Deliverable report

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Executive Summary

The overall objective of REDIFUEL is to enable the utilization of various biomass feedstocks for an ultimate renewable EN590 diesel biofuel (drop-in capable at any ratio) in a sustainable manner. The proposed drop-in biofuel contains high-cetane C11+ bio-hydrocarbons and C6-C11 bio-alcohols.

This report describes the assessment of the diesel like biofuel Redifuel's interaction with engine oil. This task was approached via two steps. On one hand the impact of engine oil contaminations on the various fuel blends has been assessed. Especially, combustion and safety relevant properties have been examined. On the other hand, the effect of contaminations of Redifuel and various blends with diesel on engine oil have been examined. Here, lubrication and oil stability relevant properties have been considered. For both cases mentioned above, the interaction of engine oil with EN 590 compliant fossil diesel was considered as a reference [1].

The results of the assessment show that an engine oil contamination of up to 3 % of Redifuel and different blends with diesel fuel has no negative impact on the ignition process of the fuel. Additionally, engine oil contaminations do not pose a safety risk according to the measured fuel flashpoint.

Contaminations of the engine oil with Redifuel and different blends with diesel of up to 3 % do not negatively impact engine oil lubricity or stability.

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1 Introduction

This deliverable is part of work package 3 and related to task 3.2.3. The task's main objective is to investigate the interaction of REDIFUEL and its diesel blends with lubricant, namely engine oil. During operation of the engine, parts of the fuel can intrude the lubrication matrix causing loss of viscosity and lubricity of the engine oil leading to wear. Similarly, parts of the lubricant can dissolve into the fuel. This can lead to low-speed pre-ignition and engine knocking.

Both can lead to serious engine damage if the properties of the operational fluids are negatively impacted by each other. Therefore, two aspects of fuel-lubricant interaction are assessed:

- The impact of engine oil contaminations on the fuel
- The impact of fuel contaminations on the engine oil

The objectives of this task have been reached. Fuel as well as fuel blends and engine oil were contaminated with each other and the physical-chemical properties of both contaminated operational fluids have been analysed and discussed. This report provides a detailed description of the conducted work and the results.

2 Theoretical Background

During engine operation fuel and lubricant can intrude each other's matrix. This can happen for example at the piston-cylinder contact where both fuel and engine oil are present (**Figure 1**).

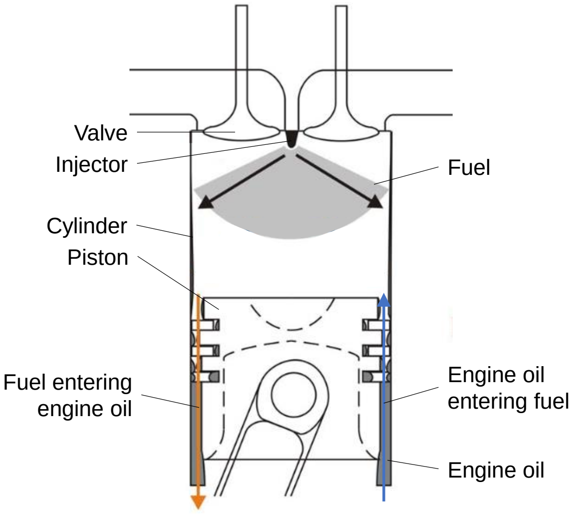

Figure 1 Fuel-Engine oil interaction at the piston-cylinder contact

The contamination of fuel by engine oil is referred to as oil dilution and can lead to problems during fuel combustion. One of the most prominent problems are low-speed pre-ignitions (LSPI). LSPI describes a premature ignition or combustion event before the ignition of fuel. It occurs especially at high load and low speeds and leads to unwanted cylinder pressure and engine knocking. Frequent LSPI occurrences can eventually lead to engine damages such as cracked pistons. [2] [3] [4]

The contamination of engine oil is also called fuel dilution. There are several causes for fuel dilution e.g., incomplete combustion due to improper fuel-air ratio. Problems caused by fuel dilution are the loss of viscosity of the engine oil and thus its lubricity as well as a loss of oil stability. These changes of oil properties eventually lead to engine wear and engine damage. [5] [6]

For these reasons the interaction of fuel and lubricant must be assessed to evaluate the drop-in capability of a new fuel. This is done by simulating the contamination of fuel and engine-oil and studying the relevant properties of both (e.g. safety, lubricity, performance etc.). As dilution rates of < 5 % can already lead to problems with conventional fuels and necessitate and oil change in the case of engine , contamination levels of 1 and 3 % engine oil in fuel and fuel in engine oil have been investigated [7] [8] [9]. This was the approach used in task 3.2.3 to study the interaction of REDIFUEL and its diesel blends with the conventional engine oil. The experimental methodology and analysis methods are detailed in the next chapter.

3 Material and Methods

This chapter details the experimental approach and the analysis methods used to evaluate the compatibility of REDIFUEL and its diesel blends with the conventional engine oils. REDIFUEL and its blends were contaminated with engine oil followed by an analysis of the relevant fuel properties. Similarly, the engine oil was contaminated with REDIFUEL, and its blends followed by a characterization of the former's properties.

The fuel blends investigated for this task include REDIFUEL and its blends with fossil diesel (**Table 1**), as proposed in task 3.1 (see deliverable 3.3). The lubricant used for the investigation was a high-performance engine oil of class 5W30 for utility vehicles (Shell Rimula R6LME 5W30), see **Figure 2**.

Table 1 Fuel blends which were investigated in this task

Blend	Composition in %		
	Diesel	REDIFUEL	UCOME
Diesel	100	-	-
60% Diesel + 40% RF_{A30P70}	60	40	-
50% Diesel + 50% RF_{A30P70}	50	50	-
46.5% Diesel + 46.5% RF_{A30P70} +7% UCOME	46.5	46.5	7
REDIFUEL RF_{A30P70}	-	100	-

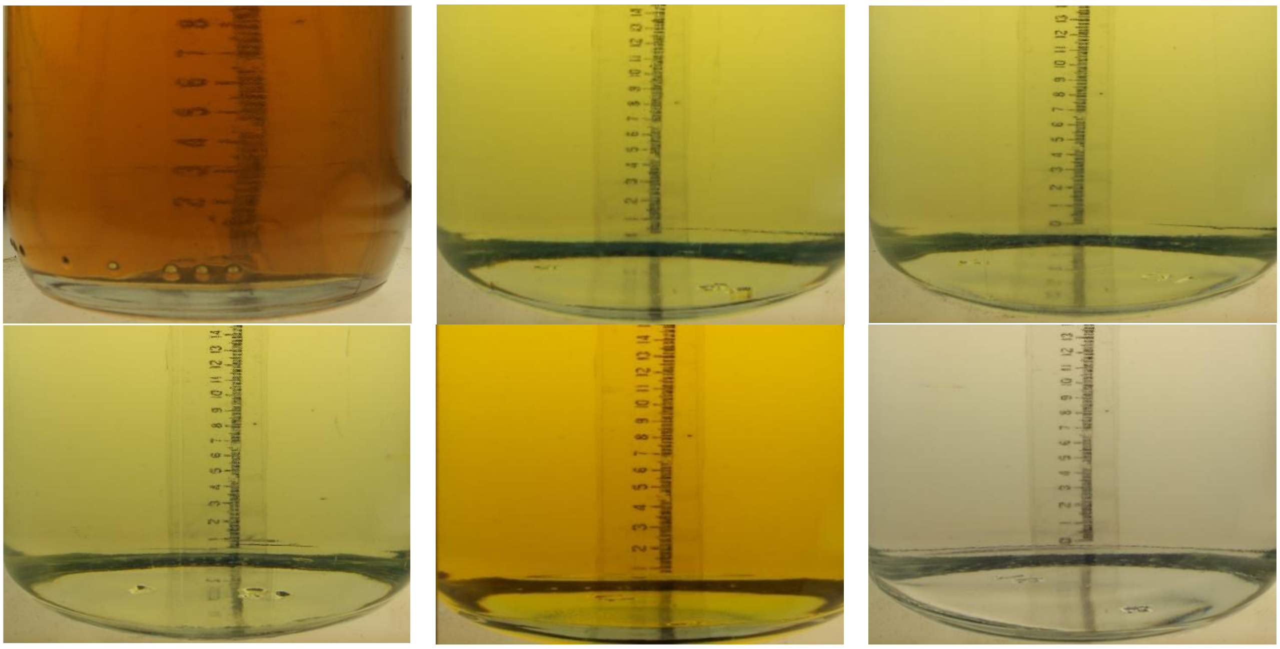

Figure 2 Top from left to right: Engine oil 5W30, Diesel, 60% Diesel + 40% RFA30P70, Bottom from left to right: 50% Diesel + 50% RFA30P70, 46.5% Diesel + 46.5% RFA30P70 +7% UCOME, REDIFUEL RFA30P70

3.1 SAMPLES AND METHODOLOGY OF THE ASSESSMENT OF THE IMPACT OF LUBRICANT ON FUEL

To investigate the impact of engine oil contaminations 1 % and 3 % engine oil were added to samples of the fuels. An overview of the samples is given in **Table 2**.

Table 2 Investigated samples to assess impact of lubricant on fuel

Blend	Contamination with engine oil in %		
	0	1	3
REDIFUEL RF_{A30P70}	x	x	x
60% Diesel + 40% RF_{A30P70}	x	x	x
50% Diesel + 50% RF_{A30P70}	x	x	x
46,5% Diesel + 46,5% RF_{A30P70} +7% UCOME	x	x	x
Diesel	x	x	x

The effect of the lubricant on the fuel was assessed via analysis of combustion relevant properties such as ignition delay and cetane number (DIN EN 17155), ignition temperature (DIN 51794) and the safety relevant parameter flashpoint (DIN EN ISO 2719) [10] [11].

The cetane number of a middle distillate is determined according to DIN EN 17155 by comparing the ignition behaviour of mixtures of primary reference fuels with a known cetane number under standardised operating conditions. A sample, which is automatically drawn from a sample flask, is pre-heated and injected (1000 bar) into a temperature- and pressure-controlled combustion chamber with a constant volume at the beginning of a combustion cycle. The chamber has previously been filled with compressed air (17.5 bar) of a certain quality and heated to a temperature of 580 °C. With each injection and the resulting combustion, there is a rapid increase in pressure in the chamber, which is detected by a dynamic pressure sensor. The test sequence is repeated 12 to 14 times so that an average value of the ignition delay times can be formed from the available data of the pressure curves. Based on the calculated ignition delay, the cetane number can be read from a previously determined calibration curve of the mixtures of primary reference fuels.

To determine the ignition temperature of flammable substances, individual ignition tests are carried out. For this purpose, a small amount of sample is placed in an open Erlenmeyer flask, which is heated in a furnace. It is observed whether ignition of the flammable substance occurs at the prevailing temperature. In a series of tests with varying temperature and sample quantity, a minimum temperature is determined at which ignition begins.

The flash point according to DIN EN ISO 2719 is determined by filling a sample into a closed vessel and heating it under constant stirring so that a constant temperature rise is achieved. An inflammation source is lowered through an opening in the vessel at regular intervals. The lowest temperature at which the vapour phase of the test batch is ignited is recorded as the flash point at absolute air pressure. This temperature is then corrected to standard pressure using a specified equation.

3.2 SAMPLES AND METHODOLOGY FOR THE ASSESSMENT OF THE IMPACT OF FUEL AND FUEL BLENDS ON LUBRICANT

For the assessment of the fuels’ impact on the lubricant engine oil, samples which contain 1 % and 3 % of fuel were prepared. An overview of the samples is given in **Table 3**.

Table 3 Investigated samples to assess impact of fuel on lubricant

Blend	Contamination of engine oil with fuel in %		
	0	1	3
none	x		
REDIFUEL RF_{A30P70}		x	x
60% Diesel + 40% RF_{A30P70}		x	x
50% Diesel + 50% RF_{A30P70}		x	x
46,5% Diesel + 46,5% RF_{A30P70} +7% UCOME		x	x
Diesel		x	x

The effect of the fuel on the lubricant was assessed via analysis of lubrication relevant properties such as viscosity (DIN EN 16896) and lubricity (DIN EN ISO 12146-1) as well as the parameters which determine oil stability, such as base number (DIN ISO 3771) and water content (DIN EN ISO 12937). [12] [13] [14] [15]. The results of the analysis of the contaminated fuels and engine oil are discussed in the next chapter.

4 Results and discussion

4.1 IMPACT OF LUBRICANT ON FUEL AND FUEL BLENDS

For the assessment of the impact of lubricant on the fuel, ignition relevant properties — ignition delay, cetane number and ignition temperature as well as the safety relevant property flashpoint have been examined. **Figure 3** shows the ignition delay of the pure and the contaminated fuel blends.

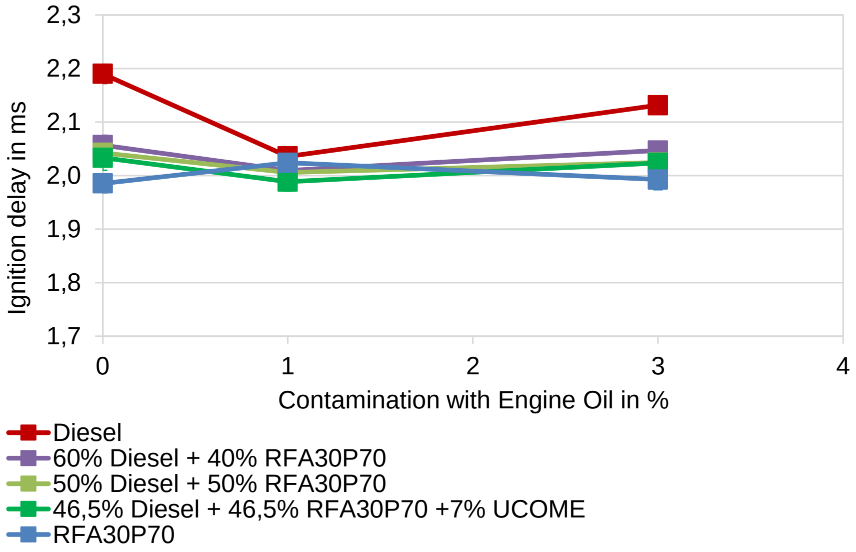

Figure 3 Ignition delay of fuels contaminated with engine oil

All measurement points are an average of at least 40 individual measurements. The standard deviations of all measurement points lie within the symbols of the measurement points. The figure shows that the ignition delay of diesel is higher than that of the fuel blends and Redifuel RF_{A30P70}. The engine oil contaminations have only little impact on the ignition delay which is nearly constant for all fuel blends over the investigated contamination range. The cetane number is calculated from the ignition delay via an empirical equation. The ignition delay itself is calculated from the average of two time periods. The first criterion is the time span from injection impulse to return to a pressure of 0 kPa. Due to the injection process and the temperature difference between the fuel and the air temperature in the combustion chamber, there is a brief underpressure. The second criterion is the reaching of a threshold value of 150 kPa. The mean value of these two time periods is the calculated ignition delay. With the help of this ignition delay time, the cetane number can be calculated on the basis of a calibration curve filed in the device.

The cetane numbers of the fuel blends and contaminated fuel blends are shown in **Figure 4**.

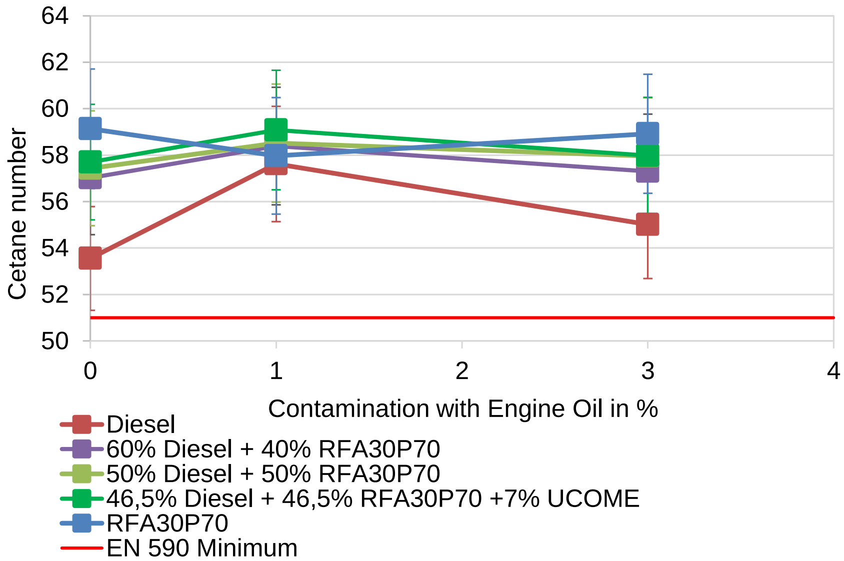

Figure 4 Cetane Number of fuels contaminated with engine oil

Redifuel RF_{A30P70} and the blends have a higher cetane number than diesel. This is caused by the paraffinic share of the fuels. The changes in cetane number of contaminated fuels lie within the comparability of the method for all the fuel blends. Thus, they are independent of the engine oil contamination in the investigated contamination range. The cetane numbers of all pure and contaminated fuel blends are higher than the EN 590 cetane number specification of minimum 51.

The results of the measurement of ignition temperature are presented in **Figure 5**.

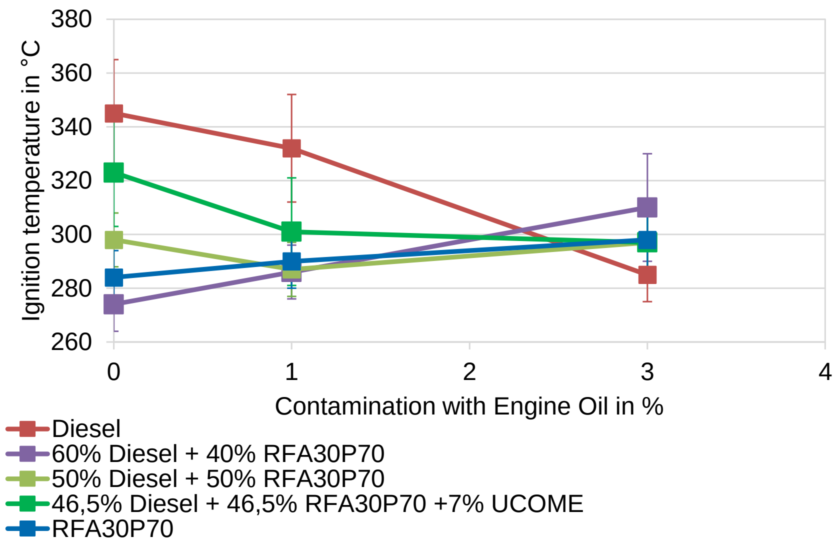

Figure 5 Ignition temperature of fuels contaminated with engine oil

Diesel fuel has a higher ignition temperature than Redifuel RF_{A30P70} and the blends of both. It is found that the ignition temperature of diesel decreases with increasing engine oil contamination. This trend cannot be observed for Redifuel RF_{A30P70} and its blends with diesel. Here, the changes of ignition temperature in the examined contamination range with engine oil lie within the comparability of the method. Therefore, they can be interpreted as constant.

Summarizing, relevant contamination levels of engine oil in Redifuel RFA30P70 and its diesel blends have no impact on ignition relevant properties. Most probably no problems concerning ignition timing and self-ignition will occur before combustion.

Furthermore, the flashpoint of contaminated fuels has been tested, to assess the risk of the development of a flammable fuel-air mixture, if fuel is directed back to the fuel tank instead of being combusted.

The results of flashpoint measurement are shown in **Figure 6**.

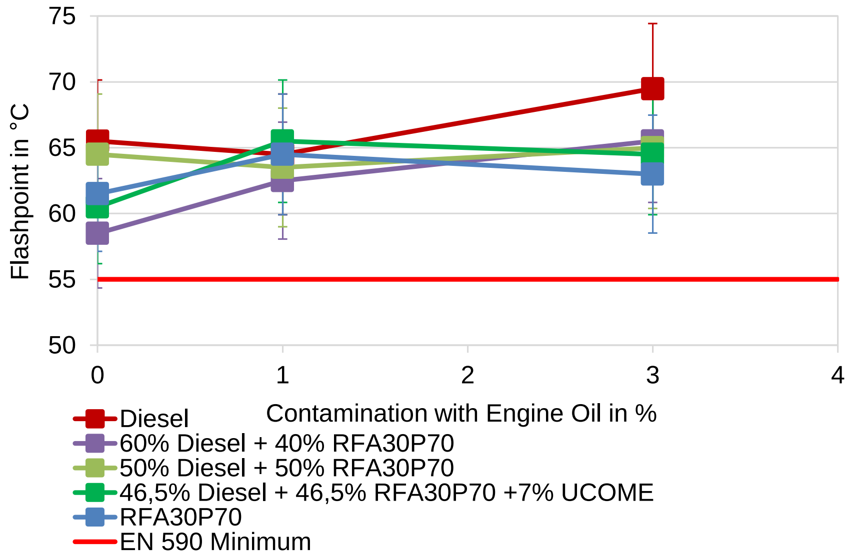

Figure 6 Flashpoint of fuels contaminated with engine oil

Compared to diesel and RFA30P70, the flashpoint of engine oil is significantly higher at 232 °C. The flashpoint differences between pure diesel fuel, Redifuel RF_{A30P70} and their blends lie within the comparability of the method. Also, the changes of flashpoint of the individual fuels and fuel blends with increasing engine oil contamination lie within the comparability of the method within the examined contamination range. All measured flashpoints lie above the EN 590 minimum limit of 55 °C.

Since the contamination even reduces significantly further, when a small amount of fuel is redirected into the fuel tank instead of being combusted, no safety risk is expected. All diesel fuel tanks are designed for fuels with a minimum flashpoint of 55 °C.

4.2 IMPACT OF FUEL AND FUEL BLENDS ON LUBRICANT

To assess the impact of fuel on the lubricant firstly the lubrication relevant properties viscosity and lubricity of the pure and contaminated engine oil were examined. Further, the base number as well as the water content were examined to assess the impact of fuel contaminations on the oil stability.

Figure 7 shows the viscosity of the pure and contaminated engine oil.

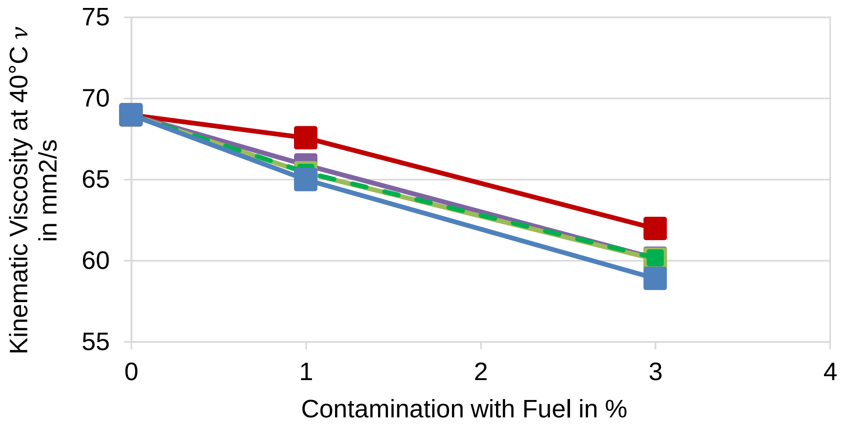

Figure 7 Viscosity of engine oil contaminated with different fuel blends

The viscosity of the engine oil decreases significantly with increasing fuel contamination for all fuel blends. This is since all the fuels' and fuel blends' viscosities are about 3 mm²/s and are significantly lower compared to that of the engine oil, which is about 69 mm²/s.

	Kin. Viscosity ν at 40°C in mm²/s	**Contamination with 1% fuel**	**Contamination with 3% fuel**
Engine Oil	68,973		
Diesel	3,2866		
RFA30P70	3,0488		
Engine Oil + Diesel	68,973	67,562	61,972
Engine Oil + 60% Diesel + 40% RFA30P07	68,973	65,894	60,135
Engine Oil + 50% Diesel + 50% RFA30P07	68,973	65,413	60,083
Engine Oil + 46,5% Diesel + 46,5% RFA30P07 + 7% UCOME	68,973	65,443	60,175
Engine Oil + RFA30P70	68,973	64,983	58,896

Even though the trend is the same, the rate of decrease is sharper increasing Redifuel RF_{A30P70} share in the fuel blend. As an engine oil's viscosity is a key factor for the strength or thickness of the lubrication film, it has to be assessed how the decreasing lubrication impacts the contaminated engine oil's lubricity. The lubricity of the pure and contaminated engine oils is shown in **Figure 8**.

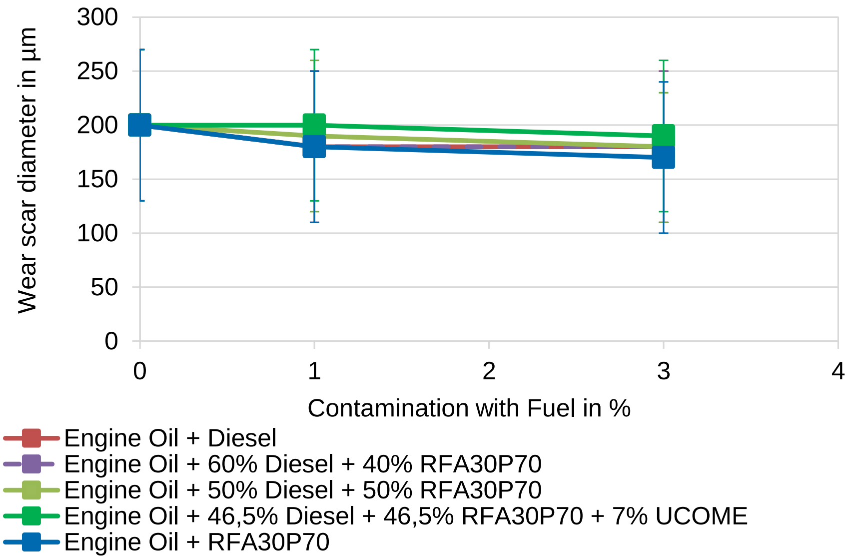

Figure 8 Lubrication behaviour (wear scar diameter) of engine oil contaminated with different fuel blends

The wear scar diameter is assessed. The wear scar diameter of the engine oil is nearly constant for all contaminations with different fuel blends over the assessed contamination range. All changes lie within the comparability of the method. That means that the wear does not increase. Thus, the lubricity shows no relevant deterioration. The reason for this effect could be the addition of lubricity improvers in the lubricant. This is also indicated in the data sheet of the engine oil [1]

Figure 9 shows the base number of the pure and contaminated engine oils.

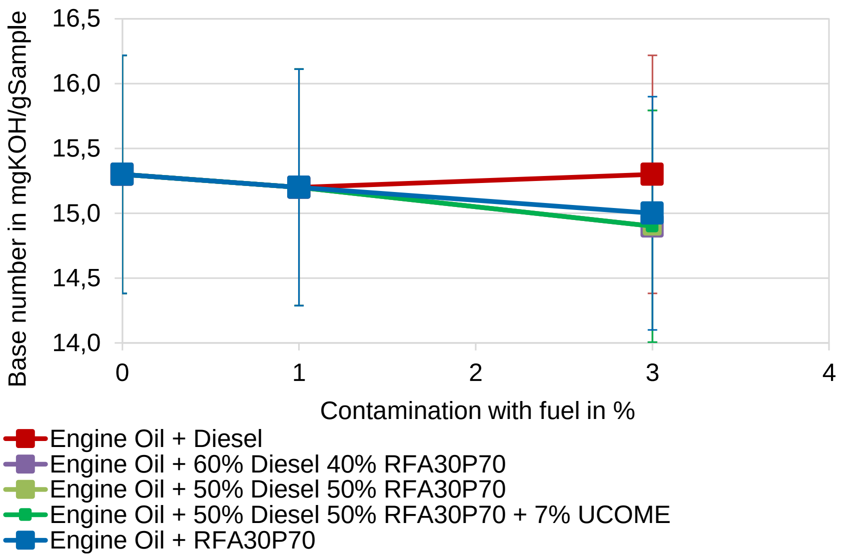

Figure 9 Base Number of engine oil contaminated with different fuel blends

The base number also is nearly constant over the assessed range of fuel contaminations for all fuel blends. All the changes lie within the comparability of the method.

Further, the water content of the pure and contaminated engine oils was examined. The results are shown in **Figure 10**.

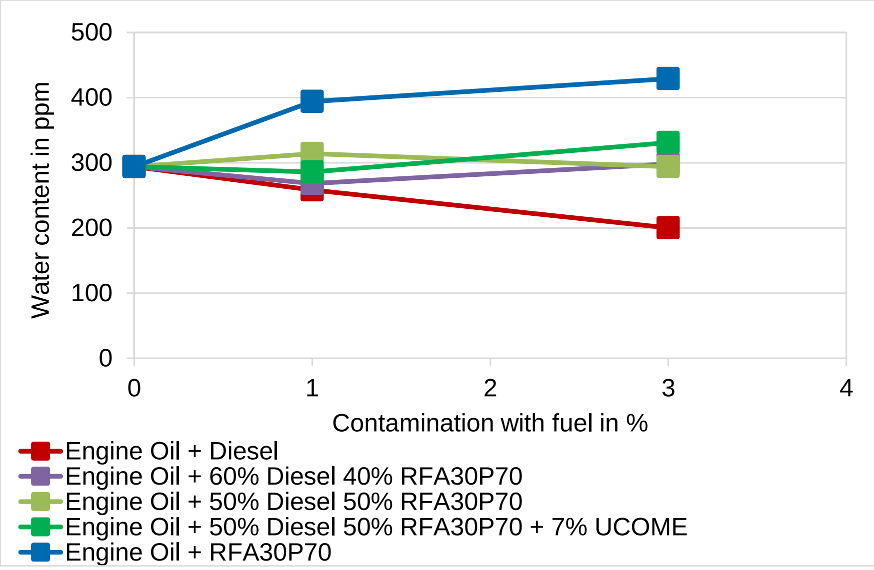

Figure 10 Water content of engine oil contaminated with different fuel blends

The water content of engine oil decreases with increasing contaminations with diesel. In contrary to that the water content increases with increasing share of Redifuel RF_{A30P70}. The water content of engine oil contaminated with blends of diesel fuel and Redifuel RF_{A30P70} is nearly constant. These effects might be caused by the non-polar nature of diesel and the polar nature of the alcohols contained in Redifuel RF_{A30P70}. In case of pure Redifuel RF_{A30P70} the water content significantly increased till 1% contamination, but above this the increase of the water content is much lower. The increased water content of the engine oil should not pose a problem for engine operation. During long drives of heavy-duty vehicles, the engine oil reaches temperatures between 80 °C and 120 °C. Thus, the additional water will most probably evaporate from the engine oil.

	Water content in ppm	Contamination with 1% fuel	Contamination with 3% fuel
Engine Oil + Diesel	294,0	258,0	200,0
Engine Oil + 60% Diesel + 40% RFA30P07	294,0	268,0	298,0
Engine Oil + 50% Diesel + 50% RFA30P07	294,0	314,0	294,0
Engine Oil + 46.5% Diesel + 46.5% RFA30P07 + 7% UCOME	294,0	286,0	331,0
Engine Oil + RFA30P70	294,0	394,0	429,0

5 Conclusions

The investigation of the interaction of fuel and engine oil showed promising results. The physical-chemical analysis revealed no negative impacts of the operational fluids on each other concerning the investigated properties and in the investigated contamination range.

The impacts of engine oil contaminations on fuel are summarized in **Table 4**.

Table 4 Impact of engine oil contaminations on fuel (0 = Impact comparable to diesel, + = impact smaller than on diesel or more positive impact, - = impact greater than on diesel or more negative impact)

Property	Diesel (reference)	60% Diesel+ 40% RF_{A30P70}	50% Diesel + 50% RF_{A30P70}	46,5% Diesel+ 46,5% RF_{A30P70} + 7% UCOME	REDIFUEL RF_{A30P70}
Ignition delay	0	0	0	0	0
Cetane number	0	0	0	0	0
Ignition temperature	0	+	+	+	+
Flashpoint	0	0	0	0	0

Engine oil contaminations of up to 3 % resulted in comparable impact on the novel fuel's and fuel blends' properties and diesel fuel properties. The ignition relevant properties such as ignition delay, cetane number and ignition temperature of Redifuel and its blends showed even less dependence on engine oil contaminations than diesel. Because of these results no impacts of small engine oil contaminations of up to 3 % on the ignition are expected. The flashpoints of all pure and contaminated fuels showed no significant dependence on engine oil contamination and are above the EN 590 limit. Therefore, conventional safety measures which are applied to diesel fuel with regards to flashpoint are also sufficient for Redifuel and its blends with diesel.

The impacts of fuel contaminations on engine oil are summarized in **Table 5**.

Table 5 Impact of fuel contaminations on engine oil (0 = Impact comparable to diesel, + = impact smaller than on diesel or more positive impact, - = impact greater than on diesel or more negative impact)

Property	Diesel (reference)	60% Diesel+ 40% RF_{A30P70}	50% Diesel + 50% RF_{A30P70}	46,5% Diesel+ 46,5% RF_{A30P70} + 7% UCOME	REDIFUEL RF_{A30P70}
Viscosity	0	0	0	0	0
Lubricity	0	0	0	0	0
Base number	0	0	0	0	0
Water content	0	-	-	-	-

With respect to the impact of fuel on the engine oil it can be observed, that Redifuel and the blends have the same effect on the engine oil's viscosity and lubricity as diesel. All fuels negatively impact the engine oil's viscosity which is related to the thickness of the engine oil lubricity gap. This loss of viscosity did not result in increased wear meaning that the lubricity itself was not impacted in the investigated contamination range. While the base number of the engine oil is also not impacted by fuel contaminations of up to 3 %, the water content is impacted. Even though the water content increases with increasing Redifuel contamination, it is not expected to cause any problems. In the usually long operation times of heavy-duty engines this water will evaporate from the engine oil when it reaches its operating temperature which can be as high as 120 C.

6 Deviations from Annex 1

There are no deviations with respect to the description of work.

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8 Acknowledgement

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