small systems containing just a few hundred gallons. Some systems are very sophisticated using computer controls. Others are very simple and rudimentary where they fill five gallon containers from a spigot. Some systems have filtration to remove solid particles and some do not.



Figure 10: Examples of Fuel Distribution

If the ambient temperature never goes below the CP of the fuels being distributed in that region, no special low temperature requirements are necessary.

Bulk Storage. In general, very large storage tanks, above 50,000 barrels, (>2 million gallons) don't have problems with low temperature. Their size and the contained fuel's thermal mass help to prevent cooling to temperatures below the CP of typical diesel fuel. It would be very unusual for a stored fuel in one of these large tanks to become colder than 5°C, except in the

coldest locations. The CPs of fuels held in these tanks would generally be lower than 0°C. In extended periods of extreme cold it is possible that some wax could form along the inside walls of these large tanks if the cloud point of the fuel is warmer than the ambient temperature but the bulk of the fuel in the tank would remain clear of wax. The outside surface of the tank would also be the first to warm up when exposed to sunlight or if the air temperature rises, providing a heat source to melt any wax that may have adhered to the inner surfaces of the tank. Terminal and refinery tanks usually have a rapid turnover rate, so this is a further aid in preventing the fuels from dropping below their CPs.

Smaller tanks, defined as containing less than 30,000 gallons are different. Because of their much smaller fuel thermal mass, they have the potential to produce a lot of wax through the normal daily atmospheric cooling and heating cycle that can restrict fuel from flowing through lines and filters in the system. There are several options to prevent formation of wax in a tank. Insulation, with or without heaters can sometimes be used to avoid wax formation. This approach may not be feasible economically and may also be impractical. Some operators install a pump to circulate the fuel continuously to minimize wax formation. Burying tanks is the typical design for most retail stations. It has the advantage of keeping the fuel and the lines from the tank to the pump warm (~10°C). However, there are some fueling stations, farming fuel delivery systems, heating oil units, and many railroad fueling locations that have above ground tanks. These facilities would be best served by proper blending of fuels and use of additives to yield CPs below the expected low temperatures for that region.

Fuel Lines. Pipes and tubing through which fuel flows are more sensitive to low temperature ambient conditions than storage tanks. Their smaller size and reduced fuel thermal mass allows them to cool faster. Even large diameter pipes as used in refineries and pipelines will cool quickly raising the potential for wax to form during cold periods. Continuous flow usually prevents any fuel gelling, but if the system experiences significant down time, fuel may cool below its CP. Insulating and heat tracing lines can work very well for refineries and terminals. That approach is less viable for very long pipe runs. In these cases, some fuel suppliers back fill the pipe with Grade No. 1-D (low CP) fuel to prevent gelling during overnight hours or extended idle periods.

Insulating and heat tracing is very effective for smaller systems with a few feet of line in the ¼ to 1" diameter range. These systems are relatively inexpensive and don't require much power to operate. Heat tracing and insulation may also be considered for above ground service stations or other similar systems where tanks and lines are exposed to the elements in colder climates. Sometimes draining the lines during idle periods will prevent gelling of the fuel in the line, but this would only be recommended for systems where small volumes (<5 gallons) are involved. Retail stations with underground storage tanks have the advantage that most of the line to the pump is underground, adequately insulated, and warmed by the earth so minimizing any wax formation. However, those retail stations and small terminals with above ground storage would benefit from insulation/heating the lines or emptying the lines during idle periods. Locations in almost continuous operation may not have any issues with fuel gelling. The constant fuel flow prevents any wax formed to agglomerate and cause the fuel to gel.

However, the fuel flow will flush any wax formed which may clog a filter in the distribution system, the subject of the next section.

Filters: Fuel filters are the most sensitive part of the fuel delivery system when it comes to low temperature operation. Filters capture dispersed solids in the fuel. The source of solids can be dirt, fuel oxidation and degradation, and metal corrosion.

Vehicle fuel delivery systems have become more sophisticated with higher injection pressures and higher temperatures and smaller and more numerous injector spray holes. Therefore, it is more important to remove smaller hard particles. Vehicle manufacturers have added tighter filters and sometimes multiple filters to their on-vehicle fuel delivery systems to prevent damage caused by these solids. Over the years the vehicle filter size has changed from 40 or 50 microns to much smaller porosity filters. Today it would not be unusual to see filter porosities as tight as 2 microns to protect crucial parts of the engine. Even these tighter filters only remove 90-95% of the particles, so equipment manufacturers look to the fuel supply system to remove more particles than ever before.

Fuel filters in the distribution systems for transportation and storage of diesel fuels have also tightened. Unfortunately, like vehicle filters, they do not distinguish between particles that are detrimental to mechanical operation and those that are not. In low temperature operation wax crystals that form will be trapped on or in the filter media just like hard particles. Vehicles can deal with this by circulating warm fuel back to the fuel tank or directly to the filter to melt off any wax that has built up but leaving the insoluble hard particles (rust, fuel sediment, etc.) on the filter. Locating the filter close to the engine also provides warmt to help melt wax. Some systems circulate warm coolant around the filter housing to boost the temperature to melt wax faster. In light-duty vehicles electric fuel filter heaters are often an option to the vehicle purchaser and make good sense for regions where low temperatures (below the diesel fuel CP) are common. Electric fuel filter heaters on heavy-duty vehicles are less common because of the electrical demand. The fuel flow through the filter is also a factor. Higher flow rate through heavy-duty vehicle filters can result in faster buildup of wax.

In fuel transportation and delivery systems filters generally do not have the options and heat sources that are available on LD vehicles. One can heat trace and insulate some filters but then filter replacement may become more difficult. When filters plug due to wax at any of the types of fuel delivery systems already discussed, it is not unusual for an operator to bypass the filter to be able to continue to pump fuel. Bypassing the fuel filter may cause the supplied fuel to contain more sediment/hard particles that could be detrimental to vehicles and pumps. This becomes more critical as engine and injection equipment manufacturers move to finer particle limits.

Ten micron filters at retail service station operations are being considered by the NCWM (National Conference on Weights and Measures) and there is general agreement that this would benefit fuel cleanliness. However, during winter months in cold climates there is concern by some Industry groups that retail dispenser filters may negatively impact operations when ambient operating temperatures fall below fuel CP and/or operations choose to meet low temperature operability requirements through the use of cold flow additives. Service station filters might become blocked as they generally do not have the benefit of the fuel heating or

warm-up devices found in vehicles if the fuels contain waxes that solidify at ambient temperatures and/or contain water that can form ice. However, underground storage tanks have a nominal year-round temperature in the range of 5 to 13°C (41 to 55°F) depending on location, which can assist in keeping the fuel temperature high enough to minimize_slow flow and/or complete filter plugging. There are documented applications of 10-micron waterremoving dispenser filters being used successfully in cold weather environments for diesel retail dispensers when good quality and housekeeping practices are employed.

In the case where filter plugging due to wax occurs, the station or terminal operator has few choices to keep fuel flowing. Removing or bypassing the filter is not recommended because of concerns already discussed, which could result in damage to crucial parts of the engine and the vehicle fuel delivery system. The clogged filter can be replaced with a fresh one and monitored for subsequent re-plugging. If monitoring and frequent filter replacement becomes impractical, heating and/or insulating the filter is a potential solution and new forecourt pumps could be designed to provide the necessary heat and space to prevent wax buildup on the filter. Existing systems with filters located in very tight spaces with very little room for power components and required safety features could require significant retrofits to add heating or insulation.

MECHANISM OF WAX CRYSTAL FORMATION

Base/Untreated Middle Distillate Fuels

Middle distillate fuels (for example: diesel fuel and heating oil) are complex mixtures of hydrocarbon molecules. A large part of the middle distillate composition are n-paraffins commonly referred to as wax. The wax can typically make up about 15% to 30% of the middle distillate fuel. The amount of wax and the size of the wax molecules vary based on the refinery process, the type of petroleum crude used and the level of processing. The carbon number chain length of these paraffin waxes is typically in the range of C_8 to C_{32} . As a middle distillate fuel is cooled, the heavier n-paraffins start to become insoluble and precipitate out of solution from the fuel. The wax that first precipitates becomes the nucleation site for continued wax crystal growth. The temperature at which the first wax crystals can be visually observed is defined as the fuel CP. As cooling continues, the wax crystals grow very quickly with n-paraffins as low as C_{16} involved in the precipitation. These wax crystals can be as large as 1 mm² and are typically in the form of flat, thin rhomboid plates (Figure 11).



Figure 11 - Wax crystals in untreated diesel fuel [12]

The plate-like crystals exhibit strong edge to edge attractive forces between individual crystals which results in the formation of a gel structure where the majority of the fuel remains a liquid, but is trapped inside the interlocking crystal lattice.

As a consequence, a very small amount of precipitated wax can be sufficient to cause gelling/solidification of the fuel (Figure 12). The temperature at which this occurs is often called the fuel's PP. Without the use of external heaters or cold flow additives, at temperatures well above the fuel PP, this crystallization phenomenon can rapidly cause vehicle fuel filters to blind with wax, resulting in fuel starvation to the engine, loss of power and eventually engine stalling.



Figure 12 - Wax Crystal Progression from Cloud Point to Pour Point [18]

In recent years, diesel fuels have become more complex as FAME, HVO, GTL, etc. have been increasingly introduced into diesel blends. HVO and GTL are paraffinic fuels composed of molecules already present in petroleum fuels. FAMEs on the other hand are chemical species that are not present in hydrocarbon fuels. However, like n-paraffins, saturated FAMEs can crystallize upon cooling and form part of the precipitated wax together with n-paraffins.

Crystallization Theory & Model

The crystallization of paraffin waxes from diesel fuels normally results in the formation of large, flat rhomboid-shaped plates Figure 11. Although the overall growth rate of the crystals is slow, the fastest rate occurs at the edge of the crystals where the long axes of the paraffin waxes stack up next to each other perpendicular to the plane of the crystal. A conceptual visualization of paraffin wax association and crystallization is depicted in Figure 13. The result is that wax

crystals grow primarily along the A- and B-axes, developing the flat plates as shown in Figure 14. There is also crystal thickening (C-axis), but this occurs at a much slower rate than at the crystal edges where there is the greatest surface energy.



Figure 13 - Vizualization of Wax Crystal Growth [18]



Figure 14 - Rhombic/Flat Plate Wax Crystals [18], [12]

The Effect of Cold Flow Additives on Wax Crystal Formation

Using the conceptual vizualization that has been presented, it is relatively easy to understand the mechanism by which a cold flow additive (CFA) inhibits wax crystal growth. One side of the CFA molecule resembles the linear paraffin wax molecule. The other side of the CFA molecule contains a blocking group or side-chain that disrupts the regular pattern of crystallization, which inhibits n-paraffin wax association at the edges of the crystal. The result is that the wax, which in the absence of CFA would form a few large plate crystals, is distributed among many smaller crystals. This visualization is depicted in Figure 15.



Figure 15 - Vizualization of CFA Effect on Wax Crystal Formation [18]

CFAs are primarily paraffinic in nature with bulky, blocking groups and have a significant effect on the growth and size of the wax crystals as seen in Figure 16 and Figure 17.



Figure 16 - Chemistry and binding nature of CFAs [12]



Figure 17 - Alternate Visualization of CFA Binding Nature [18]

One of the most commonly used CFAs is ethylene vinyl acetate (EVA) which consists of a long polyethylene backbone acting like the n-paraffin and the vinyl acetate side chains acting to physically block the laying-down of further n-paraffins in that plane (Figure 18).



Figure 18 - EVA Copolymer [12]

CFAs inhibit crystal growth rate on the normally fast growing axes (A & B), effectively increasing growth along the C-axis. This causes the growth of 'tall' needle-like crystals which when compared to the rhombic plates of an untreated fuel are more compact (Figure 19 and Figure 20).



Figure 19 - Wax crystal growth modified by CFA [12]





Untreated Fuel CFA Treated Fuel Figure 20 - Comparison of wax crystals in untreated and treated fuels [12]

Although each individual crystal will have a smaller surface area, the total surface area of all the modified wax crystals combined vastly increases. Rhombic/plate crystals would normally 'pack' closely on a fuel filter surface thus rapidly reducing porosity, however the needle shaped nature of the CFA modified crystals allows them to form a relatively thick porous cake on a filter before it blocks sufficiently to restrict fuel flow. Figure 21 compares fuel filters removed from a vehicle run at low temperature to achieve borderline operability – where the engine is at the point of fuel starvation due to filter waxing. The left filter photo, on untreated fuel, is blinded by an almost invisible layer of wax. The CFA-treated fuel results in a thick, but porous wax cake (seen on the right filter photo) – which allows vehicle operability at a temperature well below the fail-point of the untreated diesel fuel.





Fuel Filter Exposed to Untreated FuelFuel Filter Exposed to CFA Treated FuelNOTE: The wax appears red in color due to the fuel containing red dye.

Figure 21 - Vehicle Fuel Filter Comparison at Blocking Point [12]

The different stages of wax crystal growth, both with and without the use of CFAs, are shown in Figure 22.



Figure 22 - Temperature Dependance of Wax Crystal Growth [18]

Above the CP, most of the n-paraffins (and CFAs) are in solution. As the temperature decreases to the CP, CFA treated fuel contains more nucleators which produce more seed crystals than in untreated fuel. Between the CP and the PP, the fewer nuclei crystals in the untreated fuel grow

into visibly larger crystals, whereas the treated fuel contains many small crystals. The CFA treated fuel also contains growth arrestor which slows growth, alters shape and reduces adhesion. Below the PP, the large crystals in the untreated fuel adhere and form a gel thus resulting in performance failure. Action of the CFAs in the treated fuel maintains crystal separation and allows the PP to be lowered.

The wax formation steps listed above are for diesel fuels that do not contain FAME. The saturated esters present in FAME will behave in a similar fashion to that described. However, trace contaminants that are sometimes present in FAME (e.g. saturated monoglycerides and sterol glycosides) do not behave in this way.

Cold Flow Additive Types

Cold flow additives (CFAs) are used to improve the cold temperature wax formation characteristics of distillate fuels. There are several varieties of CFAs, each designed for a different purpose. They include:

- Cloud Point Depressant (CPD) Used to lower the CP of a fuel.
- Middle Distillate Flow Improver (MDFI) Used to improve vehicle operability, measured in the lab by lowering the CFPP or LTFT minimum pass temperature.
- Pour Point Depressant (PPD) Used to lower the PP of a fuel.
- Wax Anti-Settling Additive (WASA) Used to reduce the rate of wax settling in diesel fuels stored below their CP.
- Wax Anti-Settling Flow Improver (WAFI) Combines MDFI and WASA performance.

The original flow improver was the PPD. They were developed to lower the PP of diesel fuels and heating oils, that is, lower the temperature at which the gel structure forms and stops fuel from flowing. PPDs are often added to improve fuel handling characteristics in fuel logistics. PPDs exhibit limited ability in controlling the size of individual crystals, and at temperatures above the fuel's PP do not always provide a significant improvement in fuel filterability or vehicle operability performance.

Such modification is achieved using a flow improver additive. This type of additive is the most commonly used today and it can readily control the size of individual crystals and effectively improve filterability and vehicle operability. Further reduction of the wax crystal size can also be achieved using WASA as shown in Figure 23.





CFA Treated Fuel WASA Treated Fuel Figure 23 - Wax crystals in CFA vs. WASA treated fuels [12]

The benefits of smaller wax crystals with a more compact shape are twofold:

- Reduced rate of wax settling in fuels stored at temperatures below their CP. Wax settling results in a top layer which is depleted in wax and a bottom layer that is enriched with wax. This settling can obviously lead to inconsistencies in the quality of the fuel that is drawn from the tank, either in vehicles or in distribution systems.
- 2) Improved vehicle operability due to:
 - Improved porosity of the wax cake that builds up on the fuel filter allows the liquid fuel to get through more easily.
 - Small crystals re-dissolve more readily as the liquid fuel begins to warm up when the engine is running and warm fuel is recycled back to the main filter or to the tank. This further helps to extend operability to a lower temperature than achievable with conventional CFA.

CFAs normally include a mixture of polymers which have been matched to the individual characteristics of the fuel to be treated. It is imperative that some of the additive co-crystallizes with the first wax crystals and then the remainder of the additive has to come out of solution as the wax crystals continue to grow. But, on the other-hand, the additive must remain sufficiently soluble so as not to cause any filter blocking problems itself.

It is critical for cold vehicle operability that the fuel temperature during driving rises above its CP. If not, the vehicle will eventually stall due to filter plugging by wax. CFAs reduce wax crystal size and make the wax cake accumulating on the surface of the filter more porous. Filterability is improved as a result, which gives extra time for warming of the fuel system to the point where the wax that has accumulated on the filter dissolves and the bulk fuel in the vehicle tank is raised above its CP.

Measurement of Cold Flow Additive Performance

Several laboratory-based methods already discussed earlier in this guide were developed to allow a faster and more efficient estimation and correlation with low temperature vehicle operability and approximate low temperature handling limits. CP was developed as the most conservative estimate of low temperature operability. There are some cold flow additives that will depress the CP, but in general the depression is modest on the order of 1-3^oC.

For non-additized fuels the CFPP is usually near the CP. For fuels which have been treated with cold flow additives (e.g. CFA and/or WASA) the difference between CP and CFPP can be quite

significant (up to 20°C). If the CFPP is >10°C (18°F) below the CP, the actual performance may not be indicative of the CFPP result.

The LTFT for non-additized fuels is at or slightly lower than the CP. In cold flow additive treated fuels the CP-LTFT difference can also be significant but more modest than for CFPP. A 6-10^oC response in LTFT is considered extremely good. The difference in responsiveness is not surprising because the tests were designed to estimate cold flow performance for different vehicle groups. CFPP was developed for European LD vehicles and LTFT was developed for the more severe North American HD vehicles.

PP which, as already reported, is a general or directional indicator of bulk fuel low temperature handling limits is very responsive to cold flow additives. PP depression of greater than 30°C is not unusual.

VEHICLE FUEL DELIVERY SYSTEM

The vehicle fuel delivery system is designed to deliver precise amount of fuel to the engine via an injector to achieve effective combustion of fuel. The diesel engine operates under very high pressures and injects the fuel directly into the cylinder or a chamber in the cylinder. The most common types of diesel fuel systems used on the vehicle are described as follows [19]:

Distributor Pump or Inline Pump System

The Inline Pump injection system is an example of a purely mechanical system where one pump is serving injectors for several cylinders. These types of fuel systems were the most common types and dominated the diesel engine applications before the advent of common rail systems. In this system multiple cylinders are connected by the rack assembly in which the mechanical system controls the fuel pressure, metered fuel quantity, fuel timing and control pressure waves. There were different variations of this basic system in use. One significant difference between the inline and distributor pump system was the latter is lubricated by the fuel.

Unit Injector System

In the Unit Injector System (UI) the pump and the fuel injector are combined into one unit in a close proximity to the combustion cylinder. This design eliminates or significantly reduces the length of the high pressure fuel lines leading to better control of fuel injections and enables higher injection pressure capabilities (> 2000 bars) for better performance. In the old mechanical UI system the plunger and barrel assembly performed the fuel pumping and metering tasks but with the evolution of electronic control systems the solenoid valve performs the task of metering and timing. Historically, UI systems were favored more for heavy-duty diesel applications than for light-duty diesel applications. In the past decade common rail fuel systems have evolved to match fuel pressures similar to those of UI systems and also offer other benefits which have led to replacements of unit injectors with common rail systems in almost all of present light-duty applications and increasingly in heavy-duty applications as well.

Common Rail Fuel System

Introduction of direct injection passenger car diesel engines in 1980's accelerated the development of common rail fuel systems which offered flexibility in fuel injection systems parameters including variable injection timing, pilot injection, main injection, post injection,

and variable injection pressure [20]. Since the late 1990s common rail fuel systems have evolved to replace the other two common fuel injection systems with considerations for strict future emissions regulations and improved fuel economy. Additionally, it helps to reduce the discomforts attributed to diesel engines, such as noise and driveability. The engine succeeds in providing high torque yet has low noise and improved driveability with use of a common rail system. Moreover, the flexibility of this system not only helps to reduce engine out emissions but allows it to work in conjunction with potential after treatment devices to meet the demanding future emission regulations [21]. Figure 24 shows a schematic of typical common rail fuel system which includes high pressure fuel pump, fuel rails and fuel injectors. The high pressure fuel pump is a positive displacement pump to pressurize the fuel and is generally driven by the engine to compress the fuel to the desired pressure. The pump supplies a constant high pressure of fuel to the rail accumulators on the engine and the injector on each of the engine's cylinders meters the fuel as needed.

The overflow fuel from the pump and the fuel injectors is returned to the tank or in certain systems is circulated to the fuel filter under cold weather conditions to heat the fuel to help prevent wax formation. The Engine Control Module (ECM) is used to exhibit total control over the entire engine control system and the fuel injection system. The ECM monitors the common rail pressure which is regulated by direct control of the fuel pump. Based on the required fuel flow rate the rail pressure is controlled by adding a feedback control of the rail pressure. The required fuel flow rate is determined mainly by engine speed, power requirements, and target rail pressure.



Figure 24. Schematic of Common Rail Fuel System

The function of the common rail is to distribute the fuel and maintain high-pressure to each of the injectors. The rail is a thick walled tube designed to act as an accumulator which is sized optimally to reduce fuel pressure pulsation while maintaining adequate response time of the pressure during transient fuel demand conditions [22]. The volume of the rail varies from only a few cm³ in passenger cars, to as much as 60 cm³ in heavy-duty applications [19]. The common rail injector types are electrically actuated systems designed to deliver desired amounts of fuel to each cylinder in the engine. The common rail injector can deliver fuel regardless of cam timing unlike other injection systems, and allow the possibility of multiple injections. The increases in pressure demands with evolution of common rail fuel systems increases the sensitivity of critical fuel injection system components such as the injector valve seat, guide, and plunger to the contaminants in the fuel. This requires higher efficiency and finer filtration systems to protect the fuel injection equipment for the life of the vehicle.

Many fuel systems have multiple fuel filters, or for larger equipment, banks of fuel filters with each successive filter (or bank) having different ratings. For example, the first filter may be a 10 μ m rating, with the subsequent filter being a 2 μ m rating. In many cases the smaller micron rated filter is located on or very near the engine, and may include a means to utilize engine generated heat to warm the filter. However, the first or primary filter is often placed in a location convenient for service that is more exposed to cold ambient temperatures.

In many cases the engine and/or vehicle or equipment manufacturer does not know if a given product will be operated in a cold ambient environment when they are manufactured but rely on dealers in cold ambient areas to install cold weather related kits to aid operation in their respective area. However, when vehicles or equipment move from region to region over their service life it is important that the customers contact their respective dealer when moving to a cold ambient region to obtain the appropriate alterations.

Fuel Filters

Fuel filters have always been required for diesel engine applications but filtration systems and the filters have changed over time due to changes in fuel system sensitivity to contaminants. It is not uncommon for the latest diesel engine fuel system to incorporate multiple fuel filters either in parallel to increase flow capacity, or in series to allow finer filtration. In the past the finest fuel filter typically had a 10 μ m nominal rating with a Beta ratio of 2 (50% efficient). Today the finest fuel filter may be a 2 μ m filter with a Beta ratio of 200 (99.5% efficient) or better. To better understand the Beta ratio reference Table X8.2 in ASTM D975.

Fuel filters are always associated with solid contaminate (particle) removal but may also be associated with water removal, often referred to as coalescing filters. In some cases, the coalescing filter may be integrated with the primary (first in series) fuel filter, and in other cases the coalescing filter may be a separate device, installed prior to the primary filter.

Cold weather operation provides an additional challenge for fuel filters because components of the fuel that may easily pass through the filter in warmer temperatures, may be removed by the filter in colder temperatures. One of these fuel components are n-paraffins or waxes that start to appear as crystals in the fuel as the fuel cools. The CFPP temperature for a fuel may not ensure operability for all fuel filter systems depending on the fuel filters utilized and the actual fuel temperature when it arrives at the filter.

Fuel blending of Grade No. 2-D diesel fuel with low CP fuels such as Grade No. 1-D diesel is a common method to lower the CP, and typically the fuel's low temperature operability. Cold flow improver additives may also be utilized to improve cold flow characteristics, even though they may not lower the fuel's CP. Cold flow improvers generally work by modifying the size and shape of wax crystals resulting in a more porous wax cake forming on the fuel filter allowing continuous liquid fuel flow. As tank return fuel and/or fuel filter heater warms up, the wax cake will melt and dissolve allowing the whole process to repeat the next time the fuel cools down.

To minimize the potential for wax to prevent fuel flow, and therefore hinder engine operation, several different strategies can be used. Fuel filters can include an electric heater, or circulation of engine coolant to increase the local fuel temperature. Different approaches depend on the fuel system design, the operating conditions, and the fuel being used. In many systems there will be a temperature controlled valve that determines if the injection system return flow is directed to the primary fuel filter or to the fuel tank. Return flow to the primary filter heats the filter and increase fuel temperatures in the injection system to minimize cold operation related fuel delivery concerns. Changing the return flow to the tank increases the bulk fuel temperature in the tank but depending on the return flow rate and fuel tank volume the

temperature rise can be very slow. In other systems the fuel filter can be connected to the engine cooling system such that as the engine coolant warms the fuel filter is also warmed.

Fuel injection system return flow rate is dependent on the fuel system design. In some systems the pump delivery is such that a constant fuel volume in excess of the maximum required is provided with any fuel in excess of the engine requirements returned. In some systems the pump delivery is variable depending on the engine requirements such that there is a minimum amount of fuel returned. Each system design has its respective positive and negative influences on cold weather performance. The constant fuel delivery system recirculates more fuel, which heats fuel in the tank faster, but accumulates wax on the filter at a higher rate. The variable fuel delivery system circulates less fuel, which lowers the rate at which wax accumulates on filters, but will take longer to heat up fuel in the tank.

VEHICLE TESTING

Low temperature vehicle testing is important to establish specification limits and to understand the impact of fuel delivery system design and engine operation effects on low temperature performance. Fuel filter location, filter capacity, fuel tank location and size, and fuel circulation rates are examples of variables that can play a major role in the low temperature operability limit of a vehicle. Vehicle testing is also essential for developing laboratory correlation tests to determine effectiveness of cold flow additives.

Historically, field trials were used to determine low temperature operability limits of vehicles. Many were conducted from 1950 to 1990. The Kapuskasing and Winnipeg field trials in North America and a series of field trials carried out in Sundsvall, Sweden are probably the best known. Field trials have the advantage of using real vehicles under real world conditions. The vehicles were instrumented to measure pressure and fuel temperatures and a specific course was run as dictated by the design of the field trial. A great deal of planning is required to achieve the right fuel in the right vehicle at the right temperature. However, field trials are also at the mercy of Mother Nature, and the weather and temperatures often do not cooperate. Field trials are held in places where cold weather is expected, but sometimes target temperatures are not achieved and the field trial is of limited success.

In the 1970s and 1980s the industry built indoor facilities called All Weather Chassis Dynamometers (AWCD) or Cold Climate Chassis Dynamometers (CCCD) that used vehicles set on rollers that could impart resistance to the wheels, simulating driving on flat surfaces or hills; analogous to running on a treadmill. These facilities allowed the user to target specific temperatures with great accuracy year around. Most systems can achieve low temperatures of -30°C to -40°C. The vehicles are instrumented the same as they are for a field trial and still use drivers. The advantage and disadvantage of these systems are that the cooling/heating rates are very controlled unlike the real world, and the vehicle does not experience rough road surfaces or turns, which can have some impact on low temperature performance.

Testing protocols: A test protocol would be specifically designed around the parameters under evaluation. Most vehicle manufacturers are more concerned with the low temperature lubrication aspects of their vehicles rather than fuel issues because the lubricant will impact the

life of the engine while the fuel is a consumable. They assume that the fuel supplier will provide a fuel that will work in their vehicles at the required temperature.

Unlike spark ignition vehicles, diesel vehicles cover the range from HD freight hauling equipment to LD passenger car designs. The testing protocols for this range of vehicles can be quite different. A HD driver would not get in, start the engine, and immediately accelerate to traffic speed. However, an LD driver might do just that, so these two types of operations and anything in between should be considered in developing a test protocol. Figures 25 and 26 describe general test protocols for HD and LD, respectively. Both protocols have been used in published documents. [15] [6]



Figure 25: Typical HD Low Temperature Testing Protocol



Figure 26: Example LD Low Temperature Testing Protocol

There is no right or wrong temperature profile. If you look at historical overnight temperature for Massena, NY in Figure 27, you will see a large range of variability in cooling/heating rates. It is generally agreed that under real world conditions vehicles cool in the 1°C/hr to 6°C/hr range. The air temperatures may drop or rise much faster but the mass of the vehicle results in a slower cooling rate.

An example of a controlled overnight cool down of HD vehicles is given in Figure 28. The graph shows truck A, C, and D fuel tank cooling profile on an overnight cool down versus the air temperature. One truck was independently cooled in the dynamometer cell and two trucks were in the adjoining cold soak room. The figure shows that truck fuel tanks cool differently under the same cooling conditions. This is attributed to tank location and configuration as well as differences in contact with airflow. The figure also shows that the fuel tank temperature can lag behind the air temperature by several degrees for an extended period and may never reach the air temperature under normal operating conditions.



Figure 27: Example of Variable Temperature Profiles [23]



Figure 28: Example of HD Truck Fuel Tank Cool Down vs. Air Temp. in an AWCD [12]

North America is predominantly a HD market and therefore most of the testing has been conducted using HD vehicles. Europe has a large LD diesel vehicle market, so most of those tests have been conducted using LD vehicles. This has resulted in difficulties regarding lab test correlation. It is Important to note that lab tests for low temperature operability are designed to protect vehicles, not to predict performance. The variation of fuel delivery systems, filter location, filter capacity, filter heaters, etc. makes it nearly impossible to estimate performance for every vehicle.

Whether a vehicle is protected by CFPP or LTFT is dependent on more than just the size and type of the vehicle. Factors mentioned above (filter location and capacity) as well as filter porosity, fuel tank volume, and fuel recirculation rate all play a role in determining which lab test is more likely to estimate vehicle operability correctly. HD vehicles may have 5 to 10 times the volume of fuel onboard compared to a LD vehicle, but the fuel filter may only have a capacity of 2 to 3 times that of a LD vehicle. A very low recirculation rate back to the fuel tank may not warm the stored fuel enough to melt any wax that may have formed. Too high a circulation rate may build up wax too quickly on the filter, not allowing heat from the engine or warm circulating fuel enough time to melt the filter wax.

The North American industry determined that a significant portion of the HD North American vehicle market was more severe from a low temperature operability aspect and demonstrated it in the Kapuskasing field trial where the LTFT lab test better predicted performance of the most severe HD vehicles [24]. That severe HD design was approximately 30% of the market at the time and the LTFT readily protected the remainder of the market, as well. Although the published test data does not support it, when limited to the understood constraints (i.e. not > 10 C below CP), CFPP is also being used as an effective protector of HD cold flow operability.

As engine design and fuel delivery systems evolve low temperature vehicle testing must continue to be studied to make sure the vehicle operator has the best information on low temperature performance and improvements in low temperature operability.

WATER CONTAMINATION EFFECTS

Where and How Does Water Cause Low Temperature Operability Problems?

Another important low temperature operability (LTO) problem in fuel distribution and storage systems is fuel filter blockage by ice crystals. This problem sometimes occurs in fuel dispenser filters at fueling stations. Fuel filter blockage can be caused by either ice crystals or wax crystals, or sometimes both occurring simultaneously. For this reason, it can be difficult to determine whether filter blockage is caused by water contamination or wax crystals from fuel with insufficient cold flow properties.

Fuel flow can be hindered by solid ice blocking the outlet nozzles of storage tanks during cold weather. Above-ground storage tanks are especially vulnerable to this problem. The best preventative measure against ice formation is year-round tank housekeeping maintenance practices to drain water from tank bottoms routinely.

Water contamination can also cause LTO problems in onboard vehicle fuel systems. The most common LTO problems caused by water contamination in vehicle fuel systems are 1) solid ice blocking the fuel pump suction inlet or preliminary filter (often a mesh strainer) in the bottom of the fuel tank, 2) blockage of the main fuel filter by ice crystals, and 3) blockage of the water separator by ice crystals. Once again, fuel filter or water separator blockage can be caused by either ice crystals or wax crystals, or sometimes both occurring simultaneously.

Sources of Water Contamination

Water is typically introduced into fuel in the distribution and storage systems downstream of the refinery. Fuel directly downstream of processing units in a refinery typically contains very little water since the refinery distillation and other processes remover any free water. Refinery process unit are mostly pressurized, so rain water, ground water, or condensate water from humid air cannot contaminate the fuel. However, the further fuel is distributed downstream of the refinery, the more opportunity it has to pick up water or for dissolved water to condense as the fuel cools. Water contamination can occasionally occur in the refinery from leaking heat exchangers or faulty liquid level interface control systems, but maintenance programs usually detect and correct these problems very quickly.

One source of water contamination is condensation inside storage tanks. Storage tanks are vented to the atmosphere. Diurnal temperature fluctuations cause tanks to "breathe" – tanks expel air as the tank headspace warms in the daytime and ingest humid air as the tank headspace cools at night. Humid air is also ingested into tank vents to replace liquid product as it is pumped out of the tank. This air contains moisture which condenses on interior tank walls, runs down into the fuel, and collects on the tank bottom.

Another source of water contamination is precipitation. Storage tanks can be contaminated with rain water through leaky tank valves or hatches, leaky roof seals of floating roof tanks, or rain water or snow blowing through atmospheric vents during a storm.

Water can contaminate fuel during shipment in pipelines, tanker trucks, or barges. Water is often introduced into pipelines from storage tanks supplying the pipeline. Water can collect in low spots in the pipeline and contaminate fuel in significant quantities if a "slug" of water comes out with a product shipment.

Water can collect in storage tanks and contaminate fuel product leaving the tanks. This can occur if a tank is not allowed sufficient time for water to settle out before withdrawing product. If fuel is pumped out of a storage tank at the same time fresh fuel is being pumped into a tank, water bottoms can be stirred up and contaminate the fuel product leaving the tank in significant quantities. This is especially true in cases where tank water bottoms level is allowed to build up, tank water bottoms cannot be drained sufficiently (the tank bottom is not level or the drain nozzle is not on the bottom), tank fuel level is low, or tank product outlet nozzle is on or very near the bottom of the tank [25]. The best way to prevent this source of water contamination is to routinely drain water from the tank. The industry has developed best practices for terminals, etc. for water management. [26]

Similar to storage tanks, fuel shipped in tanker trucks and barges can be contaminated by condensing water from humid air entering atmospheric vents during diurnal breathing or draining of fuel product. Rain water can enter through leaky hatches or valves. In addition, tankers are often cleaned before switching product types using pressurized water or steam

cleaning methods. Residual cleaning water can remain to contaminate subsequent fuel shipments.

In marine applications, water contamination can occur in cases where seawater is used as ballast to displace fuel in fuel tanks. In this case, fuel can be in contact with seawater for extended periods of time and can become saturated with water. As saturated fuel cools, water can separate to form haze and eventually form suspended ice if it gets cold enough. Water contamination of marine fuel tanks has been documented even in cases where seawater was not used as fuel tank ballast. In one case, water contamination of shipboard fuel oil resulted from seawater impinging upon or submerging fuel tank atmospheric vents during operation in rough seas during a storm [27].

Fuel in underground storage tanks at fueling stations can be contaminated by water that condenses from humid air entering atmospheric vents. The nominal temperature for fuel in an underground storage tank is 12.8°C (55°F). A fuel being delivered in the summer could be at a significantly higher temperature and the atmospheric conditions could be 90°F with 90% humidity providing for significant introduction of water into the fuel through condensation.

Underground storage tanks are also susceptible to contamination by ground water or rainwater, especially if the manhole access hatch is under water for extended periods of time during heavy rainfall or local flooding.



Figure 29: Potential sources of water contamination for underground storage tanks (UST) at retail fueling stations

LOW TEMPERATURE SPECIFICATIONS¹

The low temperature requirements contained in various diesel fuel standards and specifications² aim to ensure storage and dispensing of diesel fuels and operation of diesel engines under the climatic conditions prevailing where the products are used.

All standards and specifications make some assertion, be it explicit or implicit, about the extent of protection that the standard or specification will provide, since it would be impossible to protect all users under all extreme temperatures. Standards use historical temperature data to determine the minimum low temperature requirements for a geographical region or a grade of diesel fuel. Some organizations use different calculations and methods to develop minimum requirements and others have agreed upon arbitrary limits, but all estimates are "evergreen" and have procedures in place to change those limits if necessary.

Three main regions will be discussed, since they offer disparate treatments of low temperature requirements. These are Canada, the United States, and Europe. It is expected that most other

¹ The reader is referred to the latest versions of specifications or standards to ensure the most relevant information is accessed.

² Generally "Standards" are legal requirements whilst "Specifications" merely describe product attributes. Although the terms may be used interchangeably in this document, the reader is encouraged to confirm the relevant national, regional, state or local legislation.

standards or specifications will be modelled on one of these three approaches, although the limits may be different. All three of these jurisdictions allow for the inclusion of biodiesel in the form of FAME - at a maximum of 5% in US and Canada and 7% in Europe - in the general diesel specification. In many parts of the world standards for higher blend levels and neat biodiesel are in place or being developed.

Standards and specifications are generally either explicit about operability temperature requirements or guidelines (e.g. Canada versus the United States) or define different product classes through certain product attributes and leave it to the enforcer of the standard to prescribe the classes relevant to different regions and seasons (e.g. Europe).

The low temperature requirements of some North American pipeline operators are also included.

Europe

Diesel fuel quality in the European Union is described by *EN 590 Automotive fuels - Diesel - Requirements and test methods* [28]. This standard defines six temperate climatic zones (Class A through F) and five arctic climatic zones (Class 0 through 4). These are distinguished by differences in CFPP that range from 5°C to -44°C.

Enforcement of the various seasonal grades is left to the member states, such that: "In a National Annex to this European Standard, each country shall detail requirements for a summer and a winter grade and may include (an) intermediate and/or regional grade(s) which shall be justified by national meteorological data" [28]. The basis on which the meteorological data was used to decide which grades are required is not clear, although there is an implicit assumption that the operability temperature is sufficiently described by the CFPP of the fuel.

The member states may also impose different grades at the retail pump and for deliveries from refineries and import terminals.

The European neat biodiesel standard, *EN 14214 Fatty acid methyl esters (FAME) for use in diesel engines and heating applications*, offers some discussion regarding the impact of FAME blending [29]. Firstly, that *"Cold flow additives, when used in FAME, should be specifically matched to the base diesel fuel and FAME quality to ensure correct performance consistent with the requirements set out in this European Standard. The choice could result in incompatibility between the cold flow additives used in the FAME and the diesel fuel."* In addition, restrictions are placed on the FAME *"in order to control maximum content of saturated monoglycerides in the final EN 590 blend to ensure trouble-free operation."*

Canada

The approach in Canada differs from that in ASTM as the low temperature operability requirements are mandatory. Additionally, all blends with biodiesel have separate standards. There are three basic Canadian standards available for diesel fuel - *CAN/CGSB-3.517 Diesel fuel* that does not contain any FAME [30], *CAN/CGSB-3.520 Diesel fuel containing low levels of biodiesel (B1-B5)* [31], and *CAN/CGSB-3.522 Diesel fuel containing biodiesel (B6-B20)* [32]. In all cases, the treatment of low temperature operability requirements is the same and mandatory.

The standards define the 2.5% low-end design temperature as "the 2.5 percentile design value is the temperature at or below which 2.5% of the hourly outside air temperatures are observed to occur for an indicated half month. This data is based upon an analysis of hourly weather readings from weather stations across Canada." and requires that "Low-temperature flow properties of the fuel shall be designed to give satisfactory performance at the temperatures indicated by the 2.5% low-end design temperature data for the period and location of intended use. However, when the 2.5% low-end design temperature is colder than -48°C, a fuel meeting a -48°C operability limit may be provided."

The standards allow two test methods to determine the operability temperature and satisfy the above requirement. These are:

- i. CP using ASTM D 2500, D5771, D5772 or D 5773; or
- ii. LTFT using ASTM D4539 or CAN/CGSB-3.0 No. 140.1. [33]

The current weather data is derived from Environment Canada data collected from 383 weather stations over the period from 1981 to 2010. [34] Notably the CFPP test is not included as ambient temperatures are lower in Canada and the vast majority of diesel fuel is sold into the heavy duty market.

A non-mandatory note states "Low-temperature operability of diesel fuels may be defined by either the cloud point or the low-temperature flow test or some combination thereof. Cloud point defines the temperature at which the smallest observable cluster of hydrocarbon crystals first appears in a fuel upon cooling under prescribed test conditions. It is the most common measure of low-temperature operability. The low-temperature flow test was developed in order to predict low-temperature operability of fuels to which a flow improver has been added. A cloud point test on such additized fuels might not accurately predict the operability limit of the fuel due to the use of these additives. The viscosity of a fuel blend will increase under cold ambient temperatures. In some situations, the fuel's viscosity can be the limiting low temperature operability factor as opposed to wax formation in the fuel."

Further non-mandatory information in the biodiesel blend standards states: "The lowtemperature operability of biodiesel fuel blends can limit availability in some jurisdictions and seasons. Biodiesel components typically have relatively high cloud points (-5 to +15°C) depending upon the source of the feedstock. Precipitation of biodiesel fuel blend components above the cloud point has been reported, and is more likely if the fuel is stored for an extended period at low temperatures, or if it experiences temperatures near the cloud point during storage or handling, and if it contains free water or excessive rust or dirt." In the B6 to B20 Standard it adds "The low temperature operability of biodiesel fuel blends can limit availability in some jurisdictions and seasons. Biodiesel components typically have relatively high cloud points (-5° to +15°C), depending on the source of the feedstock. The addition of up to 20% biodiesel to a diesel fuel can significantly degrade the low temperature operability of the biodiesel fuel blend relative to the low temperature properties of the petroleum diesel fuel component" [32].

The standard for the neat biodiesel (CAN/CGSB-3.524) [35] includes requirements for both the Cold Soak Filtration Test (CSFT, as per ASTM D7501) and a Cold Soak Filter Blocking Tendency

test (CSFBT, as per CAN/CGSB-3.0 No 142.0) [36]. Non-mandatory information states "The CSFT test method determines, by filtration time after a cold soak, the suitability of a biodiesel (B100) to provide adequate low temperature operability performance to at least the cloud point of the finished blend provided it meets all other requirements of this standard and has a cloud point below 20°C (68°F). Minor components of some biodiesel, including saturated monoglycerides, can separate above the cloud point of a biodiesel fuel blend. The CSFBT test quantifies the propensity of these materials to separate from a biodiesel, diluted with isoparaffinic solvent, after a cold soak cycle."

United States

ASTM D975 describes the *Standard Specification for Diesel Fuel Oils* and is applicable to diesel fuel sold in the United States, including FAME up to 5%. [37] ASTM D7467 describes the Specification for Diesel Fuel Oil, Biodiesel (B6 to B20). ASTM D6751 specifies the quality of FAME used in biodiesel blends. It should be noted that adherence to ASTM D975, D7467 and D6751 are not Federal requirements, although the specifications are used by a number of States as well as in commerce.

ASTM D975 and D7467 allow for the measurement of the "*Operability Requirements*" by either one of:

- CP using ASTM D2500; or
- LTFT [38]or CFPP using ASTM D4539 or D6371, respectively.

No limits are provided for these properties and instead the specification states that it is unrealistic to specify low temperature properties that will ensure satisfactory operation at all ambient conditions and CP, LTFT, and CFPP may be used to estimate low temperature operability limits. The 10th percentile minimum air temperatures are provided for U.S. locations in the specification as expected regional monthly low temperatures.

As far as cold flow additives are concerned, the footnote suggests that factors including *equipment design, operating conditions, and the use of flow-improver additives may impact low temperature performance and that* LTFT/CFPP may be useful in estimating low temperature vehicle operability limits.

The specification also indicates that low temperature operability properties should be agreed upon by the fuel supplier and purchaser for the intended use and temperature requirements.

The tenth percentile minimum ambient air temperature referred to is defined in the Appendix as: *"the lowest ambient air temperature which will not go lower on average more than 10 % of the time"* and by example suggests that *"the daily minimum ambient air temperature would on average not be expected to go below the monthly tenth percentile minimum ambient air temperature more than 3 days for a 30 day month."* It should be noted that the percentile temperature calculation used in ASTM D975 is calculated in a different way than the percentile temperatures calculated by the CGSB. ASTM used the minimum daily temperatures for every month over a period of 15 to 20 years resulting in 450-600 data points for each month at each location analyzed. The CGSB used all the hourly data over similar time periods and calculated the 2.5 percentiles from the 11,000-14,500 data points which better considers how long the air

temperature persisted at each temperature. ASTM took a different approach and selected the lowest temperature location in each state or region as the minimum low temperature recommendation for that state or region, so generally the minimum recommendations are much lower than required for most of the state or region. For example, the minimum recommendation for the State of Minnesota is based upon International Falls' historical data which is 300 miles North of Minneapolis and significantly colder.

The Appendix provides both tabulated and graphical monthly 10th percentile minimum air temperatures for October through March for the contiguous 48 states plus Alaska, but it is important to remember that the information in the Appendix is not mandatory. In all but 13 states, the temperatures are considered applicable for the entire state, even though they there may be large temperature differences between different parts of the same state.

The ASTM specification for B100 blend stock, ASTM D6751 Standard Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels contains a non-mandatory Appendix relating to Low Temperature Operability Considerations and not only makes reference to various factors that influence operability but also highlights the potential impact of *"residual constituents"* and offers limitations on cold soak filterability and total glycerin in mitigation. [39]

Pipelines

The specifications of three pipeline operators [40] [41] [42] in the United States were reviewed and found to offer a similar treatment of the low temperature operability requirements, aimed both at storage and transfer of diesel fuel during periods of low temperatures.

In general, all three operators, set maxima for both CP and PP prescribed for both summer and winter seasons. For winter, cloud point is 15°F (14°F in the case of Magellan) and pour point is 0°F whilst for summer cloud point is 20°F and pour point is 10°F.

These CP maxima are generally less restrictive than the final product operability guidelines in ASTM D975 during winter and more restrictive during summer.

The operators also differ in their treatment of cold flow additives. These range from not allowing it, to making the use subject to approval to providing an approved list of additives.

CONCLUDING REMARKS

This guide has been designed to provide information to fuel producers, end users of diesel fuel, fuel distributors and vehicle manufacturers to aid them in avoiding low temperature operability and handling problems. Although it is not practical to identify and to include all possible issues, it is hoped that the knowledge provided in this document will enable the reader to use proper judgement when dealing with analogous situations. The information provided should also guide parties against well-intentioned system modifications and practices that might have appeared helpful but in reality may be detrimental. Examples are the use of additives which may not be compatible with the fuel or with other additives, removing filters to improve flow rate, or not realizing the effect of water in the fuel.

This guide also points out some limitations or concerns regarding specific laboratory tests used to estimate low temperature operability limits for diesel fuel. This does not mean that the tests

should not be used. Tests like CP, CFPP and LTFT have proven very successful in estimating low temperature performance and protecting vehicles, but the user should note that exceptions can occur. As new fuels are introduced and changes to vehicle fuel delivery systems are made, laboratory tests may have to be modified or new tests developed to better correlate to field performance.

This guide is an "evergreen" document. CRC and other organizations often conduct research studies to add to current knowledge. The document will be updated with the development of new information about fuels, injection equipment and vehicle design, and as new regulations are introduced.

List of Acronyms

AWCD	All weather chassis dynamometer
BXX	A blend of petroleum diesel fuel and FAME containing XX vol% FAME
B100	Neat FAME
BTL	Biomass-to-liquid
CCCD	Cold climate chassis dynamometer
CFA	Cold flow additive
CFPP	Cold filter plugging point
СР	Cloud point
CPD	Cloud point depressant
CSFBT	Cold soak filter blocking tendency
ECM	Engine control module
EVA	Ethylene vinyl acetate
FAAE	Fatty Acid Alkyl Ester
FAME	Fatty acid methyl ester
FBP	Final boiling point
GC	Gas chromatography
GTL	Gas-to-liquid
HD	Heavy duty
HVO	Hydrotreated vegetable oils
IBP	Initial boiling point
LD	Light duty
LTFT	Low temperature flow test
LTO	Low temperature operability
MDFI	Middle distillate flow improvers
РР	Pour point
PPD	Pour point depressant
UI	Unit injector
WAFI	Wax anti-settling flow improver
WASA	Wax anti-settling additive

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Gault	Roger	EMA	
Gunter	Garry	Phillips 66	
Hess	Dennis	Infineum	
Lopes	Shailesh	General Motors	
Mitchell	Ken	Shell Canada	
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