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Dizelske mešanice in goriva - Vprašanja glede hladnega filtriranja

Diesel blends and fuels - Cold filterability issues

Dieselkraftstoffe und Mischungen - Kaltefiltrierbarkeit Problematiik

Combustibles et blends pour moteurs diesel (gazole) - Problems avec filtrabilité en temperatures bas

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This Technical Report was approved by CEN on 8 July 2016. It has been drawn up by the Technical Committee CEN/TC 19.

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European foreword

This document (CEN/TR 16982:2016) 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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At the plenary meeting in June 2015, CEN/TC 19 took Decision 45-2015 for new work under WG 24 to produce a Technical Report titled “CEN/TR Diesel blends - Cold filterability issues” with the scope to capture the key points raised in the presentations and discussions at the WG 24 Filter Blocking Workshop held on 1 June 2015. Consequently, this Technical Report documents the findings, interpretations and opinions of those involved in presenting the information, and these should not be considered as the opinion of WG 24.

Introduction

During recent winters, a wide range of vehicles has been affected in specific European countries and there is a possible link with FAME composition, base diesel quality, cold flow additives and oxidation stability effects. In order to solve these issues, some countries have introduced new additional requirements in their national fuel quality specifications or "best practice" market agreements:

- In the UK, a clear correlation between low temperatures and increased vehicle filter blocking was reported, with ambient temperatures below 3 °C thought to be critical. The introduction by fuel suppliers of a voluntary Filter Blocking Test limit of 2,52 in February 2014 seems to have improved the situation, but has not solved the problem.

- In Italy, ENI recommended that ASTM D2709 could be an alternative method for fast evaluation of contaminants in FAME. ENI also suggested, as an intermediate solution, a filtration step in refineries or terminals to improve FAME quality if needed. In ENI's experience, implementing this quality control "best practice" in Italy, in collaboration with their biofuel suppliers, has resulted in no further vehicle filter blocking incidents being reported in the last two years.

- In France, to solve the diesel fuel filter plugging when the decrease in temperature continues slowly over several days, the saturated methyl ester content in FAME was limited in winter to a maximum of 16 % (*m*/*m*) and in summer to a maximum of 30 % (*m*/*m*) in national law.

CEN/TC 19/WG 24 organized a workshop on the 1st of June 2015 in order to clarify the issue, to gather relevant data and to propose recommendations to CEN/TC 19 with respect to changes to the EN 590 (regular B7 diesel), EN 16734 (B10), EN 16709 (B20/B30) and EN 14214 (B100) standards to protect the market from filter blocking.

At the end of the workshop, it was agreed that a CEN Technical Report should be produced documenting the WG 24 Filter Blocking Workshop held on 01 June 2015 (i.e. this report). It therefore lays down the status-quo of the evidence on filter blocking issues in the European market at that point in time. It should be read as such and later information will still be valuable for CEN/TC 19 specification drafting.

1 Scope

This Technical Report provides the latest thinking described during a workshop on 1 June 2015 by national experts involved in the investigations, and proposes possible solutions to solve the diesel fuel filter plugging issues in these countries.

NOTE For the purposes of this Technical Report, the terms "% (*m/m*)" and "% (*V/V*)" are used to represent respectively the mass fraction, μ, and the volume fraction, φ.

2 Background to this Technical Report

A filter blocking workshop was organized by CEN/TC 19/WG 24 on 01 June 2015 in London in response to an increasing number of diesel vehicle filter blocking occurrences in several European countries (Italy, Sweden and the UK), particularly during the winter period. The purpose of the workshop was to share experiences and learnings from each affected country, identify common links and discuss possible solutions. The workshop also considered the development status of the various rig and laboratory tests designed to investigate and prevent low temperature filter blocking. The ultimate aim of the workshop was to make recommendations to WG 24 with respect to changes to the EN 590, EN 16734 (B10), EN 16709 (B30) and EN 14214 standards to protect the end user.

In his introductory comments, the WG 24 convenor advised that a wide range of vehicles is being affected in several European countries and that there is a possible link with FAME composition, base diesel quality, cold flow additives and oxidation stability effects. He also underlined the importance of ensuring that the CEN diesel fuel specifications are robust and protect the consumer.

The workshop included a number of technical presentations on the topics that are described in the Clauses 3 to 6 (order of presentation is followed). Publication of this Technical Report was one of the agreed actions from the workshop (see Clause 7).

3 Issues in specific European markets

3.1 UK experience

A summary of the diesel vehicle filter blocking trends in the UK over the past few years was provided. The monthly "baseline" level of diesel vehicle breakdowns due to filter blocking since 2009, as reported by the Automobile Association (AA), was around 200. However, during the past three winters, filter blocking breakdowns had risen to 5 times this level, with most of this increase occurring in the regions of Northeast England, Central Scotland, East Anglia and Southeast England.

A clear correlation between low temperatures and increased vehicle filter blocking was reported, with ambient temperatures below 3 °C thought to be critical. The introduction by fuel suppliers of a voluntary FBT limit of 2,52 for Bx diesel in February 2014 seemed to have improved the situation, but has not solved the problem. The UK experienced ~19 % reduction in the number of vehicle breakdowns due to filter blocking in winter 2014/15 compared to the previous winter, despite winter 2014/15 having many more cold nights below 0 °C in the most impacted regions (the minimum nightly temperature averaged for London, Glasgow and Middlesbrough was below 0 °C for 7 nights in winter 2013/14 compared to 30 nights in winter 2014/15).

Data from an extensive UK-wide retail diesel sampling program conducted by a major fuel retailer were presented. In addition, test results from a UK Department for Transport nationwide retail diesel sampling program undertaken between January 2015 to March 2015 were also presented. Data reported by region from both sampling programs included FBT (both ambient by procedure B and cold soak), Total Contamination, FAME content, saturated FAME content and particle counting. To provide a broader European context, FBT results from another retail diesel survey conducted across 8 European countries by a major international fuel retailer were shared.

A number of observations were made on the data from these fuel sampling programs:

a) Ambient FBT and Cold Soak FBT tests gave very similar results.

b) FBT results show several excursions above the UK voluntary limit of 2,52. In particular, the timing of one cluster of excursions corresponded with a higher number of vehicle failures due to filter blocking.

c) FBT results for diesel in other European countries are lower than in UK diesel (see Figure 1). Out of 111 samples, no FBT results were measured above 1,7 and most were below 1,1.

d) It was noted that the level of imported diesel fuel into the UK had increased considerably over the time period. It was also highlighted that base diesel fuels can have an impact on FBT results.

e) FAME content of UK diesel was fairly static over the period 2011 to 2015 at an average of 3 % (*V/V*) to 4 % (*V/V*). Regional differences exist with generally higher levels of FAME in Southeast England and East Anglia, and lower levels in Scotland. It was also noted that FAME blending levels were not the main trigger of occurrence of problems.

f) FAME used in the UK generally contains between (20 to 25) % (m/m) saturated FAME, however it is sometimes up to ~40 % (m/m)

g) The highest saturated FAME levels in EN 590 diesel were found in samples from Southeast England and East Anglia; whilst the lowest levels were measured in fuels from Scotland and the Midlands.

h) Particle count levels were high in some samples from the South East and East of England.

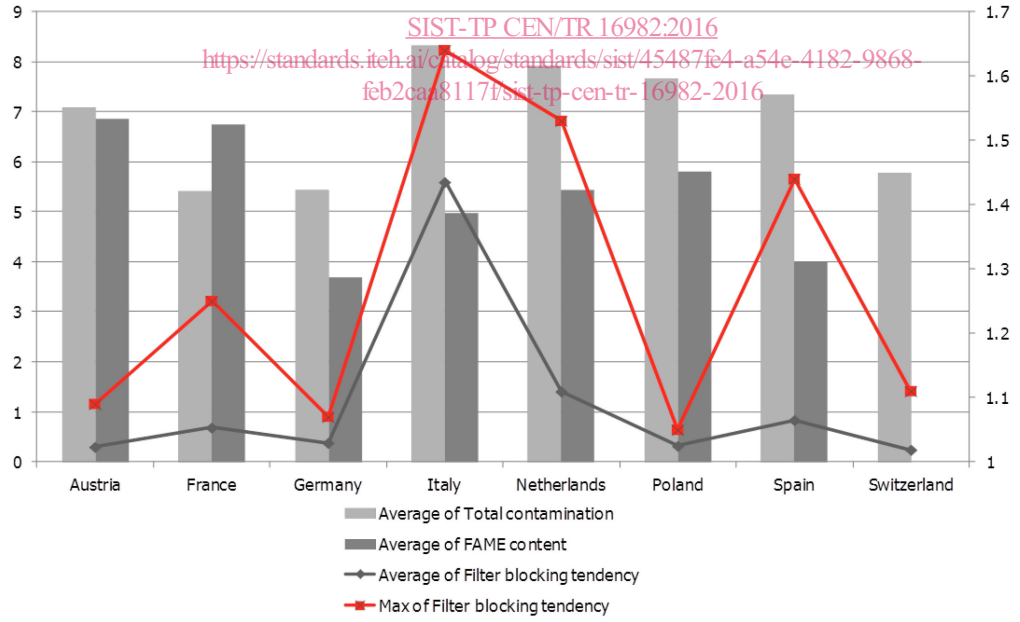

Figure 1 — Average and maximum FBT (right y-axis) by IP 387 Procedure B, average Total Contamination in mg/kg and average FAME content in % (V/V) (left y-axis) from 111 samples of diesel fuel collected from service stations in 8 European countries

The UK investigation also included analyses of deposits from blocked vehicle fuel filters and from the Infineum freezer rig. The analysis of residues from blocked vehicle filters in winter 2012/13 found the material to be mainly saturated mono-glycerides (SMG). In winter 2013/14, polyethylene, and in some

cases polyamide, was detected on blocked vehicle fuel filters. These components were also isolated from some of the fuel samples taken from the tanks of failed vehicles. Analysis of material recovered from blocked vehicle fuel filters in winter 2014/15 indicated high levels of ethylene vinyl acetate (EVA) which is one of the main ingredients in cold flow improver additives.

Filter deposits from fuel samples run through the Infineum freezer rig as part of the industry investigation were also isolated and analysed. Two sets of fuel from the same supply locations were tested – the first set was sampled in winter 2013/14 and the second set was sampled in winter 2014/15. When the rig test filters were analysed after testing three of the winter 2013/14 fuels, SMGs were detected, and in one fuel, there was also evidence of polyethylene and polyamide. Two of the rig test filters analysed after testing the winter 2014/15 fuels showed the presence of SMGs, but there were no indications of polyethylene or polyamide in any of these fuels. It was also reported, although not seen on any of the rig test filters, that Drag Reducing Additive (DRA) had been observed on some service station diesel filters.

In their concluding remarks, the UK experts stated that there has been no apparent correlation between vehicle filter blocking and the Total Contamination test. As this is a gravimetric test they felt that it may not protect the market from fuel with high organic particulate loading. They also commented that the high particle counts measured in some market diesel fuels were not reflected in the Total Contamination test results, but were reflected in the FBT results. It was concluded that, in their opinion, no correlation existed between FBT and Total Contamination. For these reasons, it was explained that the UK is considering the introduction of an FBT requirement with a maximum limit of 2,52 in the national annex of BS EN 590 to provide improved market protection, rather than introducing a lower Total Contamination test limit.

It was recognized by the UK experts that the ambient FBT or the existing Cold Soak FBT test might not fully protect the market from all the potential root causes, therefore they are continuing to investigate the possibility of a “Cold FBT” test in which the filtration step is conducted at a low temperature just above the cloud point of the fuel (i.e. somewhere in the range −3 °C to +3 °C). They also plan to continue with the freezer rig testing program at Infineum to identify the root cause(s) of the UK filter blocking issue and requested that other European countries support the work by providing representative market fuel samples.

3.2 Sweden

An overview of the recent diesel vehicle filter blocking problems in Sweden was provided from three of the key stakeholders investigating the issue: BIL Sweden (representing the OEMs), SPBI (Swedish Petroleum and Biofuels Institute) and Perstorp (Swedish FAME producer).

The BIL Sweden presentation started with a brief history of the issue. As is the case in the UK, this appears to be a winter problem in Sweden which only affects diesel cars. The problems started in winters 2011/12 and 2012/13 with some vehicles needing filter changes due to plugging. The situation worsened in winter 2013/14 with a number of OEMs starting to report increased filter changes on vehicles due to plugging. The problem was escalated to an industry issue which saw some initial discussions between BIL Sweden and SPBI during summer and autumn 2014. By winter 2014/15 there were more than 2 000 reported cases of fuel filter blocking with at least seven OEMs being affected. The filter change rate per 1 000 vehicles for some models has been higher than for corresponding models in the UK market (see Figure 2), although it has not been confirmed whether the design of the vehicle fuel systems are identical in both countries. In response, a “Deposit Investigation Task Force” was formed in spring 2015 by the Swedish Transport Agency, BIL Sweden and SPBI.

BIL Sweden shared some fuel analysis data which included FBT data for diesel samples collected from seven service stations that were linked to blockages of actual vehicles’ filters. Whilst there were no clear trends linking all the fuels analysed, attention was drawn to elevated SMG content in one of the fuels, elevated steryl glucoside content in two fuels, high FBT (Ambient and Cold Soak) in two fuels and low oxidation stability (Rancimat) in one of the fuels. It was noted that the high SMG content result of

74 mg/kg was unusual in the context of the low total monoglyceride content of 344 mg/kg, especially when the FAME had been identified as Rapeseed Methyl Ester (RME), which is highly unsaturated.

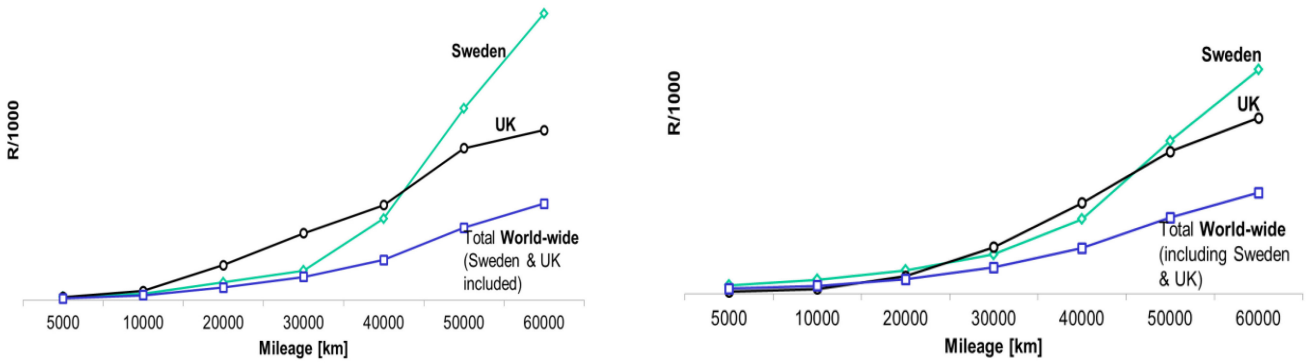

Figure 2 — Fuel filter replacement rate per 1 000 vehicles for one OEM (2 different model years)

In addition, blocked filter deposit analysis data generated by three OEMs was shared. In most cases, the filter deposits were dark and sticky. One of the OEMs reported that it seemed to be oxidized biodiesel. Attention was also drawn to the high levels of zinc and silicon measured on many of the filters. However, another OEM reported no indication of aged biodiesel and low levels of silicon and zinc in their filter deposits. When asked about where the zinc could be coming from, BIL Sweden responded that some of the impacted models use fuel tanks with zinc-containing coatings. However, it was also that no fuel injector deposits have been reported as might be expected in fuels containing elevated levels of zinc.

A common link between many of the filters analysed by the three OEMs was the high proportion of steryl glucoside deposits relative to SMG deposits. BIL Sweden felt that this was the opposite to the UK experience citing the example of one of the OEMs who had measured high SMG but low steryl glucoside deposits on a UK vehicle filter that was blocked in 2013.

In their concluding remarks, BIL Sweden stated that the root causes to the problem are not yet understood, however they offered a few of their own thoughts on what could be contributing to the problem:

— 2011/12 was the first winter when the filter plugging problems started to appear, and the problem has increased each year after that. This coincided with the first B7 diesel blends that were introduced onto the Swedish market in 2011. Over the next couple of years, some diesel containing 7 % (*V/V*) FAME + HVO up to 35 % (*V/V*) was put on the market. Therefore, BIL Sweden wondered whether a contributing factor to the filter blocking problem might be lower fuel solubility towards FAME impurities since Swedish MK1 diesel has worse solubility characteristics compared to typical EN 590 diesel and HVO reduces the solvency (aromaticity) of the fuel even further.

— Until recently there have been diesel fuels in the Swedish market that do not contain performance additives. In BIL Sweden's experience, a majority of the "problem fuels" seem to have been B7 containing no performance additives and sometimes containing HVO.

BIL Sweden called upon the workshop delegation for the urgent development of a reliable performance test on fuel filterability, which they would like CEN to include as a requirement in the EN 590 standard.

In their presentation, SPBI added to the information presented by BIL Sweden. It was noted in their opening remarks that the vehicles from all seven OEMs that are experiencing fuel filter blocking are equipped with suction fuel pumps and fuel filter heaters that activate when the temperature drops to approximately −3 °C. Placement of the main fuel filter on the suction side of the pump is known to make filters more sensitive to blocking.

It was explained that the FAME blended into diesel in Sweden is RME with a typical Cloud Point of between −4 °C to −5 °C. No used cooking oil or tallow based FAME is used. RME has the best cold properties of all commonly used FAME feedstocks. SPBI member companies also have to meet additional FAME quality requirements that are more stringent than EN 14214, such as lower water content and lower total monolgyceride content limits.

SPBI had observed three types (cases) of filter blocking in Sweden:

1) **Case 1**: filters from two vehicle brands blocked due to a black sludge deposit at approximately 0 °C. Interestingly, the filters did not block in these vehicles in the lower temperatures experienced in the far north of Sweden. When analysed, the black sludge typically contained oxidized FAME components, silicon (which could either originate from antifoam additive in the fuel or silica contained in the filter housing) and ethylene vinyl acetate (cold flow improver additive). High levels of zinc were also found in the deposit. Analyses of a new fuel filter housing of the type used in the vehicles affected by Case 1 failures showed that the surface is made from zinc and iron. The presence of zinc in diesel fuel is known to accelerate the degradation of FAME, which can contribute to filter blocking.

2) **Case 2**: fuel filter blocking reported on diesel cars with diesel particulate filters (DPF) that use a cerium / iron containing additive to facilitate the regeneration of the DPF. Under the control of the vehicle's ECU, the additive is automatically injected into the vehicle fuel tank each time the vehicle is refuelled. In the case of these blocked filters, cerium, iron and oxidation products from FAME were found on the fuel filter. SPBI conducted experiments to demonstrate the effect of the DPF additive on the fuel's oxidation stability. This showed that oxidation stability, as measured by the Rancimat and PetroOxy tests, reduced significantly with increasing DPF additive concentration (see Figures 3 and 4). The effect was far more significant for B7 than B0 diesel. SPBI commented that a frequent start-stop driving pattern seems to exacerbate the problem. It was noted, however, that this DPF additive is used extensively in other European countries markets (e.g. France) with no reported problems.

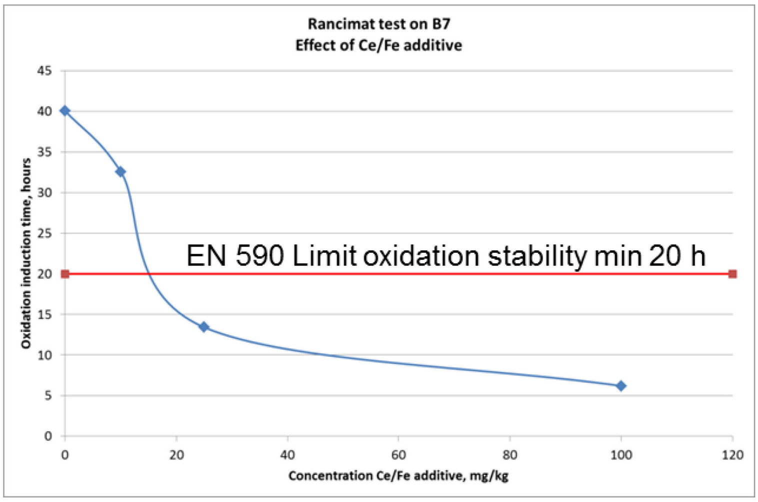

Figure 3 — Effect of DPF additive in B7 diesel on Rancimat induction period

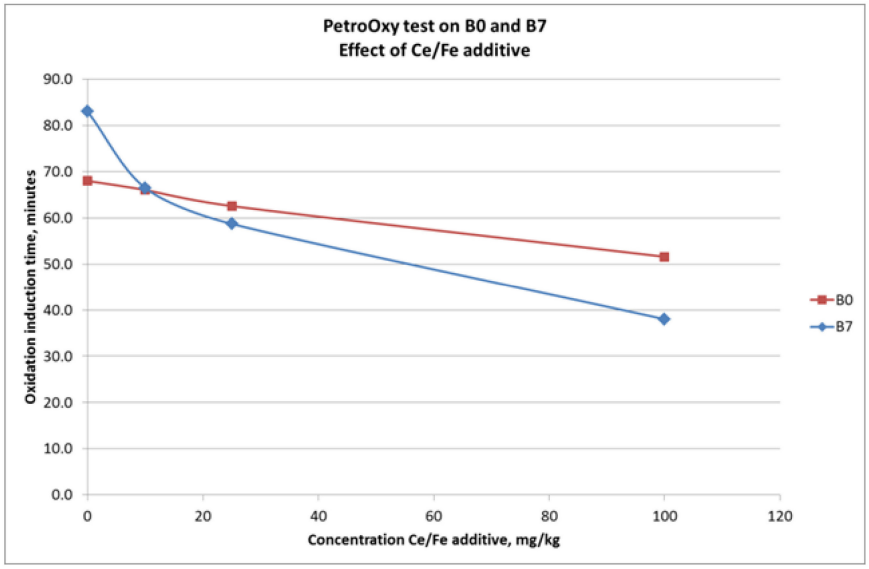

Figure 4 — Effect of DPF additive in B0 and B7 diesels on PetroOxy induction period

3) **Case 3**: other cases of filter blocking with limited information into possible causes.

Results of filterability tests on several lab blends of EN 590 and Swedish MK1 diesels with varying FAME contents were reported by SPBI. In this study, all FBT and Total Contamination test results were very low. SPBI also sampled a number of service stations in the areas surrounding three fuel distribution terminals. The fuels had FAME contents ranging between 5 % (*V/V*) and 7 % (*V/V*). The samples were tested for Cloud Point, FBT, Steel Corrosion and Rancimat. The oxidation stabilities of all the fuels were well above the EN 590 minimum Rancimat limit of 20 h. All FBT results were below 2 and most were very low. The reason for not introducing a voluntary FBT limit is due to that the final fuel for delivery to service stations are blended at the loading rack. The possibility to control the FBT of the final blend is very difficult given the large number of deliveries to service station..

In conclusion, SPBI called upon the CEN experts to investigate whether standard diesel detergent additive has an effect on degradation of FAME in the presence of metals, and to investigate the causes of filter blocking and propose possible solutions.

Perstorp presented slides on a series of experiments they have conducted on precipitates from fuel samples and analyses of blocked fuel filters. In one experiment, Perstorp prepared blends of distilled Tallow Methyl Ester (dTME) and Rapeseed Methyl Ester (RME) in Swedish MK1 at concentrations of 5 % (*V/V*), 7 % (*V/V*) and 10 % (*V/V*). The blends were stored in glass bottles for several days at −20 °C. After 6 days, all the dTME blends contained a white precipitate which was present in the largest concentration in the B10 blend. In contrast, none of the RME blends showed signs of any precipitates after 3 days (see Figure 5).

An experiment to evaluate the impact of antioxidant and cold flow improver additives on the precipitation of saturated monoglycerides (SMGs) in 7 % (*V/V*) blends of RME in Swedish MK1 was also performed by Perstorp. In this experiment, the fuel blends were stored at −20 °C for two months before shaking and cold filtering them under vacuum through a 1 µm glass fibre filter. The conclusion drawn from this experiment was that antioxidant and cold flow improver additives have little effect on SMGs.

Various studies conducted by Perstorp have shown a large number of different components contained in diesel fuel precipitates and isolated on test filters which may contribute to blocking vehicle filters. These included glycerol, steryl glucosides, saturated methyl esters, SMGs, lubricants, free sterols, polyethylene, ethylene-vinyl acetate copolymer, residues of synthetic polymers, citric acid, phosphoric