SLOVENSKI STANDARD
SIST-TP CEN/TR 16884:2016

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Goriva za motorna vozila - Dizelsko gorivo - Odvisnost med hladnim obratovalnim preskušanjem in učinkovitostjo goriva

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

Dieselkraftstoffe für Kraftfahrzeuge - Prüfung der Betriebsfähigkeit bei Kälte und Zusammenhang des Kraftstoffverhaltens

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ICS:

75.160.20	Tekoča goriva	Liquid fuels

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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 1mm^2 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).

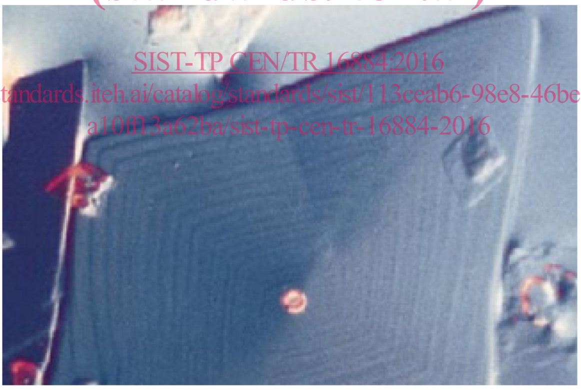


Figure 1 — Wax crystals in untreated diesel fuel (source: Infineum)

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.

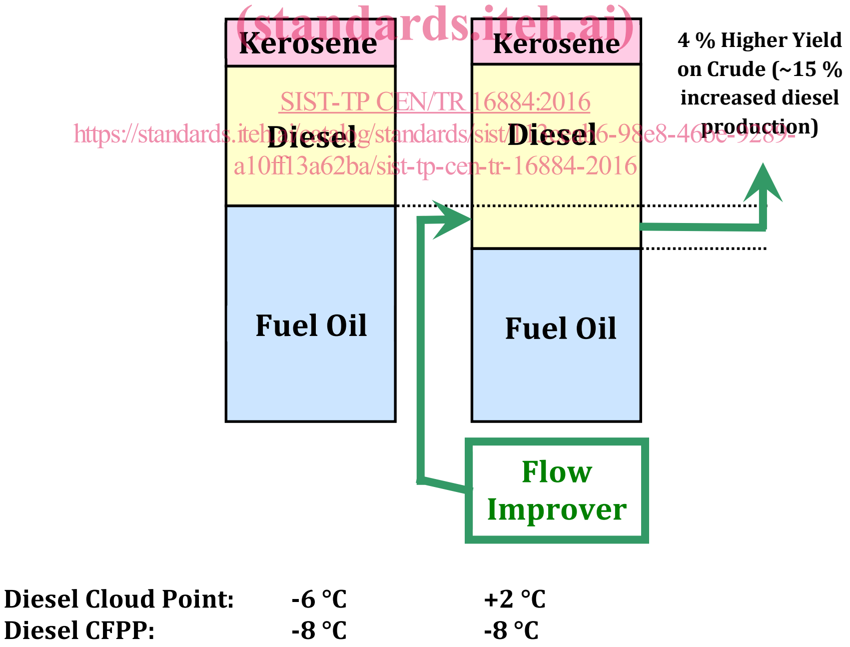


Figure 2 — Heavy gasoil upgrade (illustrative example) (source: Infineum)

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).

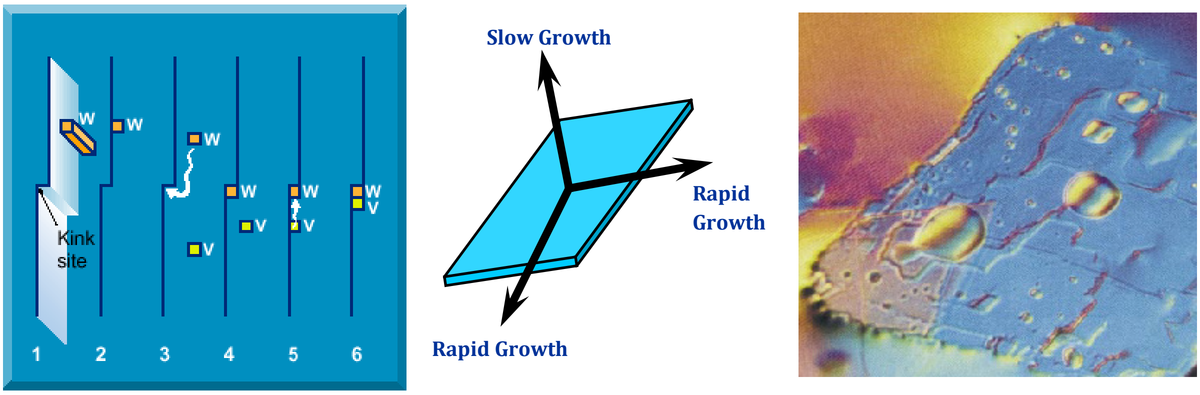


Figure 3 — Wax crystal growth (source: Infineum)

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).

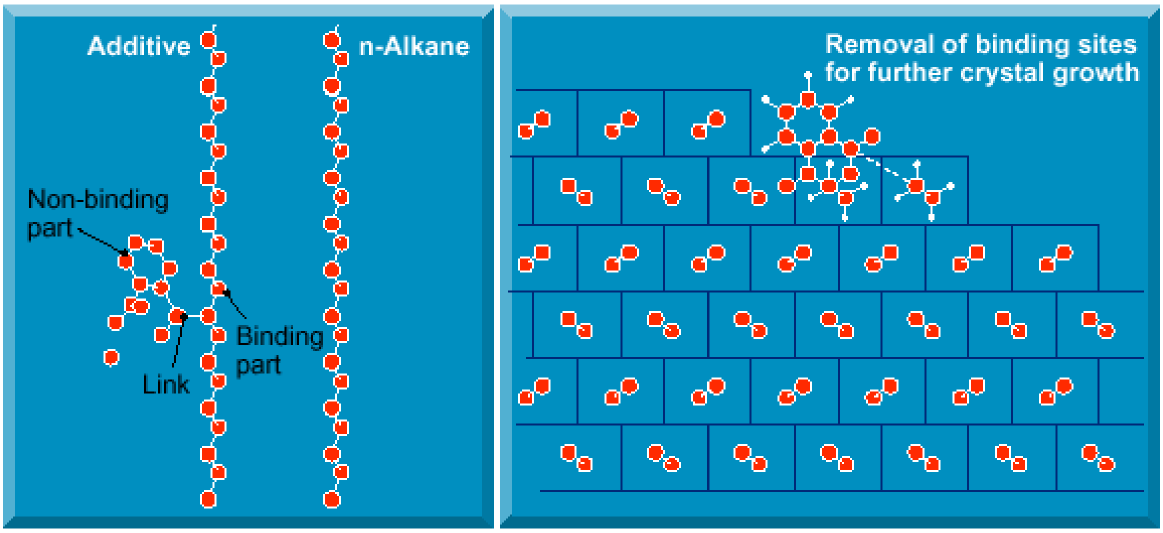


Figure 4 — Chemistry and binding nature of MDFIs (source: Infineum)

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).

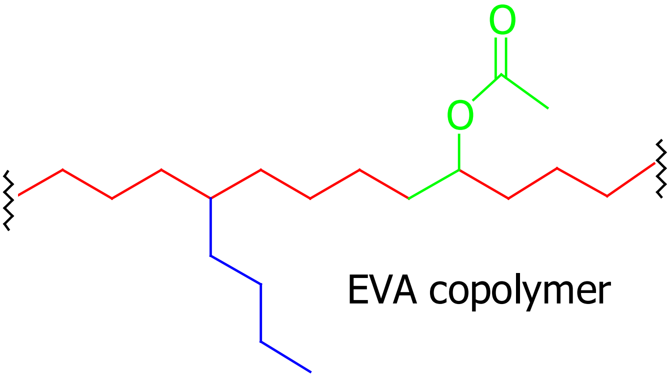


Figure 5 — EVA copolymer (source: Infineum)

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).

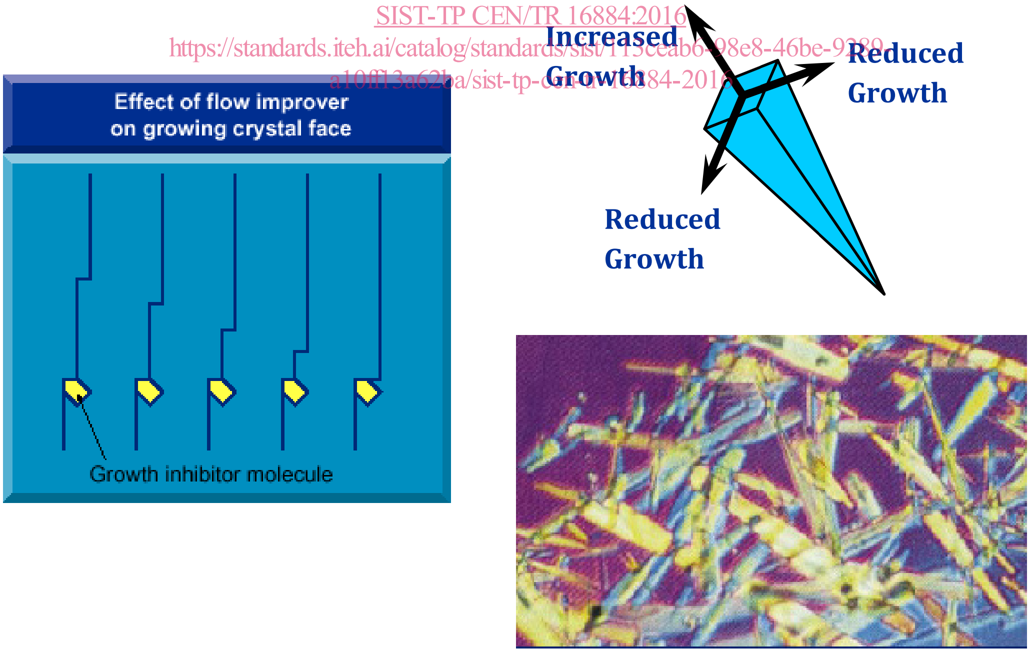
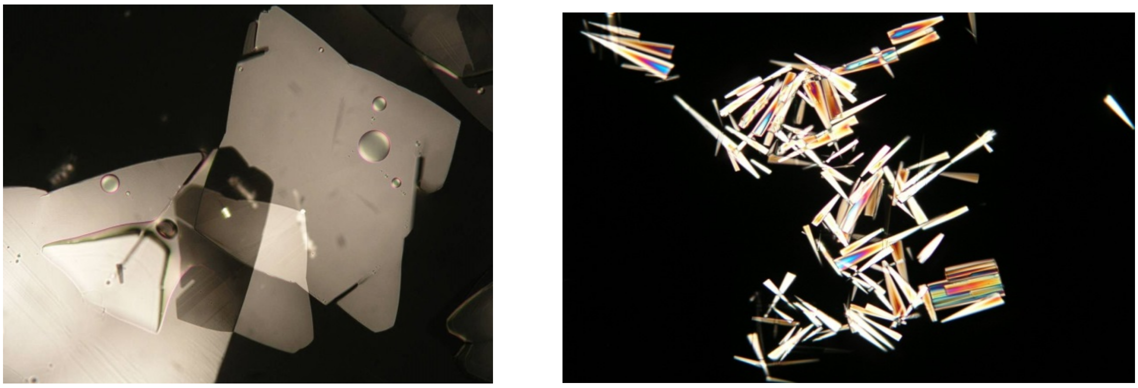


Figure 6 — Wax crystal growth modified by MDFI (source: Infineum)



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.

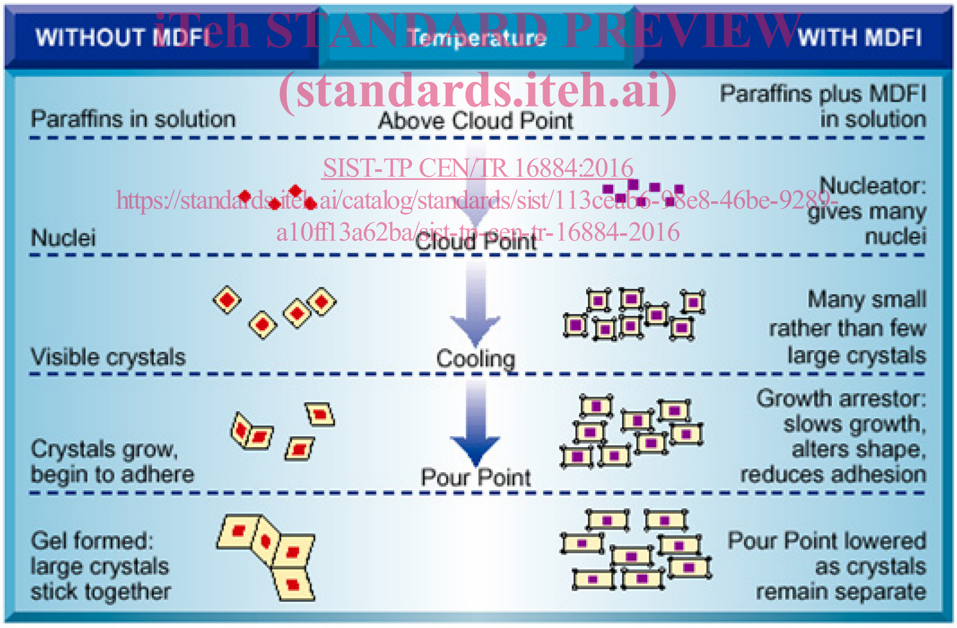


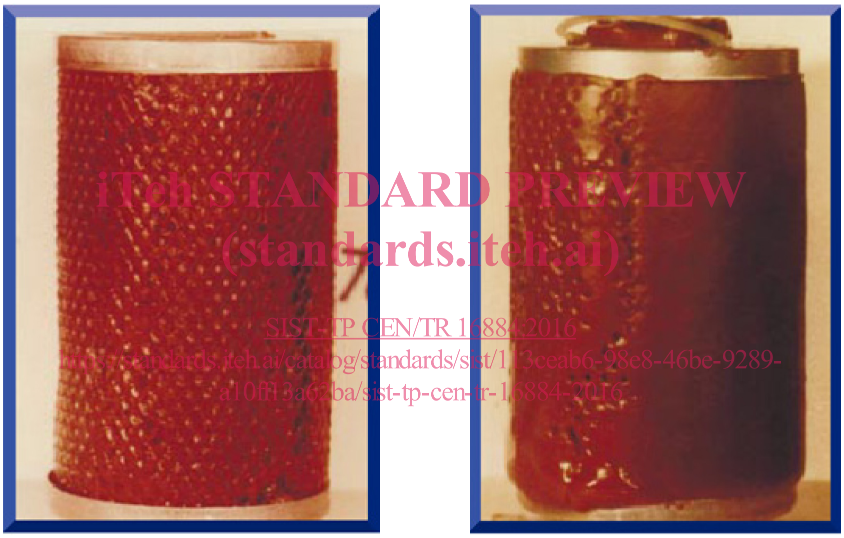
Figure 8 — Temperature dependence on wax crystal growth (source: Infineum)

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.

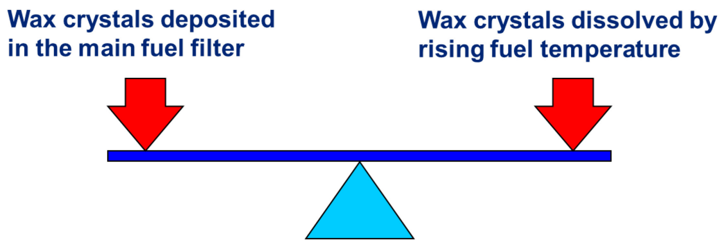


Figure 10 — The operability balance (source: Infineum)

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.

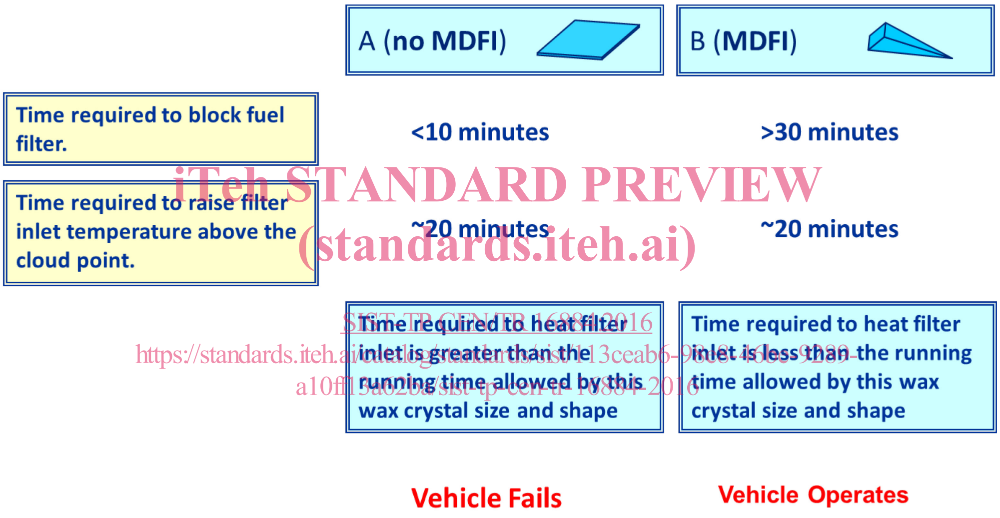


Figure 11 — Example of benefits of smaller wax crystals on vehicle operability (source: Infineum)

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).