



BSI Standards Publication

Automotive fuels — Diesel fuel — Cold operability testing and fuel performance correlation

National foreword

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Automotive fuels - Diesel fuel - Cold operability testing and fuel performance correlation

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European foreword

This document (CEN/TR 16884:2016) has been prepared by Technical Committee CEN/TC 19 “Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin”, the secretariat of which is held by NEN.

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In 2010, CEN/TC 19 adopted Resolution 2010/11 setting the title and scope of WG 34 which were defined as follows:

Title: *“Diesel fuel cold operability correlation”*

Scope: *“Develop a study on the field correlation of the different cold operability (cold flow and cloud point) test results in relation to actual automotive diesel fuel performance in engines in real world cold conditions. For this work historical data on both manual and automatic tests and on 1988, current and, if possible, future engine concepts shall be used. Real market distillate fuels and FAME, plus common blends thereof, shall be used. The working group shall advice towards WG 14, WG 31 and WG 24 on possible improvements towards their test methods and specifications. The result of the group will be, as a minimum, the development of a Technical Report on “Cold operability testing and fuel performance correlation”.*

In view of the parallel ongoing work within DGMK Project 764 “Cold flow properties of diesel and operability of vehicles in winter”, this study is focusing on an investigation into the field correlation of cold operability descriptors (e.g. CFPP, Cloud Point) with actual vehicle performance. In addition the study is evaluating the impacts of fuel properties, cold flow additives and blending, and vehicle technology on cold operability. Given the close relationship between the work of WG 34 and the DGMK Project 764, a liaison between these groups has been established throughout the drafting of this report.

WG 34 would like to acknowledge the significant contributions by members of the working group who have contributed to the publication of this report.

Introduction

Low temperature operability of diesel vehicles is a common concern for all the stakeholders including the vehicle manufacturers and the fuel suppliers. The stakeholders' shared desire is to ensure that the end user is able to operate their vehicle regardless of the ambient temperature conditions.

The Cold Filter Plugging Point (CFPP) method is included in EN 590 as a means to ensure vehicle operability. As all European countries experience different climatic conditions, the limits for cold flow properties of diesel fuel are decided by the National Standardisation Body of each member state within the framework allowed by EN 590. For member states with temperate climates, a different grade of diesel fuel with its corresponding CFPP limit is selected from Table 2 of EN 590:2013 for each season depending upon the climatic conditions.

For member states with arctic or severe winter climates, a different class of diesel fuel is selected from Table 3 of EN 590:2013 for each season depending upon the climatic conditions. In addition to a CFPP limit, Table 3 also includes a maximum Cloud Point limit for each class of diesel, as well as different limits for several other fuel properties (e.g. density, viscosity, distillation, cetane). Some member states also select different climatic grades / classes for specific geographic regions within the country (for example mountainous or colder regions). A lower density can result in diesel fuel with lower volumetric energy content which can negatively impact vehicle volumetric fuel consumption. Thus a balance between ensuring vehicle cold operability and fuel cost is needed.

The application of the CFPP test in middle distillate fuel specifications has facilitated a trade-off between the needs of the market and the costs of the whole system for the customer (i.e. the investment costs in the vehicle diesel fuel filter system and the recurrent costs of the fuel supply). To meet the CFPP specifications without significantly decreasing the yield of middle distillate fuels, the use of cold flow improver additives has been widely adopted by refineries.

Since the CFPP method was developed in the 1960s, several studies have been performed to develop other laboratory methods in an attempt to improve upon the correlation with vehicle cold operability. However this has proved difficult due to constantly changing diesel engine technologies which have necessitated changes in vehicle fuel system design driven by ever more stringent emissions legislation (e.g. the move to direct injection and common rail systems with high pressure pumps requiring changes to fuel filter materials and efficiency). At the same time, middle distillate fuel production has changed significantly over the years as refineries have had to meet increasingly tight fuel quality requirements (e.g. reductions in sulfur content, density and polyaromatic hydrocarbons as well as the introduction of biofuels and higher cetane requirements). Despite all these changes to the vehicles and the fuels, and the development of alternative lab tests, the CFPP remains the foremost test used to protect the end user from cold operability vehicle failures.

At the 37th meeting of CEN/TC 19/WG 24 (November 2009, Brussels) questions were again asked regarding the correlation between cold flow tests and actual vehicle operability at low temperature. Some participants thought that the situation had worsened due to the introduction of finer fuel filters and FAME blending.

At the 38th meeting of CEN/TC 19/WG 24 (March 2010, Teddington) the WG 24 convenor and secretary suggested a scope for a new working group to be formed. This was accepted by WG 24 and the proposal was then forwarded to CEN/TC 19 members (CEN document N1451). The proposal was accepted by CEN/TC 19 on 10 May 2010 (resolution 2010/11).

Following a number of vehicle operability issues experienced, for example during a cold period in the first half of February 2012, in Germany and Austria in particular, a DIN-FAM "mirror" working group was set-up in Germany as a taskforce to investigate the issue. A key outcome was the creation of a new DGMK project 764 "Determination of Cold Operability of Diesel vehicles" to develop and execute a joint industry project. Phase 1 was intended to evaluate several current vehicles from different OEMs to select a new reference vehicle for operability testing. It was also proposed that Phase 2 would investigate the development of a rig test and evaluate a wider range of different fuels in the selected reference vehicle. CEN/TC 19/WG 34 is maintaining close contact with the DGMK project group.

This Technical Report covers operability and tests to assess diesel fuel performance below the fuel cloud point. Although a high filter blocking tendency above the cloud point can have an impact on vehicle operability at low temperature, the development of a lab test to identify this specific issue is being pursued by CEN/TC 19/WG 31 rather than by WG 34.

Finally, it should be borne in mind that the refiners and vehicle manufacturers are not the only stakeholders when it comes to ensuring low temperature vehicle operability. There are a number of other stakeholders involved, including fuel blenders, fuel retailers, biofuel suppliers, cold flow additive suppliers, vehicle fuel system manufacturers, motorists and standardisation bodies like CEN. With this in mind, it is important that each stakeholder shares the responsibility for ensuring low temperature vehicle operability.

1 Scope

This Technical Report lays down the results of a study on the field correlation of the different cold operability (cold flow and cloud point) test results in relation to actual fuel performance in engines in real world cold conditions (below the fuel's cloud point). For this work historical data on both manual and automatic tests and on old (1988), current and future engine concepts have been used. Real market distillate fuels and Fatty Acid Methyl Esters (FAME), plus common blends thereof, have been investigated.

2 Cold flow additives

2.1 Application

2.1.1 Diesel fuel characteristics

Middle distillate fuels are primarily complex mixtures of hydrocarbon molecules. Depending on the source of the petroleum crude and on the level of refinery processing, some 15 % to 30 % of these are *n*-alkanes (also referred to as *n*-paraffins). The carbon number chain length of these alkanes is typically in the region of C8 to C28/C32. As middle distillates are cooled, the heavier *n*-alkanes start to precipitate from the fuel. These are in the form of wax crystals which can be as large as 1mm² and are typically in the form of flat, thin rhomboid plates. As the cooling continues, the wax crystals grow very quickly with *n*-alkanes as low as C18 involved in the precipitation (Figure 1).

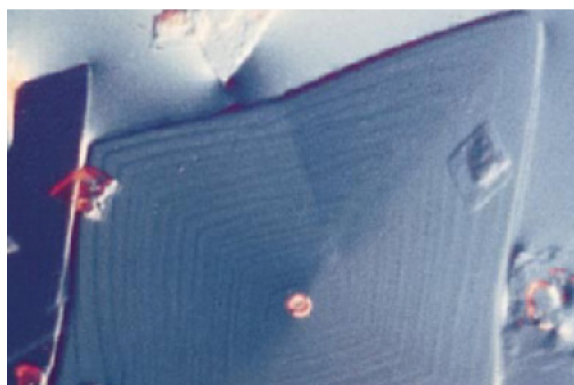


Figure 1 — Wax crystals in untreated diesel fuel (source: Infineum)

The plate-like crystals also exhibit strong edge-edge attractive forces between individual crystals which results in the formation of a gel structure where the majority of the fuel remaining in the liquid phase is trapped in the interlocking crystal lattice. As a consequence of this, a very small amount of precipitated wax may be sufficient to cause solidification of the fuel. Without the use of external heaters or cold flow additives, this phenomenon rapidly causes the fuel filters found within car and heating fuel systems to block resulting in fuel starvation to the engine, loss of power and eventually engine stalling.

In recent years diesel fuels have become more complex as fatty acid methyl ester (FAME), hydrotreated vegetable oils (HVO), Gas-To-Liquid (GTL), etc. have been increasingly introduced into diesel blends. HVO and GTL are paraffinic fuels that are composed of molecules already present in petroleum fuels. FAMES on the other hand are chemical species that are not present in fossil fuels. However – like *n*-alkanes – saturated FAMES can crystallise upon cooling and form part of the precipitated wax together with *n*-alkanes.

2.1.2 Reasons for using cold flow additives

The use of cold flow additives (or MDFI – Middle Distillate Flow Improvers) is not actually a prerequisite when it comes to producing middle distillate fuels. Their use is primarily determined by three factors:

1. Refinery economics
2. Supply and demand balance
3. Regional climate conditions

At all petroleum refineries there are price differentials between the different grades of products. Refiners may therefore improve their blending economics and reduce diesel production cost by either ‘backing out’ kerosene from diesel blends to produce more jet fuel, or by upgrading heavy gasoil components to produce more diesel fuel. Producing more diesel fuel is becoming increasingly important as diesel demand is increasing faster than for all other petroleum products. The same trend is expected to continue for the foreseeable future.

However both mechanisms result in higher fuel cloud point and degraded low temperature operability. By use of an appropriate MDFI additive, the cold temperature operability performance of the cloud point elevated diesel fuel can be maintained at that of the original, non-upgraded fuel. This is illustrated in Figure 2 where diesel production is increased by 15 %. Although this results in an 8 °C increase in the cloud point of the fuel, its operability temperature as measured by Cold Filter Plugging Point (CFPP) is maintained at that of the original fuel. For a detailed description of the cold flow properties and their related test methods mentioned in this section, please refer to 3.4.

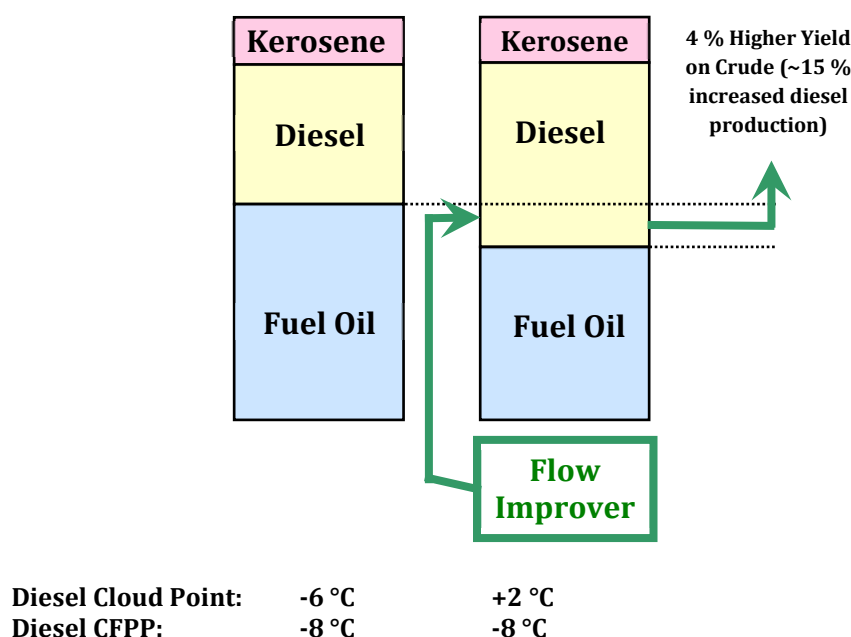


Figure 2 — Heavy gasoil upgrade (illustrative example) (source: Infineum)

2.1.3 Wax crystal growth

The crystallisation of waxes from diesel fuels, not treated with cold flow additives, normally results in the formation of large, flat plates. 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. The plates then begin to slowly thicken to form diamond type shapes. This can be explained by two theories:

- Gibb's theory of discontinuous layer addition
- Frank's theory of continuous growth via dislocations

Gibb's/Frank's theories are graphically illustrated in Figure 3. In step 1, the wax crystal molecule (W) diffuses towards the crystal edge and may be adsorbed at any point (step 2). However, molecule binding onto the existing crystal is stronger at a 'kink' site and this is where the paraffin molecule will in the end be incorporated (step 3). The new kink/dislocation site formed by (W) acts in a similar manner to the original by attracting other paraffinic molecules (V) and continuing the crystal growth (steps 4, 5 and 6).

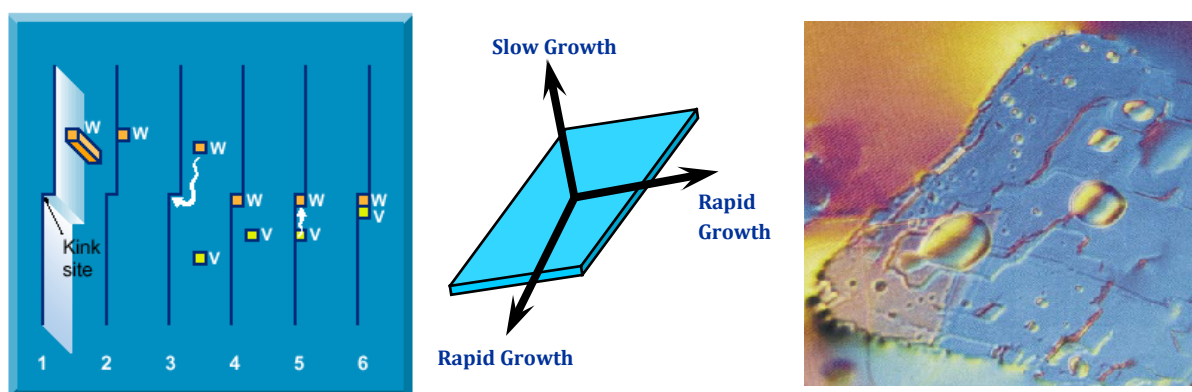


Figure 3 — Wax crystal growth (source: Infineum)

Using this model, it is relatively easy to visualise how a cold flow additive would cause crystal growth inhibition. One side of the molecule resembles a wax molecule and could readily be absorbed into a kink-site on the wax crystal surface. The other side of the molecule is not wax-like and contains a non-binding or blocking group that inhibits further *n*-alkane adsorption and slows down crystal growth. This allows other wax crystals to form with the result that there are more, smaller crystals

Such cold flow additives are primarily *n*-paraffinic in nature with bulky, blocking groups and have a significant effect on the growth and size of the wax crystals (Figure 4).

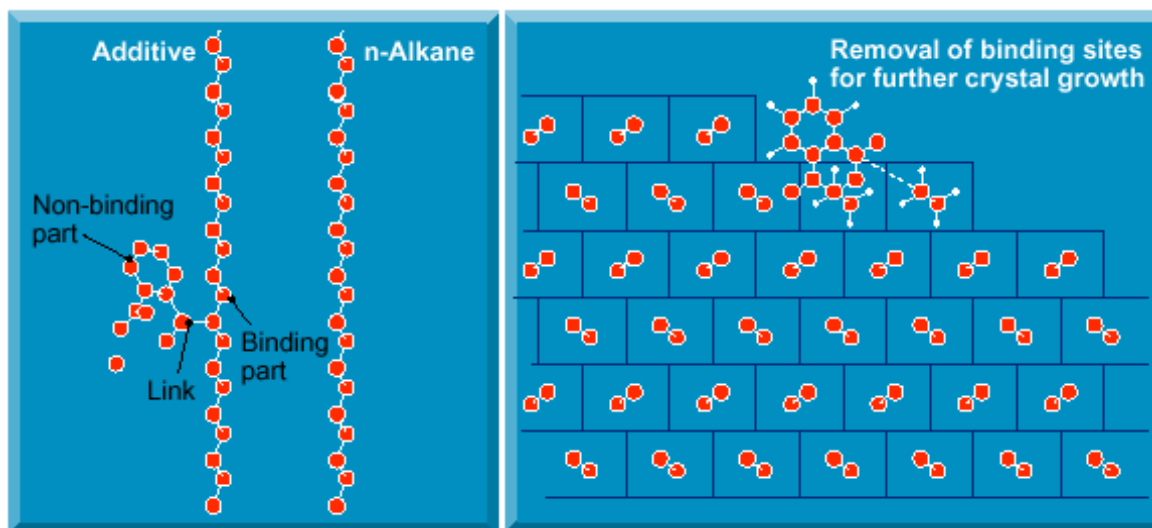


Figure 4 — Chemistry and binding nature of MDFIs (source: Infineum)

One of the most commonly used MDFIs is ethylene vinyl acetate (EVA) which consists of a long polyethylene acting like the n-paraffin and the vinyl acetate side chains acting to sterically hinder the laying-down of further n-paraffins in that plane (Figure 5).

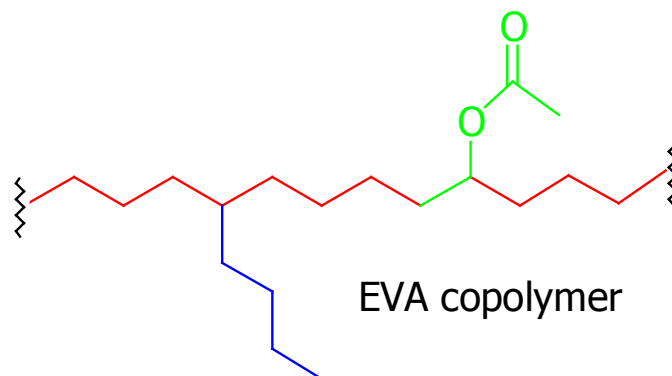


Figure 5 — EVA copolymer (source: Infineum)

Cold flow additives cause growth in the A-B planes (but not in the C plane) to slow down sufficiently such that growth in all three directions is comparatively even. This causes the growth of 'tall' needle-like crystals which compared to the rhombic plates of an untreated fuel are more compact. Although each individual crystal will have a smaller surface area, the total surface area of all the modified wax crystals combined vastly increases. Small crystals would normally 'pack' closely on a filter thus rapidly reducing porosity, however the needle shaped nature of the crystals allows them to form a relatively thick porous cake on a filter before it blocks sufficiently to restrict fuel flow (Figures 6 and 7).

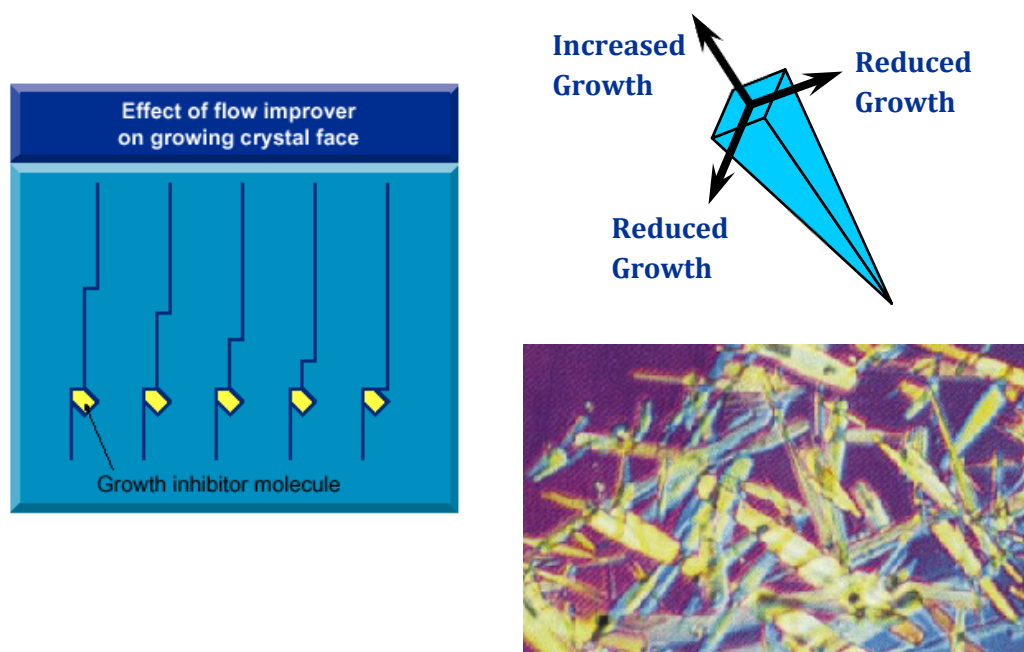
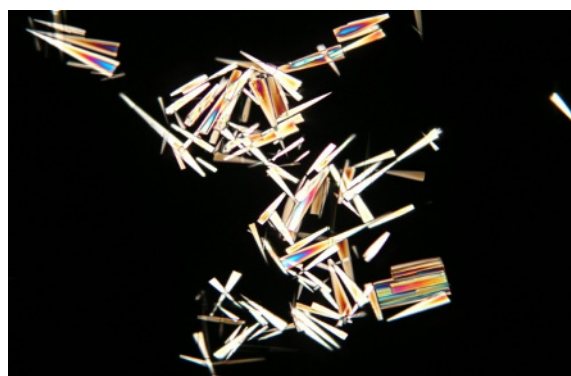
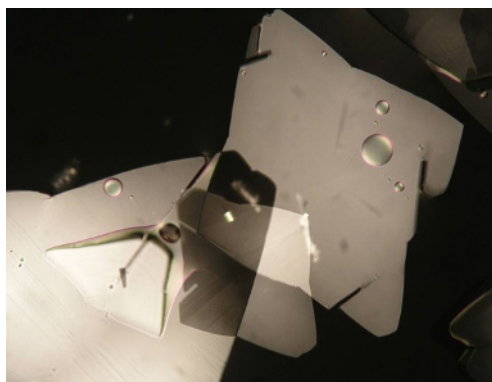


Figure 6 — Wax crystal growth modified by MDFI (source: Infineum)



Thin, flat wax crystals of up to 5 mm in diameter can form in untreated diesel fuels at low temperatures

Cold flow additives modify the wax crystals to compact needle shapes, which allow the fuel to pass through the wax layer on the filter

Figure 7 — Comparison of wax crystals in untreated and treated fuels (source: Infineum)

2.1.4 The stages of Wax Crystal Modification

The different stages of wax crystal growth, both with and without the use of MDFIs, are shown in Figure 8.

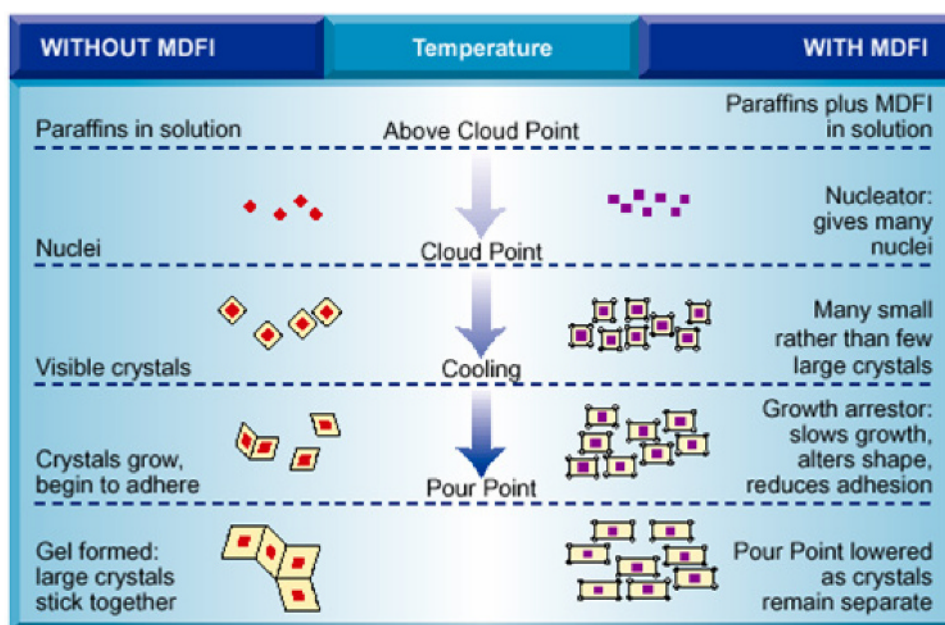


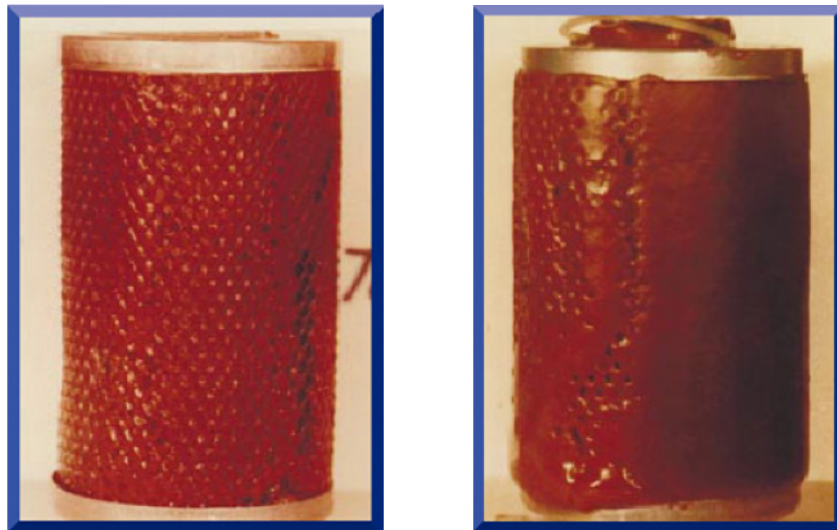
Figure 8 — Temperature dependence on wax crystal growth (source: Infineum)

Above the Cloud Point, all n-alkanes (and additives) are in solution. As the temperature decreases to the Cloud Point, MDFI treated fuel contains nucleators that produces more seed crystals than in untreated fuel. Between the Cloud Point and the Pour Point, the fewer nuclei crystals in the untreated fuel grow into visibly larger crystals, whereas the treated fuel contains many small crystals. At the Pour Point, the now much larger crystals in the untreated fuel begin to adhere. The MDFI treated fuel also contains growth arrestor which slows growth, alters shape and reduces adhesion. Below the Pour Point, the large crystals in the untreated fuel adhere and form a gel thus resulting in performance failure. Action of the MDFIs in the treated fuel maintains crystal separation and allows the Pour Point to be lowered.

The wax formation steps listed above are for diesel fuels that do not contain Fatty Acid Methyl Ester (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. They are described in a later section.

2.1.5 Vehicles and fuel systems

The use of MDFI in fuels sufficiently modifies the wax crystals formed on slow cooling such as to require a relatively 'thick wax cake' to block the filter. In comparison, an untreated fuel will rapidly block a fuel filter with a thin, almost invisible wax layer (Figure 9). That a greater amount of precipitated wax is required in MDFI treated fuels to block a filter is not the only parameter associated with fuel system failure. The cloud point and wax content of the fuel will give an indication of its operability at low temperatures as will the system flow rate and temperature (especially at the filter). The latter is considerably influenced by the design of the vehicle fuel system. It is convenient to describe a vehicle fuel system as being made up of high and low pressure sections (HP and LP) where the cold flow performance is most critical in the LP areas.



Filter exposed to non-treated fuel

Filter exposed to MDFI treated fuel

NOTE The wax appears red in colour due to the fuel containing red dye.

Figure 9 — A vehicle main fuel filter at blocking point (source: Infineum)

In particular, a common feature of most fuel systems is the Low Pressure (LP) return where excess fuel (from the fuel pump/injector feed) is returned to the fuel tank and/or the filter inlet. This excess of warm fuel is essential to the operation of diesel fuel systems at low temperature. As soon as the engine is started, wax starts to accumulate on the main filter. If wax crystal deposition is quicker than the rate of temperature rise, the filter will block and the vehicle will fail through fuel starvation. On the contrary, if the rate of temperature rise is quicker than the rate of wax crystal deposition, the filter will remain sufficiently clear of wax to allow continuous operation. This balancing effect is illustrated in Figure 10.

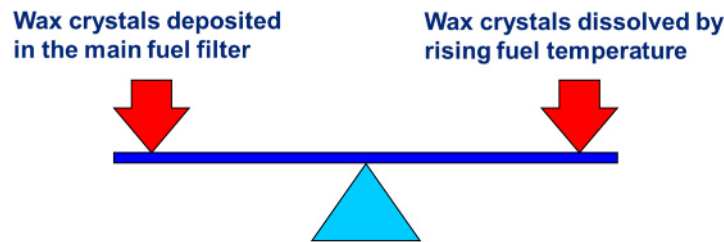


Figure 10 — The operability balance (source: Infineum)

2.1.6 How cold flow additives improve operability

As explained above, it is critical for cold operability that the fuel temperature during driving raises above its cloud point. If not, the vehicle will stop sooner or later due to filter plugging by wax. Cold flow additives 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 begins to dissolve. The example in Figure 11 illustrates this point.

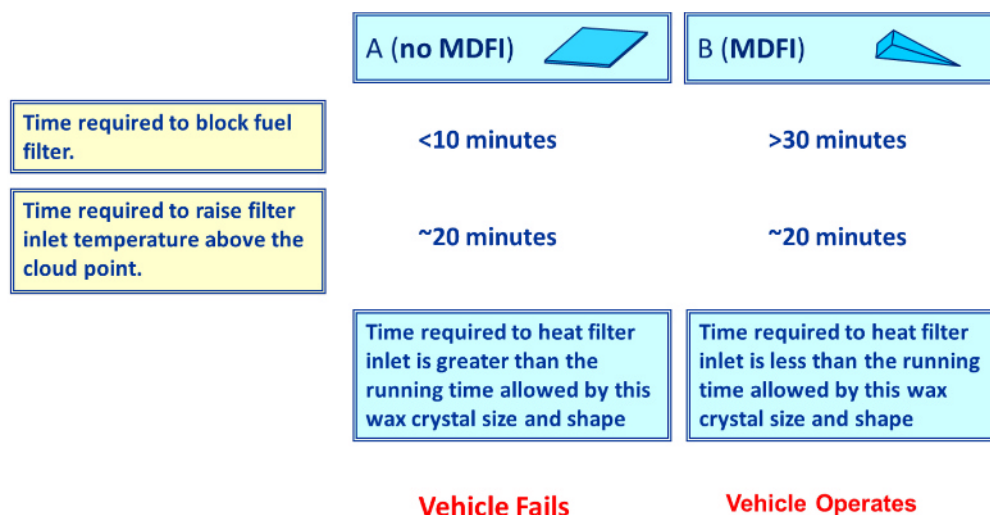


Figure 11 — Example of benefits of smaller wax crystals on vehicle operability (source: Infineum)

2.1.7 The different types of cold flow additive

There are a number of different types of cold flow additive. The original flow improver was the Pour Point Depressant (PPD). They were developed to lower the pour point of diesel fuels and heating oils, that is, lower the temperature at which the gel structure forms and filters are readily blocked. PPDs modify the morphology of the crystal limiting the edge-to-edge interaction of the wax crystals, preventing them from forming gel structures. When used, they are often added to improve fuel handling characteristics in fuel logistics. However they exhibit limited ability in controlling the size of individual crystals and do not always provide a significant improvement in filterability.

Such modification is achieved using Middle Distillate Flow Improver (MDFI). This type of additive is the most commonly used today and it can readily control the size of individual crystals and effectively improve filterability. Further reduction of the wax crystal size can also be achieved using Wax Anti-Settling Additive (WASA) (see Figure 12).

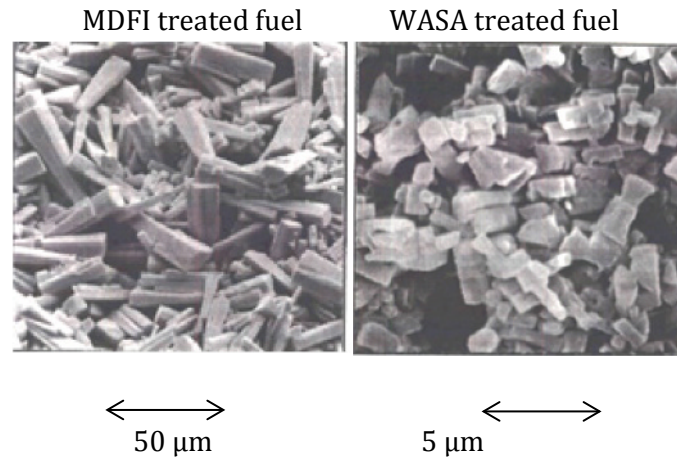


Figure 12 — Wax crystals in WASA vs. MDFI treated fuels (source: Infineum)

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

- 1) Reduced rate of wax settling in fuels stored at a temperature below their cloud point. 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 MDFI.

Improved vehicle operability is indicated by better CFPP performance and good wax anti-settling behaviour.

Cold flow additives are normally 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-crystallises 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 shall remain sufficiently soluble so as not to cause any filter blocking problems itself.

2.2 Storage, handling and blending of cold flow additives

2.2.1 Introduction

Cold flow additives are usually incorporated into the appropriate fuel at refineries, depots or terminals. Each location has a different technical and logistical environment. Therefore, general guidelines for the handling and blending of cold flow additives are only applicable to a certain degree. Each additive facility has to comply with local variations.

Furthermore, legislative and/or company internal regulations and standards in facility safety, environmental issues and health hazards have to be obeyed. It is the intention of this section of the report to provide basic information on how to handle cold flow additives in the best possible way.

2.2.2 Storage

Cold flow additives have to be stored at elevated temperatures. The appropriate storage temperature range can be obtained from the data sheet or chemical hazard data sheet of each product. The chemistry of this type of additive is complex and storing the additive below or above the recommended temperature range may result in some product inhomogeneity. Separation of additive components may then occur.

Additive tanks are generally heated either by steam coils, hot water coils or by electrical heating panels. If the tank contents are steam heated without being stirred or recirculated, then the product near the coils will have a temperature close to the coil skin temperature. If the temperature of the additive exceeds 60 °C, then degradation may occur. Therefore, steam heating is only acceptable when using low pressure steam at normal superheat levels (steam temperature 120 °C - 150 °C).

In any case, for ease of operation, it is recommended to use automatic temperature control. For economic reasons, it is advisable for heated tanks to be insulated with at least 5 cm thickness of fibreglass, mineral wool or PU foam, covered with aluminium cladding. Insulation may be thicker if the ambient temperatures are very low and/or the recommended storage handling temperature of the particular cold flow additive is high.

Assuming the additive tank has good temperature control, there could still be isolated areas of the tank (particularly in large tanks) that may exceed or fall below the recommended storage temperature. To ensure an even temperature, and therefore homogeneity of the additive, the tank should have a mechanical mixer. Depending on the size and geometry of the tank, a top entry or a side entry propeller mixer may be used. The mixer should be mounted at an angle of approximately 15° to its vertical axis and should be equipped with a cut-out device connected to the level indicator of the additive tank. The mixer should not be run without sufficient liquid coverage which may result in damage to the mixer itself or its electric motor.

If the installation of a mechanical mixer is not possible, then tank recirculation via a well-designed jet nozzle can be used. This option may be less effective than a mechanical mixer, especially if the recirculated liquid has a high viscosity. The jet nozzle installed in the recirculation loop shall have enough liquid coverage to avoid breakthrough of the jet through the liquid surface inside the tank, resulting in the generation of an air-vapour mixture with concentrations that may be well within the explosive limits.

In some applications cold flow additives are stored in dilution with kerosene or gas oil. This may be done in order to improve the additive handling characteristics (lower viscosity) or to reduce the additive storage temperature (energy conservation credit). In these cases continuous agitation of the tank contents is absolutely necessary. However, the additive manufacturer should always be consulted if you wish to pre-dilute the additive.

Tank vents to the atmosphere should be equipped with dryer caps, especially when storing Wax Anti-Settling Additives (WASA) and additive packages containing WASA. These products are very sensitive to water contamination.

The tank material can be carbon steel unless otherwise specified in the chemical hazard data sheet of the additive in question.

Housekeeping is another important factor when storing cold flow additives. Tanks should be clean, free of rust or other solid particles, and the tank shall be dried before filling with the additive. All such contaminations may have detrimental effects on the performance of the additive.

2.2.3 Injection

The most common way to inject cold flow additives into fuel is by using positive displacement pumps (piston or rotary gear pump). A positive displacement pump delivers a constant volume, regardless of the pressure. It is limited by the capability of the pump driver, normally an electric motor, to work against the pressure encountered. Therefore, the sizing of the motor is critical to ensure that it will not incidentally stop operating. The pump should be protected against over-pressure by a pressure relief valve. It is important to specify the proper operating pressure when purchasing the pump. Additionally, a flow switch on the discharge should be installed to shut down the pump in case of no flow.

While the positive displacement pumps are the most accurate means of injection, they do not supply a continual additive rate because of the fluctuation in discharge pressure. This problem can be partially overcome by installing a pulse dampener on the pump discharge.

The following general points apply to the piping layout of the cold flow additive system:

- The pump should be located as close as possible to the additive tank to minimise the length of the suction piping.
- The piping between storage tank, additive pump and injection point should be carbon steel, unless otherwise specified in the additive's chemical hazard data sheets.
- The layout of these lines should avoid high/low points, or dead space.
- The lines should be held rigid to avoid excess wear due to the pressure fluctuations generated by the positive displacement pumps.

It is desirable to avoid any additive being left stationary in the lines between pumping operations. Therefore, the lines should have flushing facilities to allow removal of the additive, or a return line should be installed between the pump and the storage tank so that recirculation can be carried out continuously. Both methods will avoid potential problems associated with deposits, corrosion and air pockets formation in the line. Particular care is required during start-up of the cold flow additive facility after a refinery turnaround operation.

In those cases where the cold flow additives are stored at higher temperatures, all lines containing the additives should be heated (steam, electrical) and insulated.

2.2.4 Blending

The overall objective is to have the cold flow additive completely dissolved in the fuel. This will ensure consistent product quality. For refinery blending, the additive should be added to the fuel stream (individual blend component or blended fuel) as the fuel blend is being made at the correct treat level ratio. Adding the additive in batches should be avoided.

For optimum dispersion of the additive in the fuel, it is recommended to use an injection nozzle located in the centre of the fuel pipeline, pointing downstream of the fuel flow (see Figure 13). The calculation procedure for an appropriate injection nozzle is given by the following formula:

$D2 \text{ [MM]} = D1 \times \sqrt{\frac{Q2}{Q1 \times 1000}}$
$D2 \text{ [MM]} = D1 \times \sqrt{\frac{R}{10^6}}$

Where

- Q1 is the Fuel Flow Rate [m³/hr]
- Q2 is the Additive Flow Rate [l/hr]
- D1 is the Main Pipe Diameter [mm]
- R is the Treat Rate of the additive [cm³/m³]

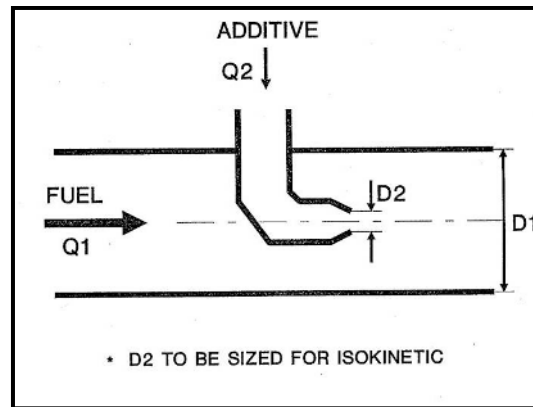


Figure 13 — Optimum additive injection location - to calculate Injection Nozzle Diameter (D2) either use Formula 1 or Formula 2 (source: Infineum)

The temperature of the fuel at the injection point has a significant effect on the proper dispersion and mixing of the cold flow additive in the fuel. The fuel temperature should be at least 25 °C. In those cases where the temperature is below 25 °C, then pre-diluting the additive with kerosene, gas oil or solvent is essential. In any event, the temperature of the fuel at the injection point should always be at least 5 °C above the fuel cloud point.

To further improve the mixing between additive and fuel, a static mixer should be installed in the fuel line, downstream of the injection point. The need for a mixer in the line can be avoided providing the fuel flow has a Reynolds number greater than 8 000 (turbulent flow) and if there is at least 100 x pipe diameter of pipeline downstream before entering the final fuel tank or the loading rack. The Reynolds number may be calculated according to the simplified Formula (1):

$$RE = \frac{353678 \times Q1}{D1 \times NUE1} \quad (1)$$

Where

- RE is the Reynolds Number
- Q1 is the Fuel Flow Rate [m³/hr]
- D1 is the Main Pipe Diameter [mm]
- NUE1 is the Kinematic Viscosity of Fuel [cSt]

2.2.5 Example layouts of Cold Flow Additive Injection Facilities

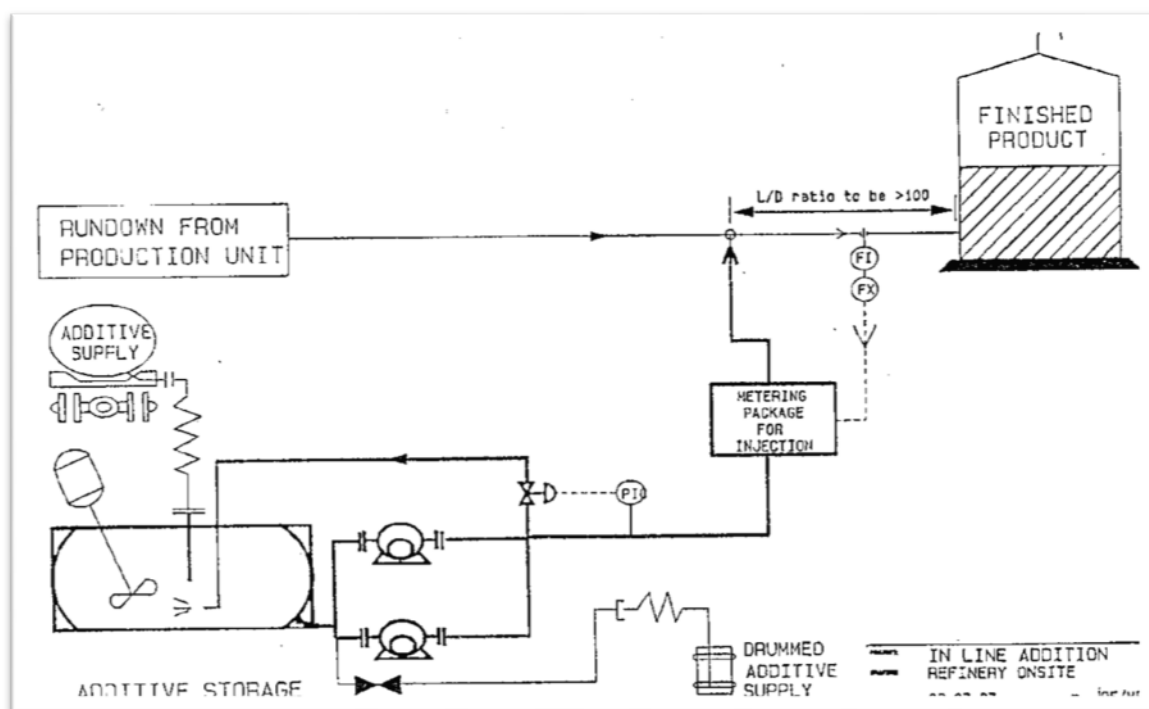


Figure 14 — In-line addition (refinery) (source: Infineum)

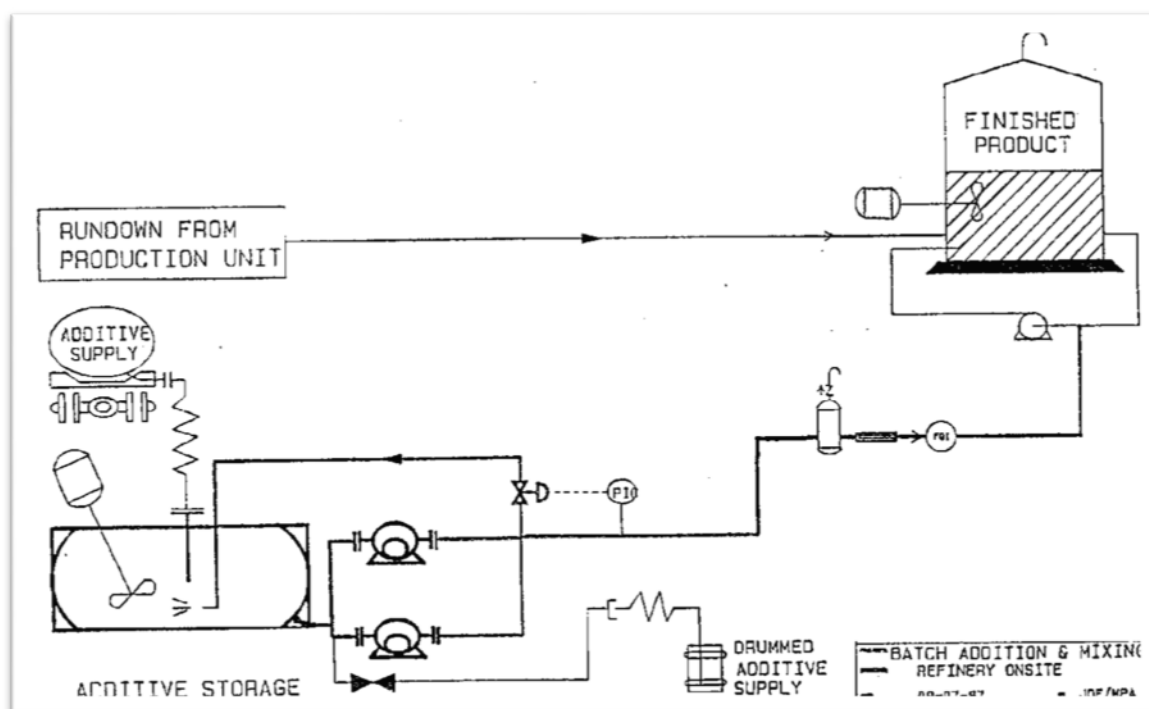


Figure 15 — Batch addition and mixing (refinery) (source: Infineum)

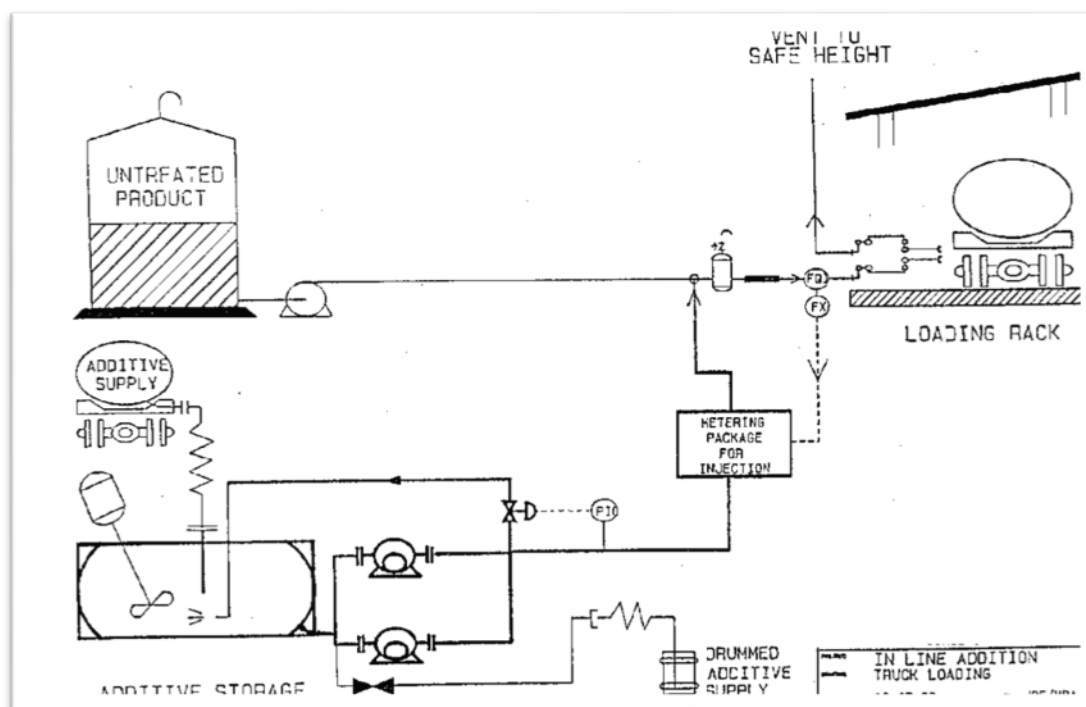


Figure 16 — In-line addition (truck loading rack) (source: Infineum)

2.2.6 Summary

Cold flow additives are high performance, high value products, used to improve fuel characteristics with benefit to the end users. The proper handling of cold flow additives and their optimum blending into the fuel are essential factors to ensure product quality is maintained throughout the supply chain, from the manufacturing site to the end user's appliances.

This report cannot cover the whole subject of handling and blending of additives due to the wide range of facilities, user needs and climatic conditions that exist. For further information on specific issues, users should contact their cold flow additive suppliers.

3 Cold flow tests

3.1 Vehicle operability

Diesel vehicle cold weather operability limit can simply be defined as the minimum temperature at which a diesel vehicle can be satisfactorily operated. However, vehicle operability is actually difficult to define in practice. Vehicles can be widely different and have widely different cold temperature operability limits. As a result, it is impossible for any test to relate to all vehicles.

Even for one particular vehicle, different testing conditions may result in a different measure of operability. The temperature cooling profile is important as it affects wax crystal growth and size. It is uncontrolled in the case of field trials, and will vary from day to day. It is controlled in the case of climate chamber tests, but can be changed to simulate different climate conditions or vehicle usage patterns.

Failure definition may also vary. For example, it can be defined as:

- Vehicle does not start

- Engine fails during the test
- Inability to maintain speed throughout the test
- Driver's rating (surge or misfires, need to adjust pedal to maintain speed).

3.2 Assessment of vehicle operability

In spite of the above caveats, it is important to check the suitability of diesel fuels for satisfactory operation of vehicles in winter time. Ideally, vehicle operability studies are performed as they give the best determination of operability when market representative vehicles are used. As there is no industry standard for cold weather vehicle operability tests, each organisation conducts tests they consider most appropriate. Historically, vehicles that have been accepted by industry as "severe vehicles" have been used in operability studies.

The CEC M-11-T-91 code of practice is available for vehicle operability tests conducted within a climate chamber using a chassis dynamometer (so called "Cold Climate Chassis Dynamometer" or "CCCD"). It is widely used in Europe (but often modified) and provides guidance on:

- Vehicle preparation
- Setting of the chassis dynamometer conditions
- Vehicle cold start test method
- Vehicle operability test method
- Driveability assessment.

It does not provide guidance on:

- Vehicle selection (type, equipment fitted etc.)
- Pass or fail criteria.

Vehicle operability studies need complex equipment, are time consuming and expensive, and are therefore usually not used in today's fuel specifications to specify diesel cold flow properties. An exception is the German oil industry exchange agreement for diesel fuels, which includes an optional vehicle cold flow operability test.

Therefore several rig tests and lab based methods have been developed to allow a faster, more efficient estimation of low temperature vehicle operability.

3.3 Low temperature operability rigs

3.3.1 General

A number of oil companies, vehicle manufacturers and cold flow additive suppliers have developed their own operability rigs to assess fuel operability.

Operability rigs typically consist of a fuel tank and a fuel injection system – including fuel pump(s), main filter, and fuel return line to the tank – inside a large programmable freezer unit. The main fuel pump is driven by an electric motor rather than by a diesel engine. The fuel systems can be from different vehicle makes, hence different designs exist.

Various sensors are used to measure air and fuel temperature, as well as fuel flow rates and pressure drop across filters. Assessment of operability is generally based on these measures, but the way of assessing operability and the pass/fail criteria may vary from one rig to another.

Fuel is cooled to the target test temperature according to a pre-determined profile, followed by a soak period at the target test temperature before starting the test. Typical test duration is between 10 min and 30 min depending on the rig.

The advantages of rigs are that they are quicker and a smaller volume of fuel is required than for CCCD or field trial operability testing. As fuel is not burned in an engine, it can be reconditioned by warming up after testing and re-used for a test at a different temperature. Testing at different temperatures is required to assess the operability level of a specific fuel.

Two different rigs are described below as examples.

3.3.2 Daimler low temperature rig

3.3.2.1 Schematic overview

In Figure 17 a schematic overview of the Daimler test rig is given. The fuel is drawn from a fuel container into a two-compartment tank system which is used in some Mercedes-Benz vehicles. In both of the fuel tank compartments, jet pumps are mounted that pump the fuel into a reservoir located in one compartment of the tank system. The low pressure electrical pump (EKP) is integrated in this reservoir. The EKP delivers the fuel to the fuel filter. Optionally, a heating device can be used to warm the filter and to simulate the warm fuel backflow from the engine. The high pressure pump (HDP) is used as a throttle only to simulate the pressure drop in the vehicle system. Pressure and temperature sensors are installed in front and after the fuel filter. A flow rate sensor is mounted after the fuel filter monitoring the flow rate, and flow rate is used as the pass/fail criterion for the rig test.

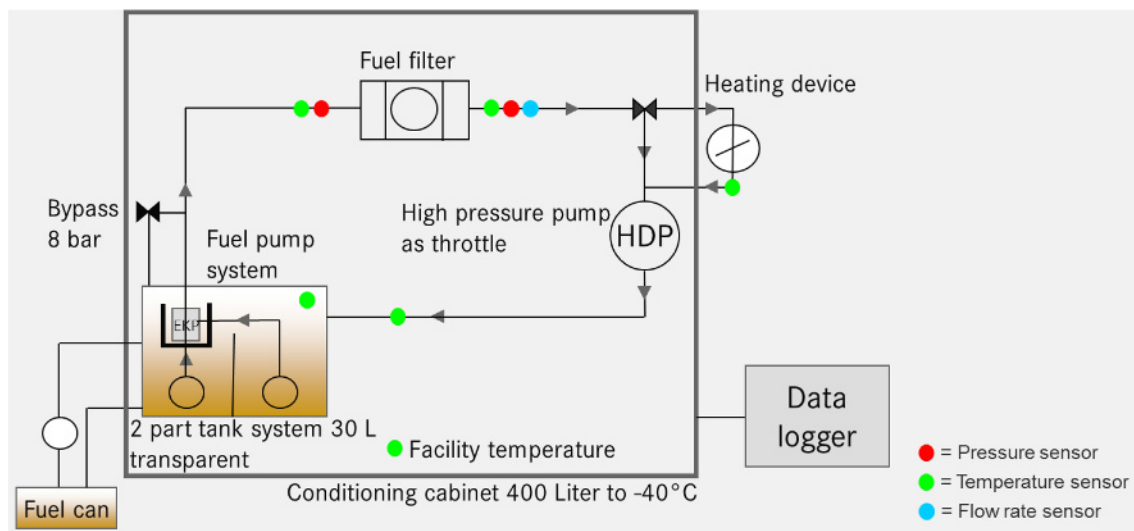


Figure 17 — Daimler operability rig test overview (source: Daimler)

A detailed rig test description is given in Table 1.

Table 1 — Details of Daimler rig test (source: Daimler)

Vehicle on which the rig is based	2007-2014 Mercedes C Class (W204) with OM651 solenoid injection system set up (replacement by other systems is possible)
Tank size	Approx. 30 l
Sample Volume	30 l
Main Filter Type	Retention of > 85 % @ 3-5 µm, 1600 cm ² filter surface area, 75,000 km filter change interval
Other filters	300 µm tank sieve
Position of filters	Close to the engine
Lift pump before filter	EKP is mounted in tank reservoir, lift pump is mounted in both tank parts
Fuel recycling	Fuel is circulated not drawn during test procedure
Possibility to heat the fuel	Optionally warm up of test fuel through heat exchanging device is possible

3.3.2.2 Daimler rig test procedure

After the fuel tank is filled up with 30 l of the test diesel fuel, the fuel tank is cooled down to the target temperature. The following cooling procedure is used:

- 2 h cooling of the conditioning cabinet from ambient to the target temperature.
- 6 h cold soak at the target temperature.
- Start of EKP pump.

Relevant signals are measured for about 20 min or until the flow rate decreases under a critical defined limit.

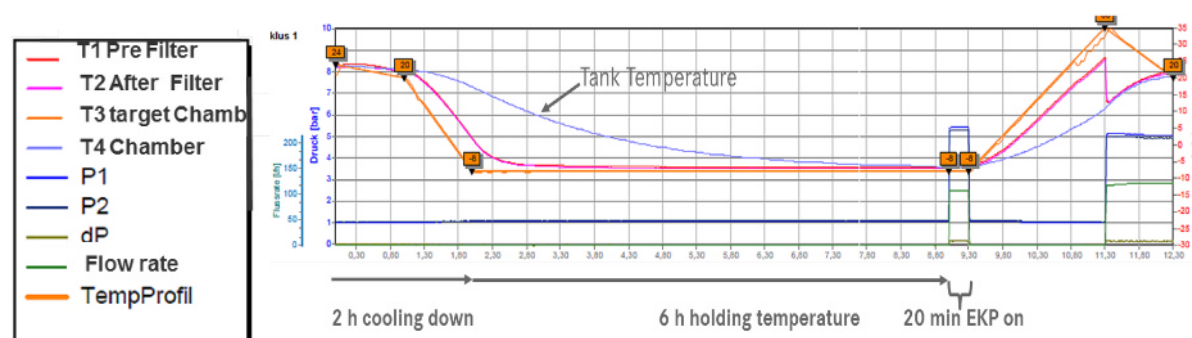


Figure 18 — Typical cooling and test profile for the Daimler operability rig test procedure (source: Daimler)

After each cooling test the fuel is warmed up to 30 °C – 40 °C to condition the test fuel. After fuel conditioning, the next cooling step is initiated and the procedure is repeated.

3.3.2.3 Pass/fail criteria

The flow rate signal from the flow rate sensor is used to determine the pass/fail of a fuel under test. If the flow rate declines under a critical level the test has failed. The critical level is dependent on the injection system and engine type. In the case of the current OM651 rig test set-up, the minimum flow rate required to operate is 40 l/h.

3.3.3 Infineum low temperature rig

3.3.3.1 Schematic overview

The Infineum rig comprises a rotary injection pump fuel system from a 1987 Ford Escort 1.6D vehicle. The fuel system is mounted inside an upright programmable freezer unit with a glass front, capable of maintaining a minimum temperature of -40 °C (see Figure 19).



Figure 19 — Photograph of Infineum low temperature rig test (source: Infineum)

Typical test temperatures are in the range of -10 °C to -25 °C, although for Arctic grade fuels -40 °C may be required. The pump is driven by a variable speed 5,2 kW electric motor with a speed range of zero to 2 500 rpm. A 5 l, tilting fuel tank assembly supplies fuel via a standard Ford Escort fuel filter to the rotary injector pump. To simulate normal engine operation the fuel pump injects fuel past the injector nozzles into four 500 ml measuring cylinders.

Fuel is returned to the tilting fuel tank via leak-off fuel lines. These are configured such that the warm fuel returns to the tank away from the fuel inlet. This is to prevent the warm fuel melting the wax near the fuel inlet. All fuel lines and fittings are manufactured from stainless steel. To simulate vehicle cornering effects the tank is rocked by an electrically driven tilting assembly. Various temperature and pressure sensors monitor the system operation during both the cooling and test cycles. These parameters, together with the injector pump speed, are monitored automatically during both the cool-down and test operation cycles.

Table 2 — Details of Infineum rig test (source: Infineum)

Vehicle on which the rig is based	1987 Ford Escort 1.6D indirect injection
Tank size	Approx. 5 l
Sample volume	4 l
Main filter retention	5-7 μm
Main filter area	$\sim 900 \text{ cm}^2$
Filter change interval for European use	N/A
Other filters	None
Position of filter	Main filter before the rotary injection pump
Lift pump before main filter	No
Fuel recycling	Recycled to tank
Possibility to heat the fuel recirculated to tank, or other means of changing the fuel temperature	No

3.3.3.2 Infineum rig test procedure

After an 18 h cool-down (consistent with the CEC procedure for cold weather performance of diesel vehicles, CEC M-11-T-91) the rig is operated for a total period of 24 min.

Prior to each test a new fuel filter element is installed and the fuel system bled. Once the computer programme has been loaded and the appropriate cool-down and test temperatures set, the 'Initial Cool Down' can commence, cooling to 5 °C above the cloud point of the fuel (10 min). After the two hour soak stage the temperature is ramped down linearly to the test temperature (12 h). The test temperature is maintained for a minimum of 4 h.

After the 18 h cool-down, the test program is advanced to the 'Idle Stage'. The electric motor/fuel pump is started and operated at 400 rpm for a period of 30 s. The rig speed is then increased to 1 000 rpm. Note shall be made if the pump fails to start fuel delivery during these stages. After 3 min and 30 s at 1 000 rpm the speed is increased to 1 500 rpm. If the pump fails to start delivery of fuel at this stage the test shall be stopped. Note shall also be made of any 'misfires' that may be heard as a result of the pump failing to deliver fuel. This is confirmed by a fluctuation in the pump inlet temperature. After 20 min at this speed (total running time of 24 min) the rig is stopped and warmed to 25 °C. The amount of fuel injected into the measuring cylinders is then recorded.

3.3.3.3 Pass/fail criteria

As in the CEC M-11-T-91 test procedure, a demerit system is used to assess the fuel performance. A delayed start or failure to start, 'misfires' during operation, the maximum filter delta and the amount of fuel delivered are used to calculate a rating of 0 to 100 demerits, 40 demerits being the pass/fail mark.

3.4 Diesel cold flow test methods

3.4.1 Introduction

Several laboratory-based methods have been developed to allow a faster and more efficient estimation and correlation with low temperature vehicle operability.

Among these methods there are two principal tests, the Cloud Point (CP) and the Cold Filter Plugging Point (CFPP), that are nowadays widely used throughout Europe and beyond. These

methods are described in the following sections including some additional test methods that have been developed or used elsewhere.

3.4.2 Cloud Point (CP)

The temperature at which a cloud of wax crystals first appears in a liquid when it is cooled under specified conditions is the CP. The CP test (EN 23015), as shown in Figure 20, indicates the earliest appearance of wax precipitation that might result in plugging of fuel filters or fuel lines under prescribed cooling conditions.

The CP is usually determined in a fully automatic detector device. Typically this type of device operates by cooling a diesel sample 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 EN 23015 is 2 °C for distillates, and reproducibility is 4 °C.

Various automatic methods exist, some using the same test jar and cooling bath assembly as EN 23015, others using smaller sample volumes (as low as 0,15 ml) in test chambers with modified dimensions. The automatic methods typically use different optical devices to detect the formation of wax crystals.

The various automatic methods specify different cooling rates – some require stepped cooling whilst others require linear/constant cooling rates. The precision of all the methods varies. However, in every case, the automatic instruments provide greater precision than the manual method. In all of the automatic test methods there is a relative bias versus the manual method (positive or negative bias). In some methods this bias is very small and statistically insignificant, whilst in others the bias is statistically significant but too small to be considered of any practical significance, whilst for some methods the bias shall be added / subtracted in order to report the result as equivalent to the manual method.

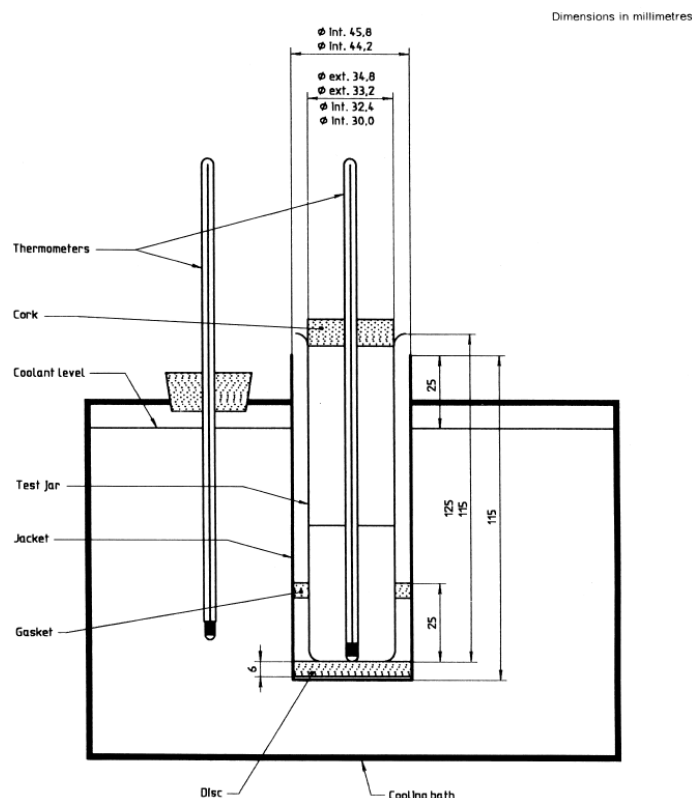


Figure 20 — Schematic of manual CP apparatus

3.4.3 Cold Filter Plugging Point (CFPP)

The Cold Filter Plugging Point (CFPP) test was first introduced in Europe in 1965 and is now specified in EN 116 and EN 16329. The CFPP was originally designed to correlate with cold operability of the majority of European vehicles.

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. The fuels differed in their cloud points - some of these fuels contained cold flow improver additives, whilst 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 parameters of the CFPP method were adjusted to provide the best correlation with the operability data generated in the trial. It is important to note that there was no intention to mimic the design of the vehicle fuel system and/or recreate the conditions of the field trial in designing the CFPP test procedure. The fuel test results generated with the finalised CFPP method confirmed that there was a good correlation to the cold operability data generated in the 1964/65 field trial (Figure 21).

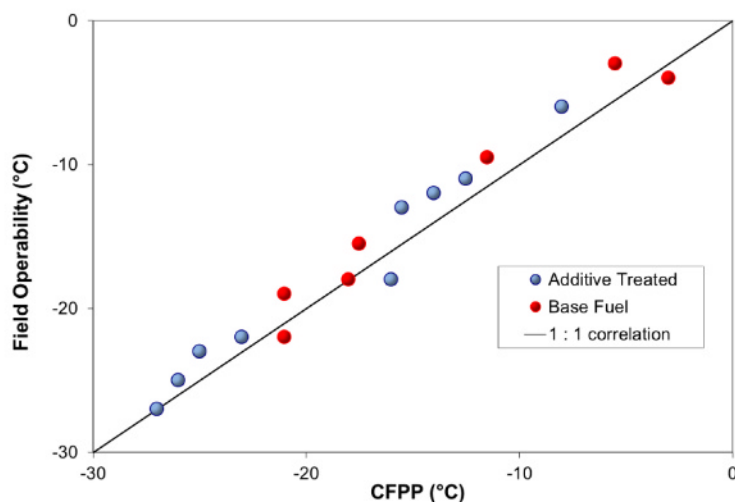


Figure 21 — Correlation of the Swedish field operability data 1964/65 vs. CFPP (source: Infineum)

In the CFPP test a specimen of the sample is cooled under specified conditions and is drawn at each test temperature step into a pipette under a controlled vacuum through a standardized wire mesh filter of 45 µm (as shown in Figure 22). The filtration step is repeated in 1 °C increments as the specimen continues to cool.

Testing continues until the amount of wax crystals that have separated out of solution is sufficient to stop or slow down the flow so that the time taken to pass through the wire mesh filter exceeds 60 s (known as CFPP Type 1 result) or the fuel fails to return completely to the test jar before the fuel has cooled by a further 1 °C (known as CFPP Type 2 result). The temperature at which the last filtration was commenced is recorded as the CFPP.

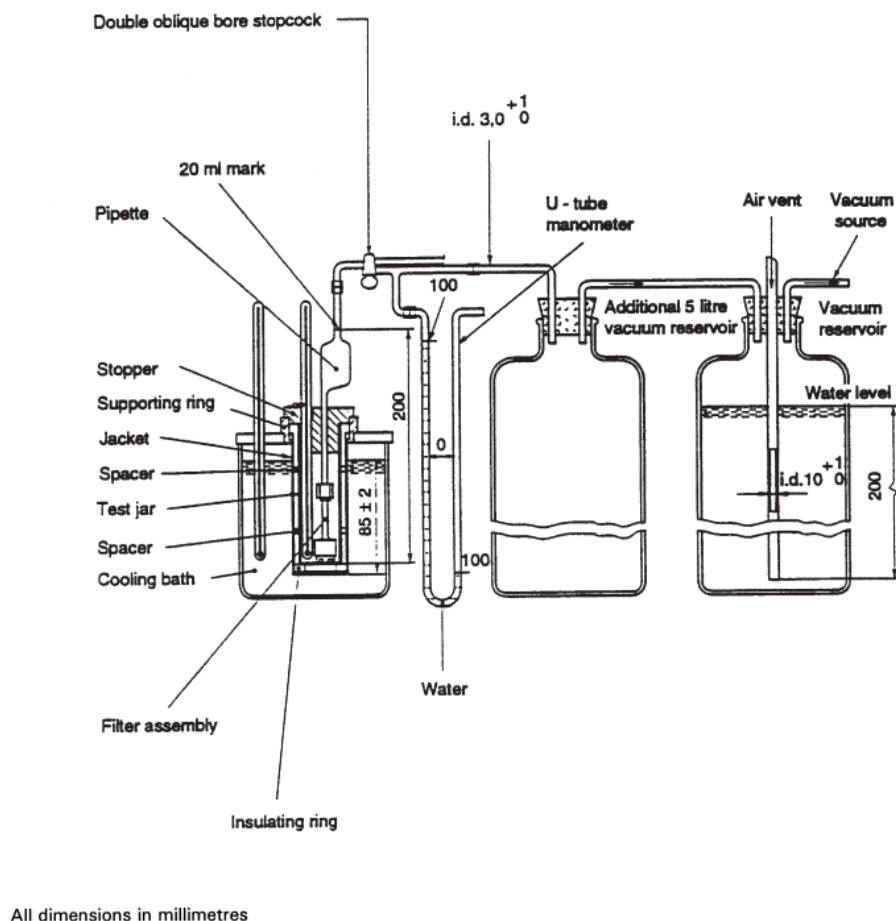


Figure 22 — Schematic design of a CFPP measuring device (according to EN 116:1997)

For non-additised fuels the CFPP is usually somewhat lower in temperature than the CP. For Fuels which have been treated with cold flow additives (e.g. MDFI and/or WASA) the difference between CP and CFPP can be quite significant (up to 20 °C). The CFPP is often regarded as a good indication of the vehicle operability, although it has been shown with some vehicles that the CFPP can be too optimistic compared to the “real” cold operability limit, especially when the difference between CP and CFPP is large (i.e. >12 °C). It is important to note, however, that the CFPP test was designed to correlate with vehicle performance and not with the actual fuel system design.

In addition to the standard CFPP method there are some less common test method variants (e.g. “CFPP 12” and “CFPP 20”) that have been proposed but not standardised. For all the CFPP method variants and the CFPP method itself, the measurement principle is identical; however there are differences in terms of the filter mesh size and/or the cooling rate adopted in the variant methods. For further details on the CFPP method and its variants, please refer to EN 116 and EN 16329.

3.4.4 CFPP hesitation effect

Usually a 1 °C reduction in temperature during the CFPP test is followed by an increase in the time required for the fuel sample to get through the filter. A fuel is considered to “hesitate”, when the filtration time reduces with falling temperature during the test. This effect is also known as an aspiration anomaly.

Only fuels which contain cold flow additives show hesitation effects. There is a trend for fuels which contain performance additive packages in addition to cold flow improvers to show a

stronger tendency towards exhibiting the hesitation effect. In addition, the hesitation effect tends to be more common in heating oils and higher final boiling point diesels that are not normally found in Europe. For these fuels, this effect can have a significant influence on CFPP test precision as a strong hesitation effect can lead to different CFPP values between equipment and between different tests on the same piece of equipment. Figure 23 shows that a small variation in filtration time (e.g. CFPP machine dependent) in the vicinity of -20 °C leads to completely different CFPP values for the same fuel represented by the blue and pink lines.

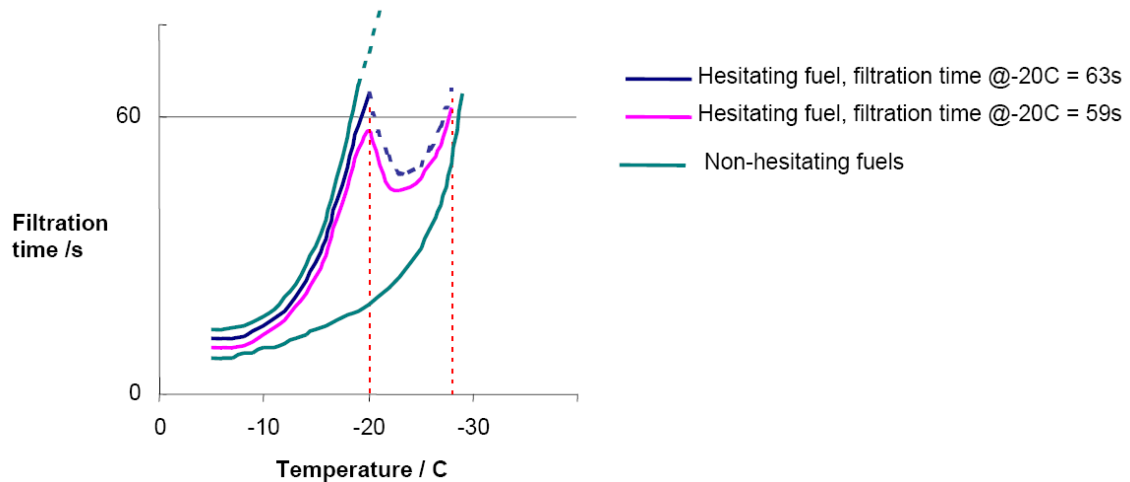


Figure 23 — Impact of hesitation effect (source: Shell)

The magnitude and appearance of the hesitation effect is dependent on the type of CFPP measuring device. In cases where a CFPP result is unexpectedly high, the raw data should be checked carefully to identify any anomalous behaviour.

3.4.5 CFPP Type 2 result

When the fuel fails to return completely to the test jar before the fuel has cooled by a further 1 °C and the next filtration is due to start, the indicated temperature at which the last filtration was commenced is recorded as the CFPP. This is known as a “CFPP Type 2” result and can also be referred to as a “CFPP-2”, “c2” or “type 2” by CFPP equipment manufacturers. It is also sometimes referred to as “stuck in tube”.

The main issue with CFPP Type 2 results is that they are not consistently repeatable:

- Replicate testing on the same fuel sample may produce either a CFPP Type 1 (normal) or a CFPP Type 2 result.
- The proportion of CFPP Type 2 vs. CFPP Type 1 results can vary between laboratories and test apparatus.
- CFPP Type 2 results are higher / warmer than CFPP Type 1 results, potentially leading to a large spread of test results and poor test precision.

CFPP-2 is a complex phenomenon which involves:

- Fuel characteristics
- Additive formulation
- Test equipment and possibly lab ambient conditions if they affect cooling rate
- Cooling rate - slower cooling tends to give more CFPP Type 2 results. For example, in stepped cooling apparatus (EN 116) an increased chance of CFPP Type 2 results is often observed when the fuel temperature is around -20 °C. This is the point at which the

cooling rate is at its slowest just before the cooling bath change-over from -34 °C to -51 °C coolant.

3.4.6 Simulated Filter Plugging Point (SFPP)

The simulated filter plugging point (IP 419 [now withdrawn], CEN CR 13837) was developed in Europe after the CFPP test in the early 1990s. It was the intention of the SFPP test to improve upon the CFPP test with regard to the prediction quality of the temperature dependent vehicle operability.

The measurement principles of both the CFPP and the SFPP tests are to a large extent identical, however some changes were made for the SFPP test such as a lower cooling rate (to more closely reflect the slow crystallization of wax crystals in the field and the effect of WASA), a smaller filter pore size, a special convection cell to better control sample temperature and the filter holder was modified. In addition, the fuel is drawn from the top of the filter to capture the effect of wax settling.

As in the CFPP test, a specimen of the sample is cooled under specified conditions and filtered at intervals of 1 °C. At each test temperature step the fuel is drawn into a pipette under a controlled vacuum through a standardized 25 µm wire mesh filter. The filtration procedure is repeated at 1 °C intervals until the amount of wax crystals that have separated out of the solution is sufficient to stop or slow down the fuel flow. Figure 24 shows a schematic overview of the automatic SFPP test equipment.

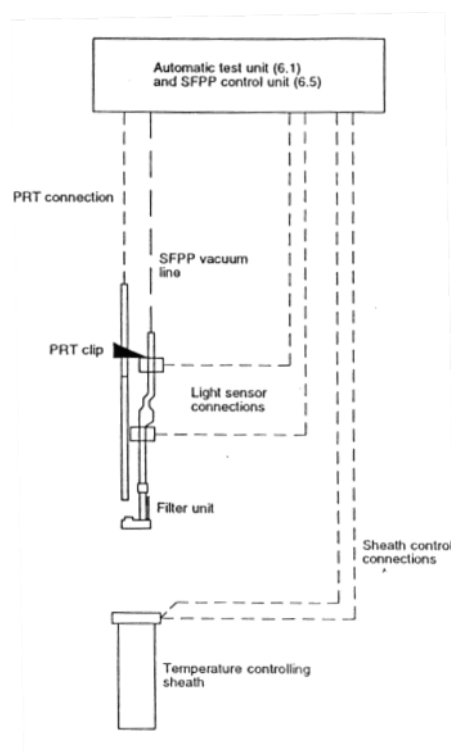


Figure 24 — General arrangement of automatic SFPP test equipment (source: Energy Institute)

3.4.7 Low Temperature Flow Test (LTFT)

The Low Temperature Flow Test (ASTM D 4539) was designed to correlate with fuel delivery systems that are used in North American heavy duty trucks. Therefore this method has been mostly applied in North America. It can be regarded as an alternative to the CFPP method.

In the LTFT the temperature of a series of test specimens of the same fuel is lowered at a prescribed cooling rate. Commencing at a desired test temperature and at each 1 °C interval thereafter, a separate specimen is filtered through a 17 µm screen until a minimum LTFT pass temperature is obtained. The minimum LTFT pass temperature is the lowest temperature, expressed to the nearest 1 °C, at which a test specimen can be filtered in 60 s or less. Alternatively a single specimen may be cooled as described above and tested at a specified temperature to determine whether it passes or fails at that temperature. The LTFT results are indicative of the low temperature operability limit.

3.4.8 Summary of the main low temperature operability lab tests

Table 3 summarizes key aspects of the CFPP, SFPP and LTFT tests: