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Impact of Biodiesel on Fuel System Component Durability

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DISCLAIMER

1. INTRODUCTION

Biodiesel is defined as the mono alkyl esters of long chain fatty acids derived from vegetable oils or animal fats. The term Fatty Acid Methyl Ester (FAME) is often used as a generic expression for the trans-esters of these naturally occurring triglycerides which find application as either a replacement for, or a blending component for use with fossil derived diesel. There is great potential for significant variation in product quality and specification for products generically described as biodiesel. Research has shown that there are fuel quality, handling, storage and vehicle operability requirements which need to be addressed where biodiesel is used in the automotive diesel vehicle fleet. This has led to numerous initiatives by fuel producers, Original Equipment Manufacturers (OEMs) and their Industry Associations to publicise these issues and to lobby fuel legislators to include a number of parameters in regional biodiesel specifications.

In the United States, this has led to the development of a specification for biodiesel, ASTM D6751-03. This standard is intended to address the quality of pure biodiesel (termed B100) when used as a blend stock of 20% and lower. No separate ASTM quality specifications currently exist for biodiesel when blended with fossil-derived fuels at 5% or 20% levels. Biodiesel blends are generally referred to as BX, where X is a number which denotes the volume percent of biodiesel incorporated in the finished fuel. The ASTM D6751 standard specifies biodiesel as long chain fatty acid esters from vegetable or animal fats containing only one alcohol molecule on one ester linkage. This effectively excludes raw or unrefined vegetable oils which contain three ester linkages. Within the United States, soybean oil is the leading source biodiesel feedstock.

Within the European Union, rapeseed oil is the most widely available biodiesel feedstock. The European Committee for Standardisation developed a uniform standard, EN 14214, to replace the respective national standards. Modifications have been made to the European diesel specification (EN590) which allows for the use of up to 5% of an EN14214 FAME as long as the finished blend meets the cold flow properties specified for the geographic area where the fuel is to be used. It is worth noting that EN 14214 generally excludes pure soybean methyl esters as fuel due to the high natural iodine values of this product.

The compatibility of seal and hose materials commonly encountered in automotive fuel systems using conventional hydrocarbon fuels has long been established and elastomer manufacturers are able to make recommendations for their use. Studies and in-use experience has shown that certain seals, gaskets, hoses, etc may degrade under certain operating conditions in contact with B100. However, the effect of biodiesel when blended with hydrocarbon fuels has not been well researched. Most elastomer materials will undergo a physical or chemical change when in contact with fuel. The degree of change depends upon the tendency of the material to absorb a fuel or on compounds being dissolved or extracted by the fuel. This can lead to a number of changes in the physical characteristic of the material including swelling, shrinkage, embrittlement and changes in tensile properties. The limit of a permissible physical change varies with the application and some degree of change can usually be tolerated. For example, a material that swells in a fuel or suffers a decrease in hardness may well continue to be fit for purpose for a long time as a static seal. However, in dynamic applications, swelling may result in increased friction and wear and so a lower degree of volume change can be tolerated.

Efforts to produce low and ultra low sulphur fuels by hydro-treatment have resulted in the removal of polar nitrogen and oxygen compounds and hence reduced the good lubricating properties of diesel fuel. Several investigations have reported that biodiesel can significantly improve the lubricity of low sulphur diesel fuel at relatively low concentrations. In addition, it has been reported that the use of biodiesel can reduce the concentration of metal wear particles and increase engine life span. However, it has also been reported that the formation of corrosive materials (such as organic acids, water and methanol), polymers, gum formation, thermal and oxidative instability and water separation may give rise to vehicle operability problems. The following vehicle operability problems have been identified during extensive field trials as being caused by these fuel characteristics:

- Corrosion of Fuel Injection Equipment (FIE) components
- Elastomeric seal failures
- Low pressure fuel system blockage
- Fuel injector spray hole blockage
- Increased dilution and polymerisation of engine sump oil
- Pump seizures due to high fuel viscosity at low temperatures
- Increased injection pressure

As a consequence, OEM’s and their Industry Associations have been cautious in their acceptance of biodiesel and biodiesel blends.

2. EXECUTIVE SUMMARY

A number of known performance concerns have the potential to influence the perception and acceptance of fatty acid methyl esters as alternative fuels for use in the automotive diesel vehicle fleet. In particular, the oxidation of biodiesels and biodiesel blends is known to form deposits and corrosive materials which may impact on vehicle operability performance. The levels of oxidation which can be tolerated in-use and the potential severity of oxidation problems have not been well quantified.

In this Coordinating Research Council Project No. AVFL-2a, we report on selected biodiesels which are used to prepare test blends at two concentrations typically used in the United States and Europe. Test blends were prepared from rapeseed methyl ester (RME), soy methyl ester (SME) and soy methyl ester which has been oxidised under controlled conditions. We examine and quantify the effects of test blends on fuel system component wear, including fuel pump and fuel injectors, and on the physical characteristics of elastomer materials which are commonly found in automotive fuel systems.

As a first step in this study, it was necessary to establish the product qualities of the diesel base fuel and biodiesels nominated for the work programme. These chemical and physical properties were compared against the relevant National Specification of ASTM D6751 and BS EN 14214. All fuels were found to meet the requirements of the Standards.

The soy-based biodiesel was then oxidised under specified conditions to a minimum level of oxidation as determined from the acid number. To meet the fuel requirements of the study, two batches of soy biodiesel (batch 1 and 2) were prepared separately resulting in two different levels of oxidation. Both fuels failed to fulfill the requirements of the standard. The oxidised soy biodiesel used to prepare the B20 blends was oxidised to a much greater extent than is likely to be encountered in actual commercial use. This was intended to serve as a worst case example, but in fact may have been so highly oxidised as to be unrealistic. The subsequent separation of this heavily oxidised B20 blend into two phases suggested that this might be because of the high degree of oxidation. Analytical work was undertaken by the Associated Octel and the National Renewable Energy Laboratory (NREL) to investigate the physical and chemical characteristics of the separated oxidised fuel blend.

The separation of the highly oxidised biodiesel blend prompted additional investigation. One additional fuel blend was formulated to determine the behaviour and pump wear effects of a B20 blend prepared from a less highly oxidised soy biodiesel. Limited analysis was conducted to measure fuel properties and one further common rail pump test was performed to determine pump wear effects of this biodiesel fuel blend.

Test blends prepared for this study using US 2-D grade ultra low sulphur diesel were as follows:

- 5% v/v RME (B5)
- 5% v/v SME (B5)
- 5% v/v SME (B5) oxidised
- 20% v/v RME (B20)
- 20% v/v SME (B20)
- 20% v/v SME (B20) oxidised (batch 1)
- 20% v/v SME (B20) oxidised (batch 2)

There are currently no ASTM quality specifications for B5 and B20 blends although the European EN590 specification permits up to 5% biodiesel in pump fuel provided that the biodiesel component meets the EN 14214 specification. Test blends were therefore analysed to establish the product qualities according to ASTM D975. The fuel blends met the requirement of the standard with the exception of the B20 oxidised fuels. In addition, it was found that standard test method peroxide value (ASTM D2340) may not be appropriate for the analysis of the blended fuels which have suffered oxidation. Further investigation is recommended.

In this study, the physical properties of five candidate elastomers commonly used in automotive fuel systems were examined before and after immersion in the six test blends and base fuel under controlled conditions. Properties such as hardness, tensile strength, volume change and compression set properties were compared against air conditioned samples. Of the five materials tested, two elastomers emerged as being most compatible with the test blends. However, some interpretation is necessary to evaluate the performance of elastomers during these tests and consideration must be made to engineering applications.

Injector wear tests were conducted on the base fuel and three B20 biodiesel blends using a 500 hour test method developed by the Associated Octel. Detailed metrology measurements were taken on fuel injector sets before and after testing to quantify injector wear. This study has concluded that there was no increase in 'out-of-roundness' of the injector needles for any of the test fuels and hence the lubricity values of the test fuels were adequate for the protection of the injector components running under conditions similar to the test method. The injector wear test on the B20 oxidised soy biodiesel was terminated after only twelve hours of running because of separation of the fuel into two phases.

Fuel pump wear tests were conducted using a 500 hour test method based on the CEC F-32-X-99 test procedure. Bosch VE in-line pumps were evaluated for wear before and after testing on the base fuel, B5 and B20 test fuels. The rating measurements from the pump lubricity tests concluded that the fuels were within the range which would normally be expected for commercial automotive diesel fuels. The rotary pump test conducted on the B20 blend of oxidised soy biodiesel failed to reach full duration due to blockage of the fuel filter. This was found to be due to separation of the fuel blend into two phases.

To conclude this AVFL-2a study, a common rail test rig was built to mimic the real life operation of a common rail passenger car system. A test procedure was developed to accelerate wear conditions on common rail pumps using a 500 hour protocol. Pumps from tests conducted on the base fuel, B5 and B20 fuels and one additional B20 test fuel were examined and the components visually rated for abrasion, fretting, corrosion and wear and polishing steps.

Examination of the pump following the test on the B20 blend containing highly oxidised soy biodiesel revealed a hard lacquer accumulation on one of the pump components and significant seal swell. This candidate fuel concluded without operational failure but significant fuel separation was noted. A further common rail test on a less highly oxidised B20 blend was concluded without operational failure and without evidence of fuel separation or signs of lacquering. Swelling of one of the seals in the test pump was again noted.

No unusual wear was found on any of the common rail test pumps used in the study. The rating measurements from the common rail pump lubricity tests concluded that the fuels were within the range which would normally be expected for commercial automotive diesel fuels.

The evidence indicates that phase separation of B20 blends prepared from oxidised biodiesel did not occur in storage. Decomposition reactions occurring under the conditions of test probably accelerated fuel separation in the more highly oxidised B20 test fuel containing very high levels of water and sediment. Phase separation did not take place in the B20 fuel containing biodiesel oxidised to a lesser extent and which contained lower water and sediment. The main factor therefore affecting the different behaviour of the oxidised fuels is thought to be the extent of oxidation.

3. PROJECT OBJECTIVES

The objectives of this study can be defined as follows:

- To quantify the impact of biodiesel blended with fossil fuel on fuel system component wear including fuel pumps and fuel injectors.

- To quantify the effects of biodiesel blended with fossil fuel on the performance of elastomers which might typically be encountered in modern automotive fuel systems.

- To quantify the impact of oxidised biodiesel blended with fossil fuel on fuel system component wear including fuel pumps, injectors and elastomers.

4. METHODOLOGY

4.1 Fuel Preparation and Analysis

4.1.1 Base fuel

A quantity of 2-D grade ultra low sulphur diesel, BP-15 was shipped from the United States to England and used throughout the study as the base fuel and for blending. This fuel was reported to be from a single batch, intended to be similar to what might be expected of on-road use in the United States in 2006. Analysis of the base fuel was conducted to ensure the quality of the fuel according to the standard method, ASTM D975.

All fuels were stored in 200 litre, mild steel drums which were stoppered and inverted to prevent ingress of water.

4.1.2 Soy based biodiesel (SME)

Soybean oil derived biodiesel (labelled NEXSOL BD-0100) was sourced by the NREL in the United States and shipped to England. The shipment consisted of two lots of slightly differing product quality. To avoid doubt about the contribution of the different physical properties of the two lots, the batches were blended together to form a single sample.

Half of this combined sample of soy biodiesel was treated shortly after receipt at the Fuel Technology Centre with a commercially available antioxidant additive, tert-butyl hydroquinone (TBHQ) at 200ppm. The objective of this was to inhibit oxidation of the fuel for the life of the study.

Laboratory analysis on this now stabilised soy biodiesel was conducted according to the ASTM D6751 standard. In addition, tests for iodine value, peroxide value, oxidative stability and fatty acid speciation were also carried out. This fuel is now referred to as stabilised soy biodiesel.

4.1.3 Oxidised soy biodiesel (SME)

One 200 litre drum of soy based biodiesel was taken from the sample described in 4.1.2 and prepared so as to achieve an acid number of at least 3.5 mg/KOH/g.

The sample was heated to an average temperature of 57°C using an electric belt heater around the outside of the drum and sparged with BTCA 178 grade air (British Technical Council for the Motor and Petroleum Industries Fuels Committee) from a gas cylinder at a flow rate of approximately 4 litres/min. This sparging technique was considered to provide adequate mixing, without causing the sample to foam and to prevent a temperature gradient between the top to the bottom of the drum. Bottled air was used to ensure that the dew point was not compromised and that hydrocarbons were not introduced into the sample from site air compressors. In addition, some membrane filters used on normal compressed air are also known to deplete the oxygen content. The temperature of the sample was monitored throughout the oxidation treatment using an electronic digital thermometer. Acid number was measured daily using method ASTM D664 until it exceeded the required 3.5mg/KOH/g. In fact, an initial acid number of 4.013mg/KOH/g was achieved after 29 days, at which point heating and sparging was stopped. Acid number, however, decreased during the following days (3.818mg/KOH/g), stabilising at around 3.605mg/KOH/g. The stabilised value is taken as the final value for this report. It is thought that erroneous results might have been caused by high levels of dissolved carbon dioxide, introduced through agitation and sparging.

The oxidised fuel was then tested to the ASTM D6751 standard with additional tests for iodine value, peroxide value, oxidative stability and fatty acid speciation being conducted. The fuel was tested for acid number throughout the project to gain an understanding of the stability of soy-based biodiesel during storage.

This sample was labelled as 'Batch 1' and used to prepare all subsequent B5 blends which required oxidised soy based biodiesel.

To fulfill the testing requirements of the project, a second 200 litre sample of oxidised soy biodiesel was prepared. An acid number of 5.1 mg/KOH/g was achieved more speedily than the previous batch. This was accomplished due to the use of a large diffuser, providing small air bubbles with relatively large surface area, thus making better gas to liquid contact.

This sample was labelled as 'Batch 2' and used to prepare all B20 test blends which required oxidised soy based biodiesel.

Laboratory analysis of the stabilised soy biodiesel was conducted to the ASTM D6751 standard. Tests for iodine value, peroxide value, oxidative stability and fatty acid speciation were also carried out.

4.1.4 Rapeseed biodiesel (RME)

A quantity of European, additive-free rapeseed methyl ester meeting the EN14214 specification was obtained by the Associated Octel for this study. It was considered prudent to stabilise the rapeseed biodiesel for the duration of the study, and so the fuel was treated with 200ppm of TBHQ in the same manner as the soy biodiesel.

The rapeseed biodiesel was tested to the ASTM D6751 standard. Tests for iodine value, peroxide value, oxidative stability and fatty acid speciation were also conducted.

This stabilised rapeseed biodiesel was used to prepare all B5 and B20 test blends requiring the addition of RME.

4.2 Test Fuel Identification

A total of seven fuel blends containing 5%v/v (B5) and 20%v/v (B20) biodiesels in BP-15 basefuel were prepared for the study.

- B5 RME – Sample ID 2031510
- B5 SME – Sample ID 2031511
- B5 Oxidised SME (batch 1) – Sample ID 2031512
- B20 RME – Sample ID 2031513
- B20 SME – Sample – ID 2031514
- B20 Oxidised SME (batch 2) – Sample ID 2031942

Each blend was assigned a unique sample number which identified the sample throughout its test life. The untreated ultra low sulphur base fuel (BP-15) was assigned sample number 2031163. Product quality analysis was conducted on each test fuel as directed by the ASTM D975 standard.

It had been noted that batch 2 oxidised soy biodiesel (acid number of 5.1mg /KOH/g) was more extensively oxidised than batch 1 (acid number of 3.605mg /KOH/g). Section 5.1.6 describes the separation of the B20 fuel prepared from batch 2 oxidised soy biodiesel and it was of interest to this study to further investigate the fuel separation i.e. to determine whether the fuel separation was as a result of the degree of oxidation or because of the greater volume of the oxidised material present in the blend or caused by products of decomposition such as water and sediment.

One additional test fuel was subsequently blended to determine the behaviour and pump wear effects of a B20 blend prepared from a less highly oxidised soy biodiesel. This B20 blend was prepared from batch 1 oxidised soy biodiesel, the balance being the BP15 ULSD. A unique sample number of was assigned to this blend as detailed below:

- B20 Oxidised SME (batch 1) – Sample ID 2050822

Limited product quality analysis was conducted to measure fuel properties and one common rail pump test was performed to determine pump wear effects of this additional biodiesel fuel blend.

4.3 Elastomer Compatibility

Five candidate elastomer types typically used in automotive fuel systems were selected for this study. The selection was based on recommendations from the NREL and CRC and from a leading specialist supplier of automotive elastomers.

The physical properties of the test specimens prior to and after ageing were measured on the basefuel and six biodiesel test blends. 'O' ring test samples were specially moulded for the study to ensure consistency of material dimensions and production batch. Test measurements were taken as required by the standard test methods, ASTM D1414, D471 and D395. The standard test methods prescribe procedures for measuring changes to physical properties such as mass, hardness, dimension changes etc on exposure to air and fluids under defined conditions. Fluid aged samples were immersed in candidate fuels at 60°C for 1000 hours according to CRC specifications. Control samples were conditioned in air at 23°C plus or minus 2°C for the same period.

4.4 Injector Wear Tests

An Octel diesel fuel test rig was modified for this aspect of the study to enable the testing of fuel injectors for wear. Testing was conducted on the base fuel and three candidate B20 test fuels. Octel test method, EL-79, was developed to determine diesel injector nozzle wear using a 500 hour test procedure. The main operational conditions of the test procedure were as follows:

- The rig was operated at a constant speed of 1440 rev/min ± 25 rev/min for a period of 500 hours ± 10 hours.
- The injector block was heated to 150°C ± 10 °C to simulate normal operating conditions of the injectors.
- The bulk fuel temperature was controlled to 40°C ± 5 °C. It was permissible for the bulk fuel temperature to be outside these limits for the first 3 hours after start up and following a fuel change.
- The injectors recommended are of the indirect injection design with corresponding nozzles. The injectors were checked for spray pattern, leaks and opening pressure at the start of each test. The opening pressures were set to 130 ±5 bar absolute.

The test rig consisted of an in-line diesel injection pump running at a constant speed of 1440 rev/min. The fuel control rack was mechanically locked to ensure a consistent injection of fuel across all tests. The injection pump cam and followers were lubricated by a separate source and so were independent of the test fuel for lubrication. This ensured consistency of testing across all candidate fuels. Following the modifications, the rig was initially operated using slave components to prove reliability.

Test injector nozzles were purchased and the needles profiled by a specialist surface measurement company. A set of four injector nozzles were tested on each fuel. The injector nozzles selected for the study were as follows:

- Lucas injector nozzles, part number RDNOSD6754

By determining the mean and standard deviation for a large number of measurements on new injector components, it was possible to produce information relating to the new or manufacturers' acceptable ‘out of roundness’. For the injectors used in this project there were 4 sets of 4 injectors and each injector needle was assessed for ‘out of roundness’ at 5 different axial locations. This provided a set of 80 measurements, on new components, as the base line ‘acceptable’ level of ‘out of roundness’.

It was then possible to compare the post-test ‘out of roundness’ measurements (for each fuel tested) with this pre-test distribution to assess whether the fuel was acceptable or not; i.e., were the post-test results inside or outside the acceptable distribution at the 95% confidence level.

4.5 Rotary Pump Wear Tests

Octel Test Method, EL-80, was developed from the CEC F-32-X-99 test method for diesel pump lubricity. The test procedure employs a 500 hour test protocol compared to the CEC F-32-X-99 method which prescribes a 1000 hour test duration. The test method was used to perform seven Bosch VE rotary pump wear tests on the base fuel and six candidate biodiesel blends. The main operational conditions of the test procedure were as follows:

Test cycle				
Phase	**Acc. Running Time [s]**	**Pump Speed [% of rated speed]**	**Fuel lever position [%]**	**Fuel Outlet Temp. [°C]**
1	0	0	100	60
2	5	110%	100	60
3	7	110%	100	60
4	9	100%	100	60
5	118	100%	100	60
6	120	80%	100	60
7	170	80%	100	60
8	175	0	100	60
9	180	0	100	60

For each test a totally new Bosch VE pump, model number 0460 494 168 was used. Care was taken to ensure that the purchased pumps were not rebuilt, reconditioned or overhauled. It is normal practice in this instance not to conduct a pre-test rating of the test components. Experience has shown that this is not necessary as the quality control of the new components and assembly by the manufacturer is very good. In addition, commercial decisions around fuel lubricity quality should not be made on the strength of a single test, whatever the test duration or pre-test preparation.

The test cycle was originally developed by the pump manufacturers, Robert Bosch GmbH and was designed to accelerate the wear of the pump components in the following manner:

- The fuel control lever is locked in the maximum fuel position so as to increase the load on the pumping element components as well as increasing the duration of injection.

- The stop / start cycle repeatedly removes the hydrodynamic lubrication film between the moving components and so accelerates wear. This also provides a more realistic test cycle as fuel pumps in normal use would undergo many thousand starts, from a static condition, during their lifetime.

- The test cycle includes an over-speed section to exercise the components of the mechanical governor mechanism. The controlled fuel temperature and pressure have been chosen to simulate real life operating conditions. At start of test and after every 100 hours fuel exchange the fuel temperature must have reached its set value of 60 ± 5 °C after a maximum of 3 hours running time.

At the end of each test, the test pump was carefully dismantled for visual rating. This rating was conducted by raters who had attended training sessions by Bosch and also participated in comparative rating exercises with other companies undertaking this type of test work. This ensures some consistency of rating across all companies involved.

4.6 Common Rail Pump Wear Tests

Common rail diesel fuel injection systems are becoming widely used by vehicle manufacturers due to their microprocessor control, enabling a more flexible approach to how much and when the fuel is injected into the engine cylinder. This increased level of control enables the manufacturers to meet the low levels of exhaust emissions demanded by the current legislation.

The engines using common rail fuel systems are predominantly the passenger car and light-duty commercial vehicles. In addition to meeting the emissions legislation, these diesel engines are designed to perform more akin to a gasoline engine and have a wide speed range and quick response to driver demands. These characteristics, along with the inherent low fuel consumption and low CO_2 emissions have contributed to the increased popularity of the diesel engine.

The common rail fuel system is similar, in principle, to a gasoline multi-point fuel system. A fuel pump generates the pressure required for injection but has no position or time relationship with the rest of the engine. Solenoid operated injectors are controlled by a microprocessor to determine when and how much fuel is injected into each cylinder of the engine. Additional sensors also ensure the correct level of control over the system and that the relevant corrections are made for factors such as changes in ambient temperature.

To provide the high pressure fuel supply to a common rail injection system is a particularly arduous task. The pump must work continually at the maximum design operating pressure (up to about 1500 bar) and operate over a wide range of engine speeds (up to about 5000 rev/min engine crankshaft speed, 2500 rev/min cam speed).

In order to avoid any contamination of the fuel, the high pressure pump is lubricated solely by the fuel. This means that it is vulnerable to variations in the quality of the fuel and, in particular, the lubricity performance of the fuel.

The philosophy behind the common rail test rig was to mimic the real life operation of a common rail passenger car fuel system but in a controlled and repeatable fashion. The test rig was the chosen research tool as full engine tests, by their nature, require a ‘once through’ use of the fuel. Without recycling the fuel, several thousand litres of fuel would have been required for each test instead of the 250 litres used for the rig test. In addition, the pump rig test cycle was designed originally by Bosch to simulate actual in-service use but in a shorter time scale. This is done by the use of rapid acceleration rates, high speed running, including running over-speed and the use of frequent shut down periods. This is not a practicable way to operate a complete engine or vehicle.

Many automotive components rely on the principles of hydrodynamic lubrication for reliable operation. This only becomes effective once a rotating component attains a certain rotational speed. Therefore, to accelerate wear and better represent real life operation, it is important to break the hydrodynamic lubrication film.

The Octel test rig consisted of driving a common rail fuel pump (the test pump) at a speed representative of the more arduous conditions. The common rail fuel pump selected for the study was a Bosch common rail pump, part number 0445 010 010.

The test cycle chosen for the study was based on the pump rig test cycle used by Bosch but limited to a 500 hour test duration. The key test conditions are as follows:

- The running speed chosen was approximately 2000 rev/min which represented 4000 rev/min engine crankshaft speed. The actual speed range was between 1950 and 2000 rev/min.

- The pump was operated through a test cycle consisting of 3 minutes running at this test speed and 5 seconds stationary. The stationary portion of the test cycle was designed to accelerate the wear and better represent the stop/start nature of real life conditions.

- The pump was arranged to operate at a target pressure of 1350 bar, which is typical of many vehicle applications. Throughout the test, the operating pressure was arranged to be between 1200 bar and 1400 bar gauge.

- The test fuel was supplied to the pump at a flow rate and feed pressure matching the specifications of the vehicle system. This was arranged to be between 2.0 and 3.0 bar gauge.

- The fuel inlet temperature was controlled at 40°C ± 5°C. Fuel heating and cooling units were employed to meet this condition.

- The high-pressure outlet of the pump was connected to a fuel rail and electronic injector, as it would have been in the vehicle application. The injector was supplied with electrical signals in order to operate it at a fixed frequency and fixed open / close time periods (mark / space ratio). The injector was supplied with electrical pulses of 5 ms duration. A purpose built electronic driver was used for this to ensure test consistency. It was not possible to use the vehicle microprocessor as this was designed to operate in conjunction with various other vehicle systems and required feedback circuits that would have prevented consistency of testing.

After the initial set-up of the test pump, it was operated at the test speed whilst the fuel pump pressure control was adjusted to meet the test system rail pressure. The pump speed and pressure were monitored throughout the test. Any major changes provided an early indication of potential failure of the test pump. Additional parameters were monitored to ensure test consistency such as fuel feed pressure, ambient temperature and bulk fuel temperature.

For this study common rail wear tests were conducted on the base fuel and B5 and B20 biodiesel test blends. One additional common rail test was also conducted to further investigate the behaviour of a B20 oxidised soy biodiesel fuel. This fuel is described in section 4.1.3

At the end of a test, each test pump was dismantled and the critical components visually rated. The pump rating examines for surface abrasion, fretting and corrosion as well as polishing and wear steps.

5. RESULTS AND DISCUSSION

5.1 Fuel Analysis

5.1.1 Test Methods

Table 1 below details the standard test methods referred to in this report for the analysis of the base fuel, biodiesel and biodiesel blends.

Table 1 – Standard Test Methods

Test Method	Method Title
ASTM D1160	Test Method for Distillation of Petroleum Products at Reduced Pressure
ASTM D130	Test Method for Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test
ASTM D1510	Standard Test Method for Carbon Black-Iodine Adsorption Method
ASTM D189	Test Method for Conradson Carbon Residue of Petroleum Products
ASTM D2500	Test Method for Cloud Point of Petroleum Oils
ASTM D2709	Test Method for Water and Sediment in Middle Distillate Fuels by Centrifuge
ASTM D3120	Test Method for Trace Quantities of Sulphur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry
ASTM D445	Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (for Calculation of Dynamic Viscosity)
ASTM D482	Test Method for Ash from Petroleum Products
ASTM D4951	Test Method for Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma Atomic Emissions Spectrometry
ASTM D524	Test Method for Ramsbottom Carbon Residue of Petroleum Products
ASTM D5772	Test method for Cloud Point of Petroleum Products (Linear Cooling Rate Method)
ASTM D6079	Test Method for Evaluating Lubricity of Diesel Fuels by the High-Frequency Reciprocating Rig (HFRR)
ASTM D613	Test Method for Cetane Number of Diesel Fuel Oil
ASTM D6584	Test Method for Determination of Free and Total Glycerine in B100 Biodiesel Methyl Esters by Gas Chromatography
ASTM D664	Test Method for Acid Number of Petroleum Products by Potentiometric Titration
ASTM D6751	Standard Specification for Biodiesel Fuel (B100) Blend Stock for Distillate Fuels
ASTM D86	Test Method for Distillation of Petroleum Products
ASTM D874	Test Method for Sulphated Ash from Lubricating Oils and Additives
ASTM D93	Test Methods for Flash Point by Pensky-Martens Closed Cup Tester
ASTM D975	Standard Specification for Diesel Fuel Oils
ASTM D976	Test Methods for Calculated Cetane Index of Distillate Fuels
ASTM D2274	Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)
ASTM D395	Standard Test Methods for Rubber Property-Compression Set
Cd 8b-90	Peroxide Value, Acetic Acid-Isooctane Method
CEC F-06-A-96	Measurement of Diesel Fuel Lubricity
IP 438	Petroleum Products-Determination of Water-Coulometric Karl Fischer Titration Method

5.1.2 BP-15 Diesel

Laboratory analysis of the base fuel is detailed in Appendix 1. The fuel meets all the specifications of the ASTM D975 standard.

Additional analysis to examine the lubricity properties of this fuel by High Frequency Reciprocating Rig (HFRR) method CEC F-06-A-96 was also conducted at the request of the NREL. This method differs from the ASTM D6079 technique in that corrections are made for temperature and humidity. The result (584µm) demonstrates that the base fuel fails to meet the maximum European EN590 wear scar diameter 1.4 specification of 460µm. It has been noted that this fuel yields an HFRR measurement of 540 µm by test method ASTM D6079 although this result was not verified in this study. A low lubricity value for a non-additised fuel is not surprising as refinery processing to reduce sulphur content would have removed much of the natural lubricity properties of the fuel. Additive systems would normally be incorporated into the finished fuel to address this. The BP-15 base fuel supplied is reported to contain an additive to provide some measure of lubricity enhancement although this was not verified in this study.

5.1.3 Soy based biodiesel

Laboratory analysis of the stabilised soy biodiesel is detailed in Appendix 2. Further testing was conducted for iodine value, peroxide value, oxidative stability and fatty acid speciation and these results are included in Appendix 2 and, in the case of fatty acid speciation, in Appendix 4.

The fatty acid speciation showed that this material is atypical of soy biodiesel in that it contained no linolenic acid (C18:3), typically reported to be present in SME at around 7% by weight. However, this material was chosen by the CRC because it was known to have been distilled, resulting in very low natural antioxidant content.

The results detailed in Appendix 2 demonstrate that the fuel meets the requirements of standard ASTM D6751.

Peroxide evaluation of B100 biodiesels by method ASTM D2340 proved to be challenging. ASTM D2340 describes a titration technique incorporating a yellow to colourless end point, and it was found that the yellowish colour of some of the fuel sample masked the titration colour change. It was thought that the TBHQ additive in the fuel might mop-up free radicals from peroxides, stabilising them around the benzene ring. This could prevent a successful determination of titration end point. An alternative technique was identified (AOCS Official Method Cd 8b-90, Peroxide Value Acetic Acid-Iso octane Method, Sampling and Analysis of Commercial Fats and Oils). This method utilises a titration end point from blue to colourless. Analysis of fresh B100 samples without added anti-oxidant was successful although the reasons for the analytical difficulties were not further investigated. The results of analysis by this technique are detailed in Appendix 2.

The data set demonstrates a wide range of peroxide values (between 14 and 662 milli equivalents of peroxide per 1000 grams of sample) over the range of biodiesels tested. The peroxide value of the stabilized SME sample was recorded as 34.62 meq/kg. This was over twice the value measured for the rapeseed derived biodiesel (14.18 meq/kg) but significantly lower than the oxidised samples.

5.1.4 Oxidised Soy biodiesel

Laboratory analysis of the two batches of oxidised soy biodiesel is detailed in Appendix 2.

The first batch was oxidised to an acid value of 3.605mg KOH/g and was used to prepare all the B5 soy biodiesel blends tested and one additional B20 blend. The second batch was oxidised to an initial acid value of 5.101mg KOH/g and this was incorporated into the B20 blends tested throughout the study.

Further testing was conducted for iodine value, peroxide value, oxidative stability and fatty acid speciation. The results of these analyses are included in Appendix 2. The results of the fatty acid speciation are detailed in Appendix 4.

Overall, the data obtained demonstrate that significantly different properties are obtained upon oxidation of the soy biodiesel. It is important to note that batch 2 was much more extensively oxidised than batch 1 and the degree of oxidation, as determined by the acid number of the sample, also has a significant effect upon the chemical and physical properties of the sample. The flash point of both batches of oxidised fuel is lowered. Kinematic viscosity, cloud point, oxidation stability and carbon residue increased with higher acid number. In addition, substantially more insolubles were generated by test method ASTM D2274, the method recommended in ASTM D975 for assessing storage stability.

Both batches of oxidised SME exhibited high peroxide values (381.11 and 662 milli equivalents of peroxide per 1000 grams of sample). Although peroxide levels of this order have been reported in highly oxidised samples of fatty acid methyl ester, it should be noted that this method has not been evaluated to determine the applicability to biofuels. In addition, the stated scope of the method is exceeded at peroxide values greater than 70 and so some caution must be exercised when considering the results.

It was not possible to determine the cetane number (CN) of the second batch of oxidised soy by the ASTM D613 test method. Using this technique, the candidate sample is tested in conjunction with two bracketing reference fuels of known cetane number as specified in the test method. The indicated cetane value of the oxidised soy exceeded that of the secondary reference fuel; i.e., 74.8 cetane number. It is known that organic hydro peroxides are produced in the oxidation of hydrocarbons and these may perform as cetane improvers. The approximation of cetane number from either the cetane number or cetane index of the subsequent blend was considered questionable. Cetane index is determined from fuel density and the 50% distillation temperature. Biodiesels have very limited boiling point curves and so the determination of the 50% distillation temperature is difficult. This is illustrated by the widely different cetane number (60.8) and cetane index (47.5) of the B20 oxidised SME. Similarly, it was not considered reasonable to approximate cetane number of the B100 component from the subsequent B20 blend without more information on the effects of oxidation on ignition quality.

The distillation characteristics of the oxidised test fuels were also significantly affected by the process of oxidation, more appreciably the second batch of oxidised soy. T90 temperature of batch 1 increased by over 25°C compared to the non-oxidised sample. The distillation of batch 2 exhibited a cracking temperature of 463°C and the recovery at this temperature was limited to 88% by volume.

5.1.5 Rapeseed biodiesel

Laboratory analysis of the oxidised soy biodiesel is detailed in Appendix 2.

Further testing was conducted for iodine value, peroxide value, oxidative stability and fatty acid speciation. The results of these analyses are also included in Appendix 2. The results of the fatty acid speciation are detailed in Appendix 4.

Where applicable, the product quality of the rapeseed biodiesel meets the requirements of EN 14214 with the exception of cetane number. The cetane value of the fuel was measured at 50.9; the requirement of the standard being 51.0. However, the reproducibility of the method is 4.3 cetane numbers and so the difference may be insignificant. The fuel also meets the requirements ASTM D6751 for all tests conducted.

5.1.6 Biodiesel blends

Laboratory analysis of the candidate B5 and B20 biodiesel test fuels are detailed in Appendix 3. Additional testing was conducted to examine the lubricity properties of the biofuel blends by CEC F-06-A-96 (HFRR) method. These results are detailed in Appendix 3.

The analysis of the additional B20 fuel prepared from batch 1 of the oxidised soy biodiesel is discussed separately in section 5.18.

The biodiesel blends meet the requirement s of the ASTM D975 standard with the exception of carbon residue in the case of the B20 oxidised SME blend. At 0.89% mass carbon residue, this property exceeded the permitted limit by over 0.5% mass.

In general, the changes in properties of the oxidised fuels are reflected in the B5 and B20 test fuels; i.e., increased viscosity and carbon residue. The distillation of the B20 blend prepared from the second batch of oxidised soy fuel presented some difficulties and this probably reflects the altered distillation and cracking characteristics of the oxidised soy fuel. Cloud point measurements generally reflect the low cloud point of the base fuel (-11°C) although the stabilised B5 RME and B5 SME blends show cloud point reductions of -15°C and -14°C respectively. The reproducibility of this test method is 3°C and this should be taken into consideration when comparing the results.

Cetane qualities of the oxidised soy biodiesel blends are significantly higher than for the non-oxidised samples. As discussed in section 5.1.4, this is thought to be due to the oxidation of hydrocarbons and the formation of naturally high cetane number hydroperoxides.

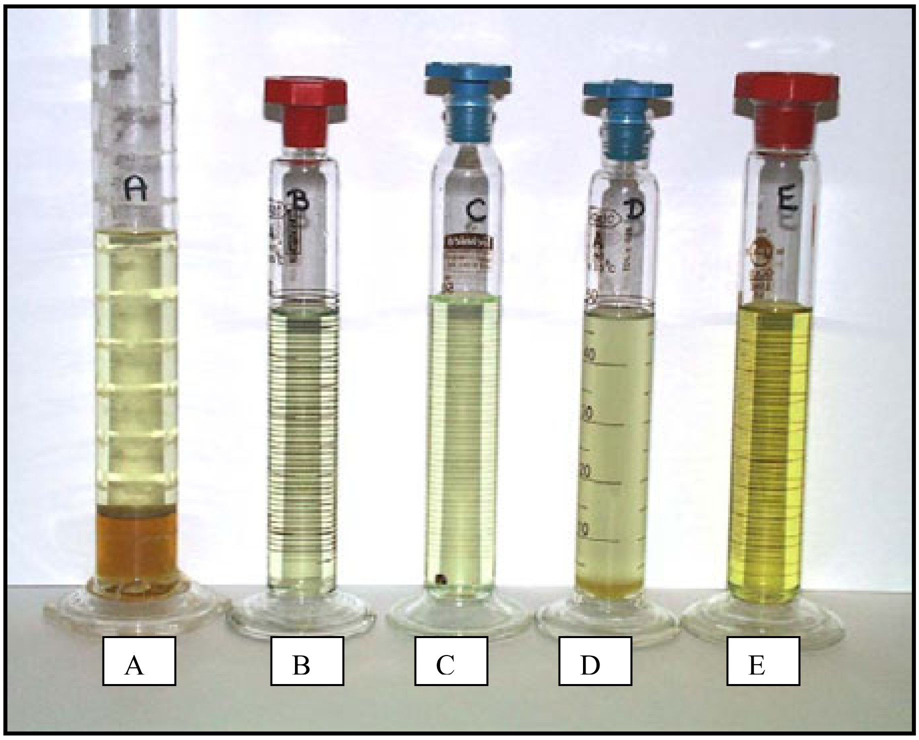
The lubricity properties of the base fuel when blended with biodiesel are significantly improved. The results of between 178 and 399 µm clearly demonstrate that all the blended fuels meet the European EN590 wsd 1.4 specification of 460µm in all cases. The most notable improvement in lubricity characteristics is shown with the B20 oxidised fuel blend (178µm).

Blockage of the fuel filter during the B20 oxidised soy biodiesel injector wear test caused the test to prematurely stop. This was subsequently found to be caused by separation of the test blend. Examination of the fuel revealed a slightly cloudy upper phase and a lower phase which was darker and significantly more viscous. The density of the bottom layer of fuel taken from the test rig fuel reservoir was measured at 1.0042kg/l. Subsequent density checks on samples taken from the top and bottom of the fuel storage drum yielded densities of 0.8511kg/l at 15°C in both cases. This compares reasonably well with the original density of the fuel blend, at 0.8462kg/l at 15°C.

Further samples were taken and observed for signs of separation in the laboratory. Although some phase separation was noted, the volume of the second, heavier phase was very small and thought to be primarily water separation. It may be concluded that separation of the fuel occurred during testing and not on storage. It had been earlier noted that the drum of B20 oxidised SME was cloudy, possibly due to the high water content previously reported. As a precaution, the fuel drum had been stored in an area which was not subject to extremes of temperature. In addition, the fuel was stirred briefly prior to extraction of sub-samples for all subsequent testing to ensure a homogenous test sample.

Photographs of the samples taken to evaluate test fuel 2031942 are included in Figure 1.

Figure 1 – B20 Oxidised Soy Biodiesel, Sample Number 2031942



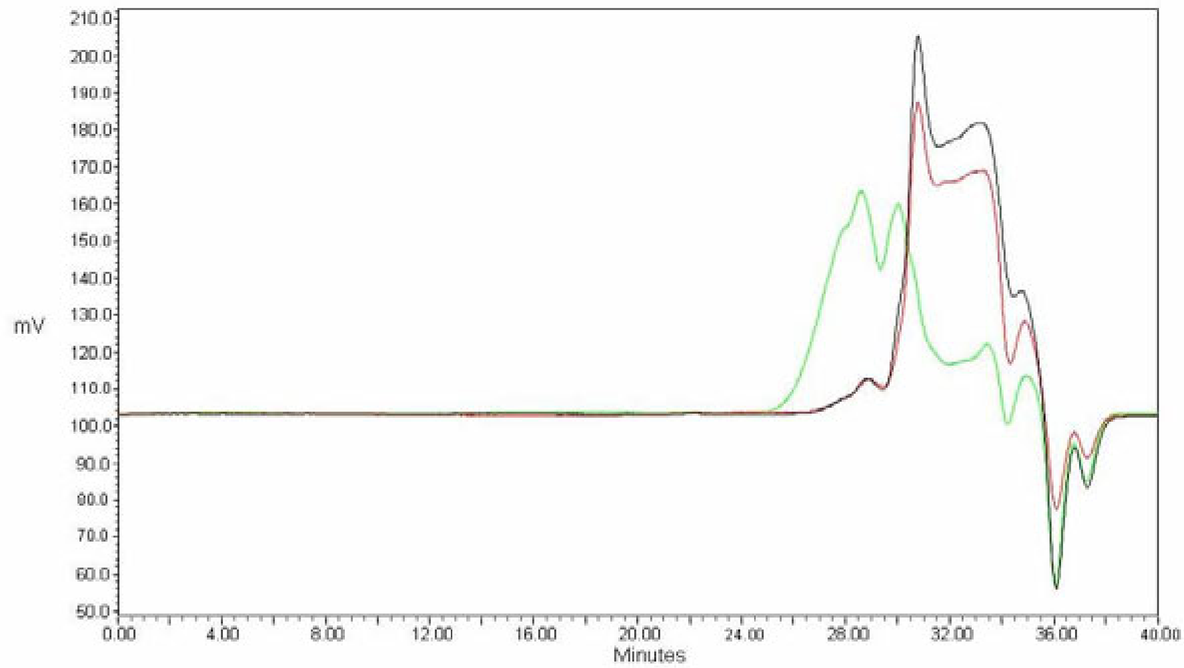
Sample **A** – Sample B20 Oxidised SME taken immediately after injector pump wear test
Sample **B** – Fresh sample B20 Oxidised SME taken from top of storage drum
Sample **C** – Fresh sample B20 Oxidised SME taken from bottom of storage drum
Sample **D** – Sample B20 Oxidised SME taken after 5 hours circulating through test rig
Sample **E** – Sample of B5 Oxidised SME (for comparison)

Further evaluations of fresh samples of the fuel test blend and the separated phases of the fuel were conducted by Associated Octel and the NREL. The findings are reported below.

- Gel-Permeation Chromatography (GPC)

This is a size separation technique commonly used to measure the molecular weight of polymers. The sample is dissolved in a suitable solvent such as tetrahydrofuran and forced through columns containing porous polymeric gel. Components elute in order of decreasing size or molecular weight.

Figure 2: GPC analysis of B20 oxidised soy biodiesel: fresh sample and separated phases



The chromatogram shown in Figure 2 shows the fresh sample of blend 2031942 (shown here in red) and the top layer (shown in black) to have similar molecular weights. In addition, they both contain relatively low molecular weight material. The green trace represents the bottom phase of the separated blend and indicates the presence of higher molecular weight material (peak molecular weight of around 937 relative to polystyrene standards). This suggests that the bottom phase of the sample may contain high molecular weight polymerised species.

- X-Ray Fluorescence (XRF)

Fresh samples of the stored oxidised soy B20 test fuel and the separated components of the fuel were examined for trace metal content by X-Ray Fluorescence. It was considered that increased metal content would be indicative of catalytic activity between metal conjunctions and acidic components and water present in the fuel. However, all three samples showed the presence of Cu, Mg, Si, S and Cl in similar quantities and no conclusion can be drawn from this analysis.

- Acid Value

Table 2 gives the results of two titration methods used to determine the acidities of a fresh sample of the B20 oxidised soy biodiesel blend and the same blend after separation. An alternative method employing lithium methoxide was proposed to overcome a perceived saponification problem with the use of potassium hydroxide (KOH) titrant in method ASTM D664.

Although no attempt was made to determine a correlation between the two techniques, both sets of results showed the bottom phase of the fuel to be significantly more acidic. In addition, the top layer of the separated fuel was slightly more acidic than the fresh fuel blend.

Table 2 – Acid Values of B20 oxidised soy biodiesel blend

Sample	Acid Value, mgNaOH/g	Acid Value, mgKOH/g ASTM D664
Fresh Sample	<1	-
Top Phase after separation	1.4	1.72
Bottom Phase after separation	15.5	24.03

From the results we can conclude that the bottom phase of the separated fuel is significantly more acidic in nature than the top phase.

Samples of the B20 soy biodiesel blend and the separated fuel phases were provided to the NREL. GC-MS analysis of the samples by the NREL concluded that the top phase consisted of slightly lower than 20% biodiesel content. The bottom phase was found to contain around 30% biodiesel and very little petroleum diesel. The balance of the bottom phase was found to be a mixture of biodiesel oxidation cleavage products consistent with known hydroperoxide decomposition chemistry and included highly polar oligomeric products. These results were confirmed by proton-NMR.

It may be theorised that the oxidized B20 blend had a high peroxide content and that these peroxides may have undergone decomposition during pump testing, forming polar products which are highly insoluble in diesel fuel. This may have been catalyzed by contact with metal surface or by the test conditions such as heat and pressure.

5.1.7 Water Analysis

During the course of the study, a number test results led us to query the values obtained from test method ASTM D2709 (Water and Sediment in Middle Distillate Fuels). For example, anomalous cloud point determinations and the slightly cloudy appearance of the oxidised soy biodiesel and corresponding B20 blend led us to consider the possibility of high water content in some fuels. However, with the exception of the B20 oxidised soy blend (0.007% vol.), water and sediment was not detected by the ASTM D2709 method. Indeed, ASTM standard D6751 does acknowledge that the precision and bias of test method ASTM D2709 with biodiesel is unknown.

Additional tests were undertaken to determine the water content in the base fuel, candidate biodiesels and biodiesel blends by an alternative method. This work, by the Karl Fisher coulometric titration method (IP438, EN ISO 12937), is reported in Table 3. The results by this method show that both batches of oxidised soy biodiesel contain significantly elevated levels of water, not detected by the ASTM D2709 test. The RME biodiesel also demonstrates relatively high water content by this test method.

It should be noted that the ASTM D2709 method is normally used to detect free water and sediment; i.e., water present as droplets or an emulsion. IP 438 has been established to determine the water content in products which have no visible water; i.e., dissolved water. Some caution must therefore be exercised when considering the results. Although no further work was conducted to establish a correlation between the test methods, it is recommended that consideration is given to the suitability, precision and bias of the test methods discussed when testing biodiesel and biodiesel blends.

Table 3 - Water content of biodiesel and biodiesel blends by Karl Fischer Titration method

Sample Number	Sample	Water Content , IP 438 % mass
2031163	Base fuel	0.004
2031382	Oxidised SME Biodiesel (Batch 1)	0.236
2031870	Oxidised SME Biodiesel (Batch 2)	0.763
2031034	SME Biodiesel (stabilised)	0.024
2031040	RME Biodiesel (stabilised)	0.096
2031510	B5 RME Blend	0.007
2031512	B5 Oxidised SME Blend	0.007
2031513	B20 RME Blend	0.012
2031942	B20 Oxidised SME Blend	0.115

5.1.8 Analysis of Additional Oxidised B20 SME Blend

A limited number of tests were conducted on batch 1 oxidised SME (sample number 2031382) to establish the quality of the fuel after several months of storage. This batch of biodiesel was used to prepare a further B20 test blend. The results of this are given in Table 4 below. Analyses of batch 1 oxidised SME conducted at the start of the study are presented for comparison.

Table 4 – Analysis of batch 1 oxidised soy biodiesel

Test Property	Method	ASTM D975 Limits	Oxidised SME biodiesel Batch 1 at end of study	Oxidised SME biodiesel Batch 1 at start of study
Acid Number	ASTM D664	0.80 mg KOH/g max	3.40mg KOH/g	3.370 mg KOH/g
Peroxide Value	Cd-8b-90	-	446.30 meq/kg	381.11 meq/kg

The results indicate that the acid number of the oxidised biodiesel had not changed significantly on storage. The measured peroxide value had significantly increased although caution must be exercised when considering the results as the measurements exceed the analytical scopes of both the ASTM D664 and Cd-8b-90 methods. In addition, Cd-8b-90 has not been established as an acceptable test method for analysis of biodiesels.

Additional testing was also conducted on the B20 blend prepared from batch 1 of the oxidised SME. This work was undertaken to establish the quality of the resulting blend and to support data obtained from an additional common rail pump wear test conducted on this test fuel. The results of this analysis are presented in Table 5.

Table 5 – Analysis of B20 blend prepared from batch 1, oxidised soy biodiesel

Test Property	Method	ASTM D975 Limits	B20 Oxidised SME (Batch 1) Sample No. 2050822	B20 Oxidised SME (Batch 2) Sample No. 2031942	Base Fuel BP-15 Sample No. 2031163
Flash Point	ASTM D93 (IP 34)	52 °C min	60.5	-	-
Water & Sediment	ASTM D2709	0.050% vol. max	1.0	7.0 (0.007*)	0.000 (0.000*)
Kinematic Viscosity	ASTM D445 (IP 71)	1.9-4.1 mm^2/sec	3.036	-	-
Copper Strip Corrosion	ASTM D130 (IP 154)	No 3 max.	1a	-	-
HFRR wsd 1.4	CEC F-06-A-96	460µm	272	-	-
Density	IP 365	N/A (kg/l)	0.8514	-	-

*Test values at start of study in brackets

Analysis of sample 2050822 shows that with the exception of water and sediment, the properties are within the acceptable limits of ASTM D975. The value for this property significantly exceeds the limit specified by the standard. Measured water and sediment of the base fuel is unchanged on storage and does not contribute to the elevated value.

In an effort to understand the relationship, if any, of water and sediment content and fuel separation, the stored B20 blend prepared from batch 2 of the oxidised SME was inspected. The fuel showed no evidence of the phase separation discussed in section 5.1.6 but it is thought that water in the fuel had dropped to the bottom of the drum as evidenced by an initial water/sediment value of 0.4% vol. The value of around 7% vol. reported in Table 5 was measured after the drum was inverted, rolled and sampled again.

Fatty acid methyl esters are known to be hygroscopic and water separates and collects at the bottom of the storage tank once solubility limits are exceeded. It is possible that hydrolytic reactions such as the conversion of fatty acid methyl esters into free fatty acids may have contributed to the oxidative degeneration of the fuel blend.

5.2 Elastomer Compatibility

Five elastomer types typically used in automotive fuel systems were selected for evaluation in this study. Table 6 below details the ASTM standard test methods used to determine the physical effects of the fuels on candidate elastomer materials.

Table 6 – Standard Test Methods

Test Method	Method Title
ASTM D1414	Standard Test Methods for Rubber O-Rings
ASTM D471	Standard Test Method for Rubber Property-Effects of Liquids
ASTM D395	Standard Test Methods for Rubber Property-Compressions Set

The physical properties of the test specimens prior to and after ageing in different fuel combinations were examined. The 'O' ring test samples were specially moulded for the study to ensure consistency of material dimensions and production batch. Test measurements were taken as prescribed by the test methods, ASTM D1414, D471 and D395. The standard test methods prescribe procedures and defined conditions for measuring changes to physical properties such as volume swell, hardness, dimensional changes and compression and tensile properties. Fluid aged samples were immersed in candidate fuels at 60°C for 1000 hours according to CRC specifications. Control samples were conditioned in air at 23°C plus or minus 2°C for the same time period.

Candidate fuel samples are detailed in Table 7.

Table 7 : Candidate Fuels for Elastomer Compatibility Testing

Test Fuel/Blend	Sample Number	Fuel I.D.
Basefuel	2031163	Fluid 5
B5 Rapeseed Methyl Ester	2031510	Fluid 6
B5 Soy Methyl Ester	2031511	Fluid 7
B5 Oxidised Methyl Ester	2031512	Fluid 3
B20 Rapeseed Methyl Ester	2301513	Fluid 2
B20 Soy Methyl Ester	2031514	Fluid 1
B20 Oxidised Soy Methyl Ester	2031942	Fluid 4

Elastomer materials selected for the study are detailed in Table 8.

Table 8 : Candidate Elastomer Materials for Elastomer Compatibility Testing

Elastomer Code	Description
NO674-70	Sulphur cured acrylonitrile butadiene nitrile rubber (NBR). Medium acrylonitrile content of 30-35%
NB104-75	Peroxide cured acrylonitrile butadiene nitrile rubber (NBR). Higher acrylonitrile content.
KB162-80	Hydrogenated nitrile polymer (HNBR)
VB153-75	Fluorocarbon polymer. 67% fluorine content.
V1164-75	Fluorocarbon polymer. 66% fluorine content.

The results of the tests conducted by a specialist test laboratory are detailed in appendices as follows:

Appendix 5 – Determination of Volume Swell of ‘O’ Rings
Appendix 6 – Determination of Hardness Properties of ‘O’ Rings
Appendix 7 – Determination of Dimensional Change of ‘O’ Rings
Appendix 8 – Determination of Compression Set Properties of ‘O’ Rings
Appendix 9 – Determination of Tensile Properties of ‘O’ Rings

Rating systems used by seal manufacturers are based not simply on changes to physical properties, which are dependant on temperature, length of exposure etc during testing, but also rely heavily on in-use service reports. It is important that the operational properties of an elastomer should not be significantly affected by the fuel nor should the life of the material be reduced. The service application, whether static or dynamic, and the medium which the material is likely to be in contact are of primary importance. The operational temperature is also important. For example, significant volume shrinkage can result in O ring leakage whether the mechanical application is static or dynamic. However, a compound which swells or is subject to elongation or changes to hardness or tensile strength may remain serviceable as a static seal despite unfavourable conditions. Many material combinations do not fall neatly into a single category and some engineering interpretation is necessary.

Overall, materials VB153-75 and V1164-75 were compatible with all the candidate biodiesel test fuels. V1164-75 especially exhibited good compression characteristics in all the test fuels. These materials showed best overall resistance to volume change and to changes to hardness and tensile properties. This is indicative of suitability for use in biodiesel applications up to B20 and a high overall compatibility rating would be assigned to these materials. However, this may not be applicable for higher concentrations of biodiesel.

Material NO674-70 exhibited good performance in all fluids with the exception of the B20 oxidised SME blend. This material is likely to be given a high rating for use in biodiesel applications although changes in physical properties and high volume swell in highly oxidised fuel may be observed.

Material KB162-80 gave acceptable performance in all fluids with the exception of the B20 soy biodiesel and B20 oxidised soy biodiesel where volume swell measurements were high. Manufactures' are likely to give this material a high overall compatibility rating in all test fluids with the exceptions noted here.

NB104-75 test material performed well in terms of hardness and volume change but overall exhibited substantial physical property changes across the range of test fluids. As a result, this material is likely to be rated as acceptable or depending on the application, not recommended.

- **Volume Swell**

Volume change is the change in volume of a material in contact with a fluid or vapour. An increase in volume of an elastomer is often accompanied by a decrease in hardness or softening. A reduced tear or abrasion resistance may result, permitting extrusion of a seal under pressure. Shrinkage of an elastomer may thus cause the seal to pull away from sealing surfaces, providing a path for fluid or vapour leakage.

Elastomer manufacturers' literature typically provides a rule-of-thumb whereby 'O' ring volume swell of up to 30% can be tolerated for static applications. Elastomers in dynamic applications are generally deemed to have lower 'acceptable' tolerances of around 10-15% volume swell.

The results of the volume swell analysis conducted in accordance to ASTM D1414-94, are detailed in Appendix 5.

The test data shows that the fluorocarbon 'O'ring materials, VB153-75 and V1164-75 exhibit the best overall chemical resistance in the fuel combinations tested, in terms of volume change. The maximum measured volume change of 6.8 % is acceptable for both dynamic and static applications.

Both the B5 and B20 blends of oxidised soy biodiesels have significant effect on the N0674-70, NB104-75 and KB162-80 test samples. In the B20 oxidised SME blend, all three materials present measurements which exceed 30% volume swell. Values of between 15.1% and 20.8% volume swell were measured in contact with the B5 oxidised SME blends. These elastomers might be subject to deterioration of mechanical properties in oxidised fuels especially in terms of extrusion resistance.

Test sample KB162-80 demonstrates poor swell resistance (24.3% volume swell) in contact with the B20 soy biodiesel blend which might be considered unacceptable for dynamic uses

With the exceptions noted here, permissible volume change for these materials in other fuels examined are within acceptable limits.

- **Hardness Properties**

The curved surfaces of the 'O' ring cross-section dictate that the industry standard Shore A type hardness scale cannot be used. The International Rubber Hardness Degrees (IRHD) is employed which allows for more accurate measurements on curved surfaces.

Elastomers with lower hardness readings fit easily into mating parts. This is important for seals used in low pressure systems because they are not activated by fluid pressure. Where seals are used in dynamic applications, the hardness of the 'O' ring is important because it significantly effects running and breakout friction.

Industry standard practice normally report actual points of change rather than the percent change from the original values as directed in ASTM D471. A rating of around 20 points or less for hardness change is generally considered acceptable for most applications. The results of the hardness analysis conducted in accordance to ASTM D1414-94, are detailed in Appendix 6.

Hardness measurements conducted before and after fluid immersion show the nitrile rubber samples to exhibit most overall reduction in hardness in all test fluids. The NBR material (NO674-70) exhibited most hardness change in the B5 and B20 oxidised soy biofuel, giving 9 and 14 point reductions respectively when compared to the unaged samples. The hydrogenated nitrile polymer (KB162-80) was most significantly affected by the B20 soy biodiesel blend with a reduction in hardness of 16 points. Test sample NB104-75 performed well in all fuels, exhibiting most hardness change, only 7 points, in the B20 oxidised blend.

The fluorocarbon materials (VB153-75 and V1164-75) exhibited the least decrease in hardness with maximum measurements of 5 points in all candidate fuels. These candidate test samples showed the best performance under the prescribed test conditions. However, it should be noted that all the materials tested are within what might be considered acceptable limits.

- **Dimensional Changes**

The dimensional change of the five 'O' ring materials was determined in accordance with the test standards. The internal diameter, external diameter and the thickness were measured before and after fluid immersion. The overall percentage change in dimensions was then calculated. The results are given in Appendix 7.

All materials showed a positive change in overall dimension measurements with greatest dimensional changes occurring following immersion in the B20 and B5 oxidised soy blends. In general, shrinkage of a material is generally considered to be the more usual cause of seal failure and dimensional changes must to be considered along with other properties such as volume change, hardness and compression set.

Fluorocarbon materials, V1164-75 and VB153-75, demonstrated the best overall resistance to dimensional change (1.3% maximum change) in all fuels tested. Test sample NB104-75 was the most significantly affected across the range of fuels with between 6.6% and 9.9% dimensional change occurring in the B5 and B20 oxidised fuels. Similarly, test samples NO674-70 and KB162-80 showed greatest dimensional change (5.0% to 9.0%) in the oxidised fuels.

- **Compression Set Properties**

This is generally reported as percentage change by elastomer manufacturers' rather than percentage change in compression set relative to the original material deflection as required by ASTM D395. It is a measurement of how the elastomer recovers after a fixed time under specified conditions of temperature and 'squeeze' (compression). Zero percent indicates that no relaxation of the material has taken place whereas 100% indicates total relaxation. A seal may subsequently contact mating surfaces but may not exert sufficient force against those surfaces. As with all the physical properties of elastomers, a good balance is generally required. For example, swelling of an elastomer may compensate for a poor compression set. A high compression set and dimensional shrinkage can lead to early seal failure except under conditions of high mechanical squeeze.

The results of the compressions set measurements are given in Appendix 8.

Overall, fluorocarbon test sample V1164-75 exhibited the best compression set characteristics for all material tested across the range of fuels (maximum 10 % change). Fluorocarbon material, VB153-75 did not perform well under this test (24.5-57.4 % change) even in base fuel. However, it should be noted that the test sample which was air conditioned at standard temperature and humidity also yielded relatively high compression-set measurements (29.3 % change).

Sample NB104-75 exhibited acceptable overall compression set in all fuels, with values ranging from -9.3 to 8.5% change. However, 32.4 % change was measured in contact with the base fuel. Test samples NO674-70 and KB162-80 demonstrated acceptable overall performance in all test fuels giving values of between -3.1 to 19.7 % change.

- **Tensile Properties**

Tensile strength may be used to indicate good strength characteristics required for long-term sealability and wear resistance in moving systems.

The results of the measurements taken to determine the tensile properties of candidate ‘O’ rings are given in Appendix 9. The laboratory tests measured each material using a universal tensile machine. The elongation was measured by means of a grip separation and the test speed was set to 500mm/min. The average percentage change in tensile strength and elongation at break were then calculated.

The data in Appendix 11 demonstrate that the tensile properties of the two fluorocarbon materials (VB153-75 and V1164-75) were not significantly affected by any of the test blends. A maximum value of 19.6 % change in tensile strength was measured in contact with the B20 oxidised fuel.

The nitrile ‘O’ ring seals (KB104-75) were most substantially affected overall with up -85.3% change in tensile strength and -79.7% elongation at break in contact with the two oxidised soy biodiesel blends. Test samples NO674-70 and KB162-80 gave acceptable results of around 50% or less for changes in tensile strength and elongation at break in all candidate fuels.

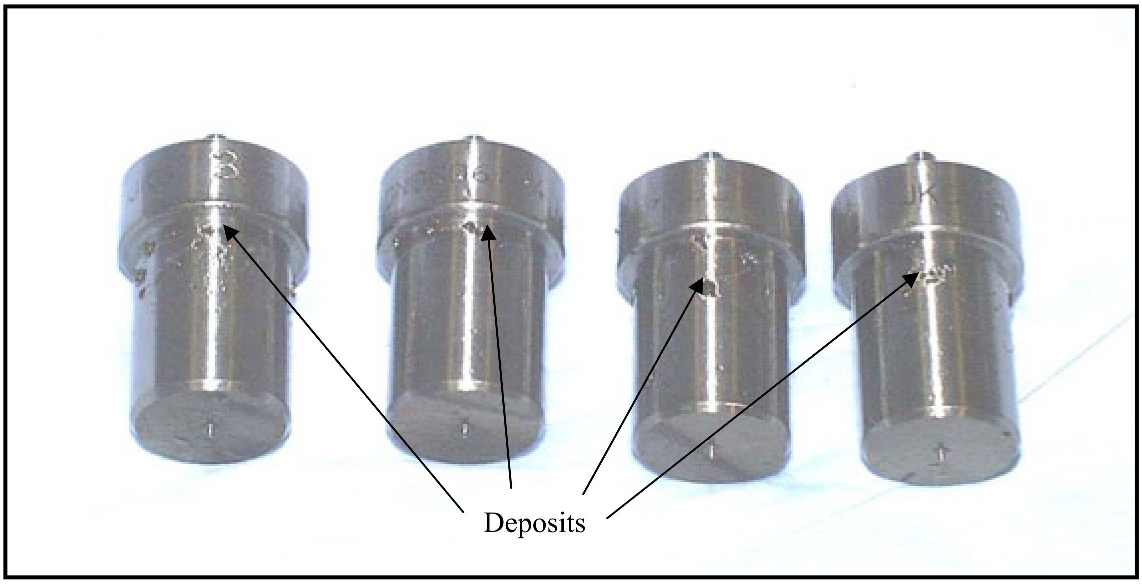
5.3 Injector Wear Tests

Under the terms of the study with the CRC, injector wear tests were conducted on the base fuel and three B20 biodiesel blends. Octel test method, EL-79, was developed to determine diesel nozzle wear using a 500 hour test procedure. This test method is included in Appendix 17. A photograph of the injector wear test rig is included in Appendix 10.

The injector wear test on the B20 oxidised soy biodiesel was terminated after only twelve hours of running following separation of the test fuel. Additional analysis was conducted on the fuel and this is detailed in section 5.1.6 of this report.

Injector sets from the four injector wear tests were assessed for roundness and taper by a specialist metrology laboratory. An example of one such evaluation is given in Appendix 11. These measurements were made before and after testing. Following each test, the injectors were examined, cleaned and stored in an appropriate manner to avoid corrosion prior to despatch to the specialist metrology laboratory. Although the injector wear test is designed to examine the wear of internal injector components, not deposits on the exterior surfaces, each injector was examined for any obvious sign of material deposition. With the exception of one injector set, all injectors were found to be free from obvious deposits or 'lacquering'. The photograph in Figure 3 shows the injector nozzles taken after the test on the B20 soy biodiesel (sample number 2031514) which had been stabilised with anti-oxidant. The photograph shows that some deposition was evident on the outside of the injector nozzles. However, this material is not thought to be lacquer, defined as a hard, dry, generally lustrous, oil insoluble deposit, as it was easily removed on washing.

Figure 3 - Injectors nozzles from test on B20 Soy Biodiesel, sample number 2031514



Injectors from the tests on B20 oxidised soy biodiesel (sample 2031942) were examined following the brief running period. No evidence of gum formation was observed but significant needle sticking was noted.

As part of the EL-79 test procedure, the test injectors were checked for opening pressures, spray pattern and leakage before and after each injector wear test. Some small increases in the 'leak back' measurements after testing are thought to be due to the seating of the needle in the nozzle during the test operation. There is no evidence that the fuel caused appreciable wear of the fuel inlet passage in the injector nozzles. In addition, post test fuel delivery measurements were similar across all test fuels and measurements were repeatable across the individual injectors.

The major parameter of concern when dealing with diesel fuel injector components is the quality of the surface finish of the high pressure mating components and whether these have deteriorated from the original surface finish when the components were manufactured.

For cylindrical components, the surface finish may be evaluated by the ‘out of roundness’ measurement. A cylindrical component may be visualised as having some measurable surface roughness. Theoretical circles could be fitted inside and outside the peaks and troughs of the surface. The difference between the radii of these circles is known as the ‘out of roundness’ measurement.

It is generally accepted that the measurement of industrial components of this kind conforms to a ‘normal’ distribution, due mainly to the large number of variables which are involved in manufacturing a component. Fuel injectors are no exception to this phenomenon.

Within a normal distribution, approximately 95% of the population will be enclosed between the bounds of $\pm 2\sigma$ ($\pm$ 2 standard deviations) from the mean of all measurements. Hence, if we take any single measurement, from this population, it will have a 95% chance of being within $\pm 2\sigma$ of the mean result.

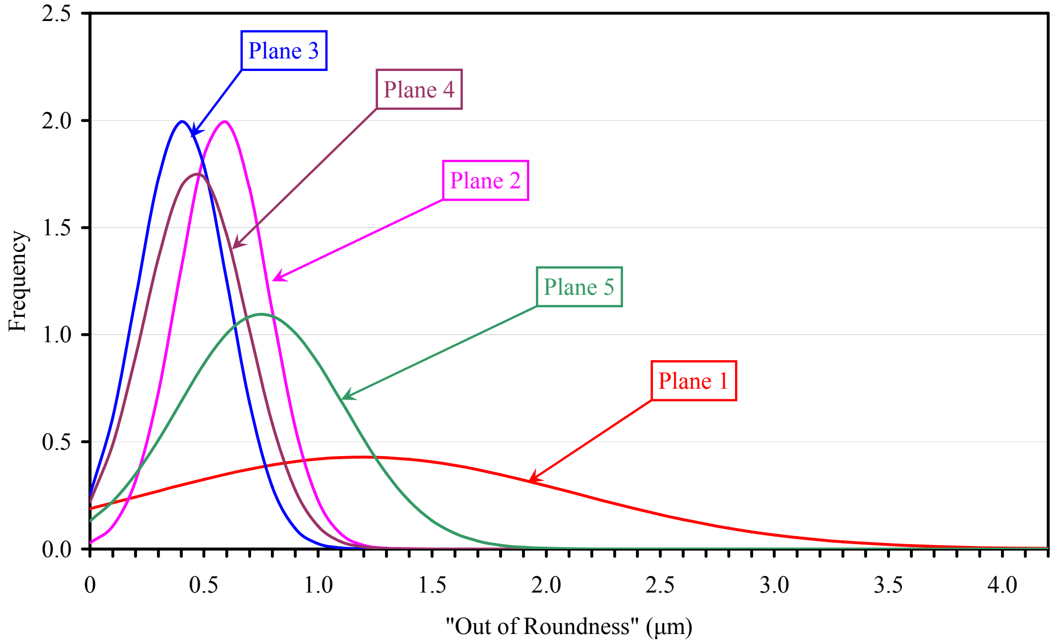
By determining the mean and standard deviation for a large number of measurements on new injector components, it is possible to produce information relating to the new or manufacturers acceptable ‘out of roundness’. For the injectors used in this project there were 4 sets of 4 injectors and each injector needle was assessed for ‘out of roundness’ at 5 different axial locations. This provides a set of 80 measurements, on new components, as the base line ‘acceptable’ level of ‘out of roundness’.

It is then possible to compare the post-test ‘out of roundness’ measurements for each fuel tested with this pre-test distribution. This provides a measure of whether the fuel is acceptable or not; i.e., is the post-test results inside or outside the acceptable distribution at the 95% confidence level.

Table 1 of Appendix 12 shows the ‘out of roundness’ data for each measurement on each injector needle both pre and post-test. If the pre-test data is taken as a whole or whether each measurement plane is taken as a different data set the result for injector 3-2 has been identified as an outlier by the Dixon Test. This result is therefore excluded from the subsequent analysis.

If each pre-test measurement plane is assessed as a different data set it is possible to draw a ‘normal’ distribution having the same mean and standard deviation as the measured data for that particular measurement plane. This has been done and the resultant curves are shown in Figure 4. From this chart it is clear that whist planes 2 to 4 show a similar distribution, plane 1 and plane 5 exhibit noticeably different characteristics. This could be explained by the physical characteristics of the injector needle. Planes 2 to 4 are on the ground part of the needle whilst planes 1 and 5 are probably on the turned part of the needle. A photograph of an injector needle showing planes 1 to 5 is given in Figure 5. For the subsequent analysis the data from plane 2 to 4 are taken as being comparable, thus there are 60 pre-test measurements and for each fuel, post-test there are 12 measurements.

Figure 4 – 'Normal' distributions for the 5 planes using the pre-test data



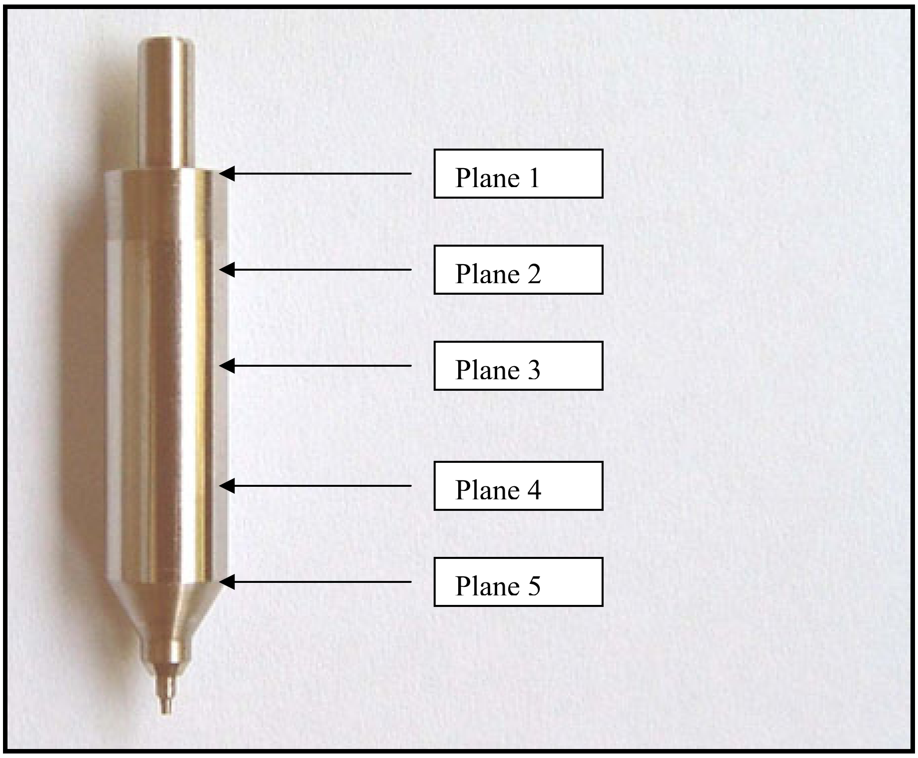
The statistical data for all 60 pre-test measurements show that the mean 'out of roundness' is 0.48 µm (see Table 2, Appendix 12). The 95% confidence band is thus 0.05 µm to 0.92 µm.

The post-test 'out of roundness' mean and standard deviation measurements for each fuel test can now be compared with these pre-test acceptable values to identify any which potentially fall outside the acceptable range (see Table 3, Appendix 12). A similar analysis can be performed for plane 1 and plane 5 as separate data sets, however as there are then only 4 post-tests measurements per fuel the statistical confidence is reduced.

All of the post-test measurements lie within the 95% confidence interval of the pre-test data. Therefore it may be concluded that the lubricity values of the fuels tested are adequate for the protection of the diesel injector components running under conditions similar to this test method.

Caution must be exercised in relation to fuel 2031942 (B20 oxidised soy biodiesel) as other fuel performance factors prevented this test being operated to completion. It must also be noted that no correlation work has been conducted to demonstrate how the EL-79 test might relate to real life operating conditions.

Figure 5 – Injector needle showing 5 measurement planes



5.4 Rotary Pump Wear Tests

Octel Test Method, EL-80, was developed from the CEC F-32-X-99 test method for diesel pump lubricity. This test method has been included as Appendix 18.

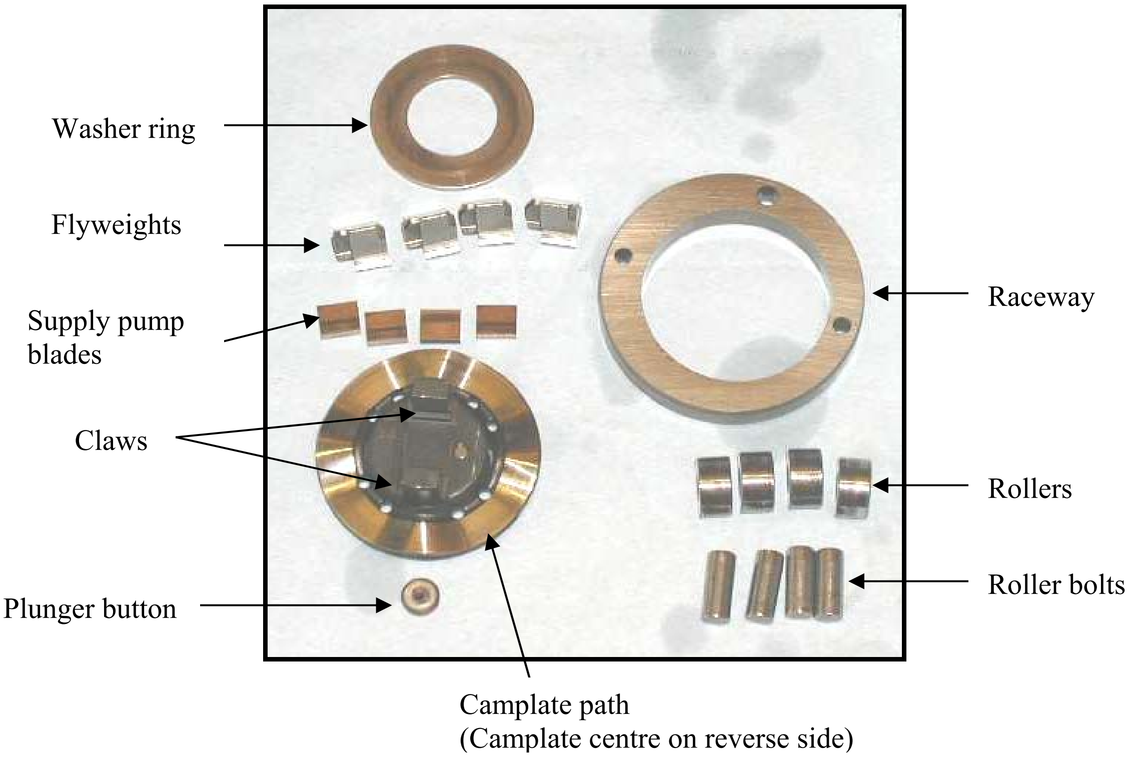
The tests conducted are based on the Bosch VE four cylinder rotary diesel injection pump with mechanical governor. The test protocol had a duration of 500 hours and included a stop/start regime. A total of seven fuels were tested on Bosch VE rotary pumps.

At the end of each test a wear rating assessment was conducted on each test pump and the results are tabulated in Appendix 13. A photograph of one of the dismantled test pumps is shown in Figure 6.

Figure 6. Dismantled Bosch Rotary Test Pump

Pump components examined for wear or damage are shown in Figure 7.

Figure 7 - Bosch VE Rotary Pump Components for Wear Rating



The rating scale is included in the test method and is based on a scale of 1 to 10. Ratings up to 3 indicate normal wear levels which would be expected and show no signs of damage which would lead to premature failure of the pump. A rating of 3.5 is the absolute limit of the 'normal' wear and is generally taken as the pass/fail border line. Ratings between 4 and 6 are given where there is some evidence indicating a reduced service life of the pump. This may be large amounts of wear, scuffing or fretting of the rated components. Ratings of 7 and above indicate major problems and likely catastrophic failure of the pump. Most pump tests conducted produce ratings below 6. Very poor lubricity fuels tend to cause seizure of the pump after only a few hours running and before major damage has occurred too many of the components. However, tests which fail to reach their full duration would receive an automatic 'fail', whatever the condition of the components.

The rotary pump test conducted on the B20 oxidised soy biodiesel blend, failed to reach full duration, stopping after 66 hours. Examination of the data log showed that the computer control system had registered low pressure of the fuel supply system feeding the rotary pump. The cause was found to be blockage of the fuel filter. Closer inspection of the test fuel showed that it had separated into two distinct phases; the bottom phase being significantly more viscous and darker than the top phase. This was similar to our experience of the same fuel during the injector wear test and it would seem reasonable to conclude that the fuel had undergone decomposition during testing.

It should be noted that the B5 oxidised soy biodiesel blend reached the full 500 hour test duration and excessive wear was not evident on examination and rating of the corresponding fuel pump.

Wear rating assessments conducted on all the candidate rotary pumps used in the study are tabulated in Appendix 13. The individual pump components for rating and the rating methodology are prescribed in Section 08 of the test method (Appendix 18). An overall rating was determined from the pump lubricity tests and these are summarised in Table 9.

Table 9 – Summary of Wear Rating Assessments of Bosch VE Rotary Pumps

Test Fuel/Blend	Sample Number	Pump I.D.	Overall Rating
Basefuel	2031163	54050	2.5
B5 RME	2031510	60321	2.5
B5 SME	2031511	54035	2.5
B5 Oxidised SME (Batch 1)	2031512	60315	3
B20 RME	2031513	50352	3
B20 SME	2031514	60323	3
B20 Oxidised SME (Batch 2)	2031942	53970	Fail (2.5)

The ratings produced from the pump lubricity tests conducted indicate that all the fuels are within the range normally expected for commercial automotive diesel fuels.

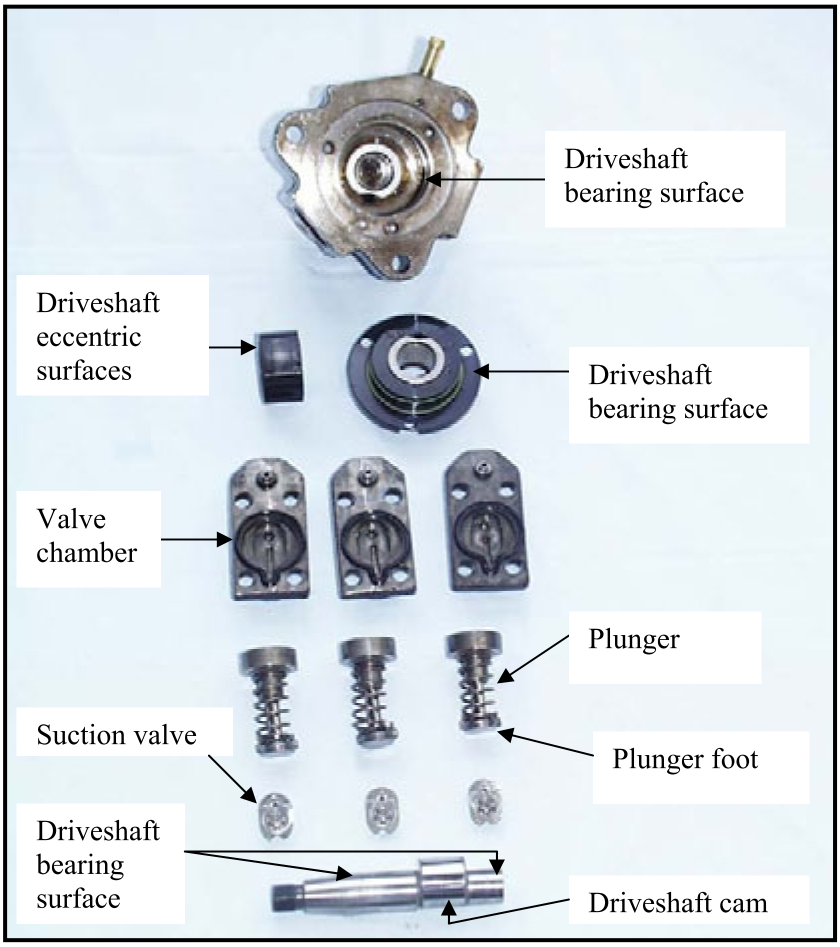
The amount of wear and consequently the ratings may have been slightly higher if the tests had continued to 1000 hours, but there is no evidence from the test components to suggest that the wear would have been outside the 'normal' range.

5.5 Common Rail Pump Wear Tests

An in-house test method was developed to determine pump wear using a 500 hour test procedure. This is described in detail in section 4.6. A photograph of the common rail wear test rig is included in Appendix 16. Common rail wear tests were conducted on the base fuel and candidate B5 and B20 biodiesel test blends. At the end of the study, an additional common rail test was conducted to further investigate the behaviour of a B20 oxidised soy biodiesel fuel.

The pumps from these tests were dismantled and visually assessed for surface abrasion, fretting and corrosion as well as polishing and wear steps. Pump components examined for wear or damage are shown in Figure 8.

Figure 8. Dismantled Bosch Common Rail Test Pump



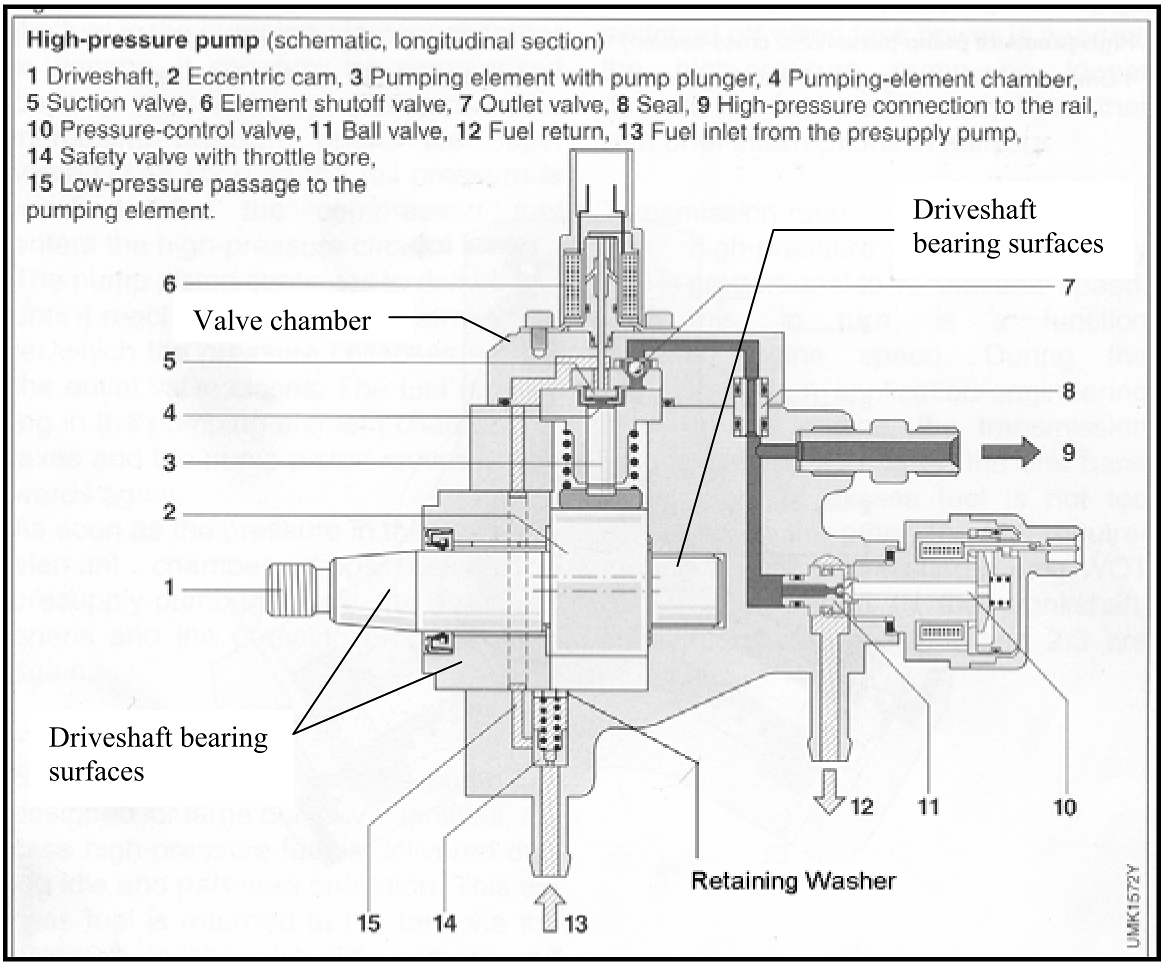
An overall rating was determined from the common rail pump wear tests and these are summarised in Table 10. The rating of individual pump components is detailed in Appendix 15.

Table 10 – Summary of Wear Rating Assessments of Bosch Common Rail test Pumps

Test Fuel/Blend	Sample Number	Pump I.D.	Overall Rating
Basefuel	2031163	6393	2.0
B5 Rapeseed Methyl Ester	2031510	6437	1.5
B5 Soy Biodiesel	2031511	6291	1.5
B5 Oxidised Soy Biodiesel	2031512	6218	2
B20 Rapeseed Methyl Ester	2031513	6438	2
B20 Soy Biodiesel	2031514	6433	2.5
B20 Oxidised Soy Biodiesel (Batch 2)	2031942	3374	1.5
B20 Oxidised Soy Biodiesel (Batch 1)	2050822	3159	1.5

It is worth noting that a further common rail pump component was considered for inclusion in the wear assessments of the test pumps. This component is a retaining washer which would not normally be included as a rated part. It was found to show significant signs of wear in all cases. The component is not a mechanically loaded part and is not subjected to the usual wear criteria. It is not a moving part but the level of wear suggests that it is in contact with the pump rotor. From sectional drawings available to us, we believe that these parts were not designed to be in contact. This same problem was observed on every test pump dismantled and it is our view that this is a design flaw rather than any assembly or build problem. The pumps tested were supplied as brand new (not reconditioned or rebuilt) and were not dismantled prior to test. The location of the retaining washer is shown in the common rail pump schematic (Figure 9). It is our belief that the retaining washer is only held on half of its surface and so is susceptible to its un-restrained portion tilting towards the rotor. As the rotor revolves eccentrically, it comes into within close proximity of the pump body and the retaining washer. If the retaining washer protrudes from its bore, it may then be contacted by the rotor. Witness marks on the rotor appear to confirm this. Although it was decided to exclude this pump component from the wear assessments for this study, our concern remains that any metal particles resulting from wear of this component have the potential to cause damage to other components.

Figure 9 : Common Rail Pump Schematic



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All of the candidate test fuels completed the 500 hour test procedure. Overall, none of the fuel blends tested showed any adverse effects on the wear ratings of the common rail fuel pumps. With the exception of the B20 fuel blend (2031942) prepared from batch 2 of the oxidised SME, there was no evidence of unusual deposits, gums or lacquers on the pumps or any rated parts.

Examination of the fuel pump used to test the highly oxidised B20 fuel (2031942) revealed a thin lacquer coating on the shaft bearing surface. The lacquer may be described as hard and dry with a lustrous appearance, which was oil insoluble and which was not removed on washing. In addition, there was evidence of seal swelling on dismantling the pump. Photographs of the lacquered component and the elastomer seal in the valve chamber which had become swollen are given in Figures 10 and 11.

Figure 10. Lacquering on Common Rail Test Pump Component

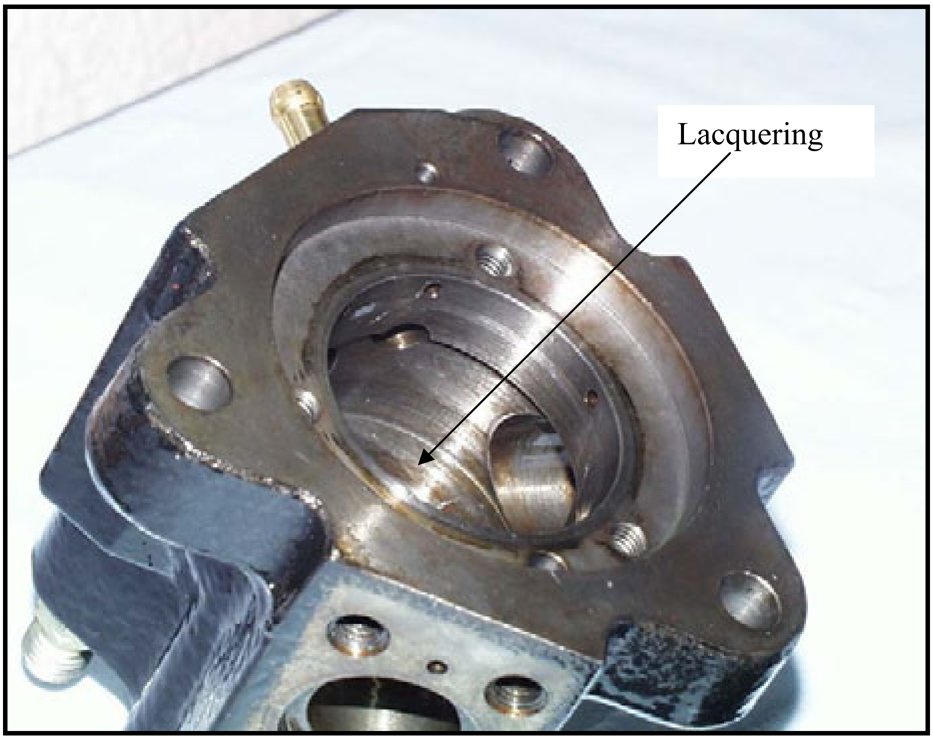
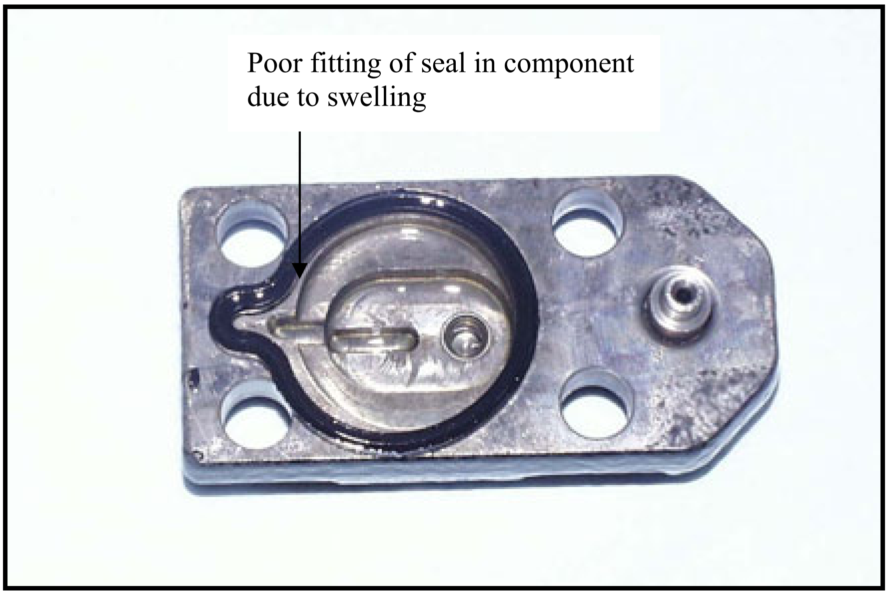


Figure 11. Common Rail Test Pump Component Showing Seal Swell



This test fuel consisted of 20% by volume of the highly oxidised soy biodiesel and had earlier resulted in failure of the Bosch VE rotary pump test and the injector test. Although the common rail test on this candidate fuel was completed without incident, a significant volume of a dark, viscous material was present in the bottom of the fuel tank when the fuel was changed after each scheduled 100 hours test run. Care had been taken to ensure that all test fuels were not subject to extremes of temperature in storage and that representative and mixed samples were offered up for testing. Inspection of the test fuel revealed that two phase separation had taken place; the bottom phase being significantly heavier and more viscous and than the top phase. This was comparable to our experience of the same fuel during the injector wear test and it would seem reasonable to conclude that the fuel had undergone decomposition during testing.

Water and sediment contents of this test blend were measured by method ASTM D2709 at the start and end of the study. The measured value increased substantially from 0.007% by volume to 7% by volume. However, there is a time disparity between testing the fuel on the common rail test rig and measuring the final water and sediment value and this should be taken into consideration when comparing the results. Although the acid value of the test blend is not available, the acid value of batch 2 oxidised SME after approximately six months storage was found to have reduced from 5.10 mg KOH/g to 4.55 mg KOH/g.

It is thought that filter blockage observed on the rotary pump rig did not occur on the common rail test rig due to the much higher feed pressures used; 2.5 bar compared with 0.4 bar. In addition, the common rail test rig has a greater volume flow rate of fuel circulating via a by-pass system and the outlet from the common rail test pump is higher (5 -10°C) than the VE pump. The fuel is also forced ('squeezed') through the filter which may prevent filter blockage.

On the basis of the separation problem, fuel of this composition would not be recommended for commercial automotive fuels.

The B20 fuel prepared from the less oxidised batch of soy biodiesel (sample number 2050822) completed the 500 hours of testing on the common rail test rig. Careful inspection of the fuel before testing provided no evidence of fuel separation and heavier, more viscous fuel components were not evident in the fuel tank after completion of the test. However, there was clear evidence of seal swelling of the valve chamber seals on dismantling the pump. It has been established that fuel pump manufacturers typically employ 'O' ring seals prepared from hydrogenated nitrile polymers (HNBR) similar to test material KB162-80. From the results of the elastomer compatibility testing (Section 5.2), this material exhibited significant volume swell (24.3%) and softening (16 point reduction in hardness) in the B20 oxidised SME fuel. Given these two physical changes, this might permit the extrusion of 'O' ring seals in certain applications such as in high pressure systems. However, it should be noted that both of the B20 test fuels discussed were oxidised and untypical of fuels meeting national specifications.

It is clear that the less oxidised B20 fuel (sample number 2050822) does not behave in the same way as the highly oxidised B20 fuel under the conditions of the test. i.e., significant phase separation does not take place. The test fuel was prepared immediately prior to testing and so was not subject to long periods of storage. Although a water and sediment content of 1% by volume was measured in the finished blend by the ASTM D2709 test method, acid value measured on batch 1 oxidised SME at the end of the study showed that this parameter had not changed significantly on storage (see Section 5.1.8).

The available evidence indicates that phase separation did not occur in either of the B20 oxidised fuel blends on storage. Decomposition reactions occurring under the conditions of test probably accelerated fuel separation in the more highly oxidised B20 test fuel. Phase separation did not occur during or after testing of the less oxidised B20 fuel. This indicates that the concentration of the oxidised component in the blend is not the main factor which determines fuel separation. The B20 fuel prepared from less oxidised biodiesel also contained lower amounts of water and sediment compared to the more highly oxidised B20 fuel. The different behaviour of the oxidised fuels is likely to be due to the extent of oxidation of the biodiesel component in the fuel.

6. CONCLUSIONS

- The highly oxidised B100 biodiesel and biodiesel blends prepared for this study have significantly different physical and chemical characteristics to non-oxidised biodiesel and biodiesel blends. The B20 test blend containing highly oxidised biodiesel may have been more highly oxidised than is likely to occur in the real world.

- Fuel filter blocking and fuel separation was observed during testing of the highly oxidised B20 test fuel in this study. Products of oxidation in the test fuel and decomposition reactions occurring under the conditions of test probably accelerated fuel separation in the fuel blend. Fuels likely to present this composition cannot be recommended for use for commercial automotive fuels.

- Phase separation and filter blockage did not occur during testing of B5 and B20 blends prepared from biodiesel which had been less extensively oxidised and which contained lower water and sediment contents. The tests indicate the behaviour of oxidised fuels under conditions of test are not dependant on the concentration of oxidised component and may be due to the extent of oxidation of the biodiesel component.

- B5 fuel prepared from oxidised biodiesel did not cause abnormal wear in either the injector or pump wear tests conducted in this study. Fuel filter blocking and fuel separation was not encountered during testing of this fuel.

- The results produced from injector wear tests indicate that the lubricity of the test fuels are adequate for the protection of diesel injector components running under similar conditions. The injector component wear test on the highly oxidised B20 blend failed to reach completion due to fuel filter blockage. It should be noted that the test method used for this study was a novel 500 hour test procedure. Commercial decisions around lubricity quality should not be based on a single test.

- The ratings produced from pump lubricity tests indicate that all test fuels are within the range normally expected for commercially available automotive diesel fuel running under the test conditions selected for this 500 hour test procedure. The rotary pump wear test on the highly oxidised B20 blend failed due reach completion due to fuel filter blockage. It should be noted that commercial decisions around lubricity quality should not be based on the results of single test.

- None of the candidate test fuel blends tested showed any adverse effects on the wear ratings of the common rail fuel pumps using a novel 500 hour test procedure. The test results indicate that the lubricity of the test fuels is adequate for the protection of common rail pumps running under similar conditions. It should be noted that commercial decisions concerning lubricity should be based on more than one test.

- Material compatibility testing of candidate elastomers has shown that fluorocarbon elastomers of medium to high fluorine content are most compatible with the test fuels under the specified conditions at concentrations of 20% or below. The results show that other candidate materials tested exhibited good resistance to changes in physical properties but exceeded the typically acceptable levels of degradation in one or more tests. These materials may be less compatible with biodiesel blends under certain applications.

ACKNOWLEDGEMENTS

The author is glad to acknowledge the help of many colleagues, most notably Mr Ian Bradbury and Dr Paul Richards for invaluable information on bed engine testing. In addition, the author is grateful for the invaluable services and information supplied by Parker Hannifin Corporation, O Ring Division.

APPENDICES

APPENDIX 1

Analysis of BP-15 Diesel Base Fuel

ASTM D 975

Property	ASTM Method or equivalent	Limits	BP- 15 2031163
Flash Point	IP 34 (D93)	52°C min	57.5
Water & Sediment	D2709	0.050% vol.max	0.000
Kinematic Viscosity,40°C	IP 71 (D445)	1.9-4.1mm^2/sec.	2.499
Sulphur	D3120	0.05% mass max	16µg/g
Copper Strip Corrosion	IP 154 (D130)	No.3 max	1a
Cetane Number	D613	40 min	49.3
Cloud Point	D5772	Report to customer	-11°C
Ash	D482	0.01%mass max	0.0
Carbon Residue,100% sample	D524	0.35% mass max.	-
Distillation, equiv. temp.,90% vol recovered	IP 123 (D86)	282-338°C max	322
Calculated Cetane Index	D976	-	50.3
HFRR wsd 1.4	CEC F-06-A-96	460µm	584

Analysis of Biodiesels
ASTM D6751

Property	Test Method	Limits	Rapeseed Biodiesel 2031040	Soy Biodiesel (Stabilised) 2031034	Batch 1 Soy Biodiesel (Oxidised) 2031382	Batch 2 Soy Biodiesel (Oxidised) 2031870
Flash Point	IP 34 (D93)	130°C min	>130	>130	>120	113.5
Water & Sediment	D2709	0.050% vol.max	0.000	0.000	0.000	0.000
Kinematic Viscosity,40°C	IP 71 (D445)	1.9-6.0 mm^2/sec.	4.686	3.972	7.276	9.837
Sulphated Ash	D874	0.020% mass max.	0.003	>0.001	0.004	0.003
Sulphur	D3120	0.05% mass max	<1.0µg/g	<1.0µg/g	<0.2µg/g	N/A
Copper Strip Corrosion	IP 154 (D130)	No.3 max	1a	1a	1a	1a
Cetane Number	D613	47 min.	50.9	50.4	59.1	* Note 1
Cloud Point	D2500	Report	-6	0	3	6
Oxidation Stability	D2274	-	1.52mg/100 ml	1.15mg/100 ml	8.28mg/100ml	8.38mg/100ml
Iodine Value	D1510	-	115	131	106	94
Peroxide Value	Cd 8b-90	-	14.18 meq/kg	34.62 meq/kg	381.11 meq/kg	662.43 meq/kg
Fatty Acid Speciation	GC/MS	-	Reported	Reported	Reported	Reported
Carbon Residue,100% sample	D189	0.050% mass max.	0.23	0.019	6.24	9.4
Acid Number	D664	0.80mg KOH/g max.	0.492 *Note 2 0.490 *Note 3	0.036	3.605*Note 2 3.370 *Note 3	5.101*Note 2 4.55 *Note 3
Free Glycerine	D6584	0.020% mass max.	<0.01	<0.01	<0.01	<0.01
Total Glycerine	D6584	0.240% mass max.	0.21	<0.01	0.03	<0.01
Phosphorus	D4951	0.001% mass max.	<10mg/kg	<10mg/kg	<10mg/kg	N/A
Distillation, atmospheric equiv. temp.,90% recovered	D1160	360°C max	179	175	301	463 *See Note 4

Note 1 : Unable to rate fuel. Result exceeds T22 secondary reference fuel, 74.8 cetane number
Note 2 : Acid value measured immediately after oxidation
Note 3 : Acid value measured after approximately six months storage
Note 4 : 90% recovery not attained. Cracking temperature reported. Recovery at cracking temperature: 88.0% vol.

APPENDIX 3

Analysis of Biodiesel Blends
ASTM D 975

Property	ASTM Method	Limits	B5 RME biodiesel 2031510	B5 Soy Biodiesel 2031511	B5 Soy Biodiesel (oxidised) 2031512	B20 RME Biodiesel 2031513	B20 Soy Biodiesel 2031514	B20 Soy Biodiesel (oxidised) 2031942
Flash Point	IP 34 (D93)	52°C min	64	66	61	59	66	60.5
Water & Sediment	D2709	0.050% vol.max	0.000	0.000	0.000	0.000	0.000	0.007
Kinematic Viscosity,40°C	IP 71 (D445)	1.9-4.1mm^2/sec.	2.544	2.561	2.619	2.833	2.705	3.068
Sulphur	D3120	0.05% mass max	11µg/g	10µg/g	10µg/g	9µg/g	8µg/g	N/A
Copper Strip Corrosion	IP 154 (D130)	No.3 max	1a	1a	1a	1a	1a	1a
Cetane Number	D613	40 min	48.2	48.6	52.7	49.2	49.2	60.8
Cloud Point	D2500	Report to customer	-15°C	-14°C	-10°C	-8°C	-9°C	-10°C
Ash	D482	0.01%mass max	0.007	0.006	0.002	0.004	0.009	0.00
Cetane Index	D976	40 min	50.2	49.5	51.2	50.1	51.4	47.5
Carbon Residue,100% sample	D524	0.35% mass max.	0.04	0.07	0.12	0.07	0.06	0.89
Distillation, equiv. temp.,90% vol recovered	IP 123 (D86)	282-338°C max	325.5	325.5	325.0	334.0	332.5	336.5
HFRR wsd 1.4	CEC F-06-A-96	460µm	188	355	234	178	399	167

APPENDIX 4

Fatty Acid Speciation of Biodiesels by GC-MS

	Soy biodiesel 2031034		Rapeseed biodiesel 2031040		Oxidised Soy biodiesel 2031382	
Ester Compound	**Area**	**Area %**	**Area**	**Area %**	**Area**	**Area %**
Palmitoleic acid	1,970,070	0.14	6,245,539	0.37	3,598,633	0.26
Palmitic acid	149,099,734	10.35	88,591,962	5.25	165,063,161	11.79
Heptadecanoic acid	2,076,239	0.14	1,009,331	0.06	3,289,198	0.23
Linoleic acid	766,463,990	53.19	403,287,170	23.92	514,472,119	36.75
Oleic acid	438,681,408	30.44	1,036,382,541	61.47	402,274,584	28.73
Stearic acid	73,575,690	5.11	56,097,203	3.33	90,292,765	6.45
Gadoleic acid	4,436,999	0.31	42,931,542	2.55	17,768,000	1.27
Arachidic acid	4,702,346	0.33	21,271,543	1.26	19,878,586	1.42
Erucic acid	0	0	8,838,091	0.52	0	0
Behenic acid	0	0	14,793,707	0.88	0	0
Lignoceric acid	0	0	6,468,051	0.38	0	0
C8 Alcohol	0	0	0	0	5,858,673	0.42
C10 Alcohol	0	0	0	0	1,676,555	0.12
Octanoic acid	0	0	0	0	3,946,236	0.28
Decadienal	0	0	0	0	506,840	0.04
Oxo-nonanioc acid	0	0	0	0	9,180,128	0.66
Octadecenal	0	0	0	0	2,867,219	0.20
C10-C12 Diol	0	0	0	0	2,987,576	0.21
Myristic acid	0	0	0	0	2,268,986	0.16
C14-C16 Alcohols	0	0	0	0	2,173,475	0.16
Possible C19 Alcohols	0	0	0	0	94,262,997	6.73
Possible C21 Acids	0	0	0	0	33,280,693	2.38
Possible C20 ester	0	0	0	0	24,369,129	1.74
TOTAL	1,441,006,47	100.00	1,685,916,680	100.00	1,400,015,55	100.00

APPENDIX 5

Determination of Volume Swell of ‘O’ Rings

ASTM D1414

Material	Volume Swell (%)							
	Unaged	Fluid 1 B20 Soy Blend	Fluid 2 B20 RME Blend	Fluid 3 B5 Oxidised Soy Blend	Fluid 4 B20 Oxidised Soy Blend	Fluid 5 Basefuel	Fluid 6 B5 RME Blend	Fluid 7 B5 Soy Blend
NO674-70	0.0	12.2	12.7	15.1	30.6	11.0	11.3	11.2
NB104-75	-0.1	16.1	15.6	20.8	33.7	12.1	13.8	15.1
KB162-80	0.9	24.3	14.3	20.8	30.9	12.5	15.5	13.8
VB153-75	0.3	6.8	6.2	4.3	2.7	1.1	2.4	3.4
V1164-75	-0.2	5.0	4.5	2.0	4.4	1.2	1.3	1.7

APPENDIX 6

Determination of Hardness Properties of 'O' Rings

ASTM D1414

Material	Micro Hardness (IRDH)							
	Unaged	Fluid 1 B20 Soy Blend	Fluid 2 B20 RME Blend	Fluid 3 B5 Oxidised Soy Blend	Fluid 4 B20 Oxidised Soy Blend	Fluid 5 Basefuel	Fluid 6 B5 RME Blend	Fluid 7 B5 Soy Blend
NO674-70	64	59	59	55	50	60	59	60
NB104-75	65	62	59	64	58	60	60	63
KB162-80	75	59	68	70	72	69	71	68
VB153-75	68	63	63	64	63	65	64	64
V1164-75	76	73	72	71	72	74	73	72

Material	Change in Micro Hardness (%)							
	Unaged	Fluid 1 B20 Soy Blend	Fluid 2 B20 RME Blend	Fluid 3 B5 Oxidised Soy Blend	Fluid 4 B20 Oxidised Soy Blend	Fluid 5 Basefuel	Fluid 6 B5 RME Blend	Fluid 7 B5 Soy Blend
NO674-70	-	-7.8	-7.8	-14.1	-21.9	-6.3	-7.8	-6.3
NB104-75	-	-4.6	-9.2	-1.5	-10.8	-7.7	-7.7	-3.1
KB162-80	-	-8.0	-9.3	-6.7	-4.0	-8.0	-5.3	-9.3
VB153-75	-	-7.4	-7.4	-5.9	-7.4	-4.4	-5.9	-5.9
V1164-75	-	-3.9	-5.3	-6.6	-5.3	-2.6	-3.9	-5.3

Determination of Dimensional Change of ‘O’ Rings

ASTM D1414

Material	Dimensional Change (%)							
	Unaged	Fluid 1 B20 Soy Blend	Fluid 2 B20 RME Blend	Fluid 3 B5 Oxidised Soy Blend	Fluid 4 B20 Oxidised Soy Blend	Fluid 5 Basefuel	Fluid 6 B5 RME Blend	Fluid 7 B5 Soy Blend
NO674-70	0.2	4.0	3.9	5.0	9.0	3.7	3.6	3.5
NB104-75	-0.3	5.1	6.1	6.6	9.9	4.1	4.4	4.7
KB162-80	-0.4	4.5	4.2	5.6	8.6	3.8	4.6	3.6
VB153-75	-0.3	0.5	0.7	0.9	1.2	0.6	0.7	0.5
V1164-75	-0.4	0.5	0.4	0.9	1.3	0.8	0.6	0.9

APPENDIX 8

Determination of Compression Set Properties of 'O' Rings

ASTM D1414

Material	Compression Set (% change)							
	Unaged	Fluid 1 B20 Soy Blend	Fluid 2 B20 RME Blend	Fluid 3 B5 Oxidised Soy Blend	Fluid 4 B20 Oxidised Soy Blend	Fluid 5 Basefuel	Fluid 6 B5 RME Blend	Fluid 7 B5 Soy Blend
NO674-70	8.7	14.3	15.2	2.6	16.0	10.2	12.1	19.7
NB104-75	8.5	-2.6	-1.9	-2.2	-9.3	32.4	3.0	-0.4
KB162-80	9.4	3.4	12.2	4.5	-3.1	3.4	5.2	-3.8
VB153-75	29.3	44.7	32.2	57.4	24.5	31.4	40.6	31.7
V1164-75	13.9	9.5	10.0	8.1	9.0	4.7	10.0	9.3

APPENDIX 9

Determination of Tensile Properties of 'O' Rings

ASTM D1414

Material	Tensile Properties							
	Unaged		Fluid 1 B20 Soy Blend		Fluid 2 B20 RME Blend		Fluid 3 B5 Oxidised Soy Blend	
	Tensile Strength (MPa)	Elongation at Break (%)	Tensile Strength (MPa)	Elongation at Break (%)	Tensile Strength (MPa)	Elongation at Break (%)	Tensile Strength (MPa)	Elongation at Break (%)
NO674-70	17.2	605	14.0	380	14.4	405	13.3	385
NB104-75	15.6	395	3.1	120	4.6	170	2.3	80
KB162-80	21.8	255	14.8	160	12.8	160	16.7	175
VB153-75	10.2	340	9.9	300	9.8	290	8.9	280
V1164-75	11.2	270	11.2	270	11.5	280	11.8	290

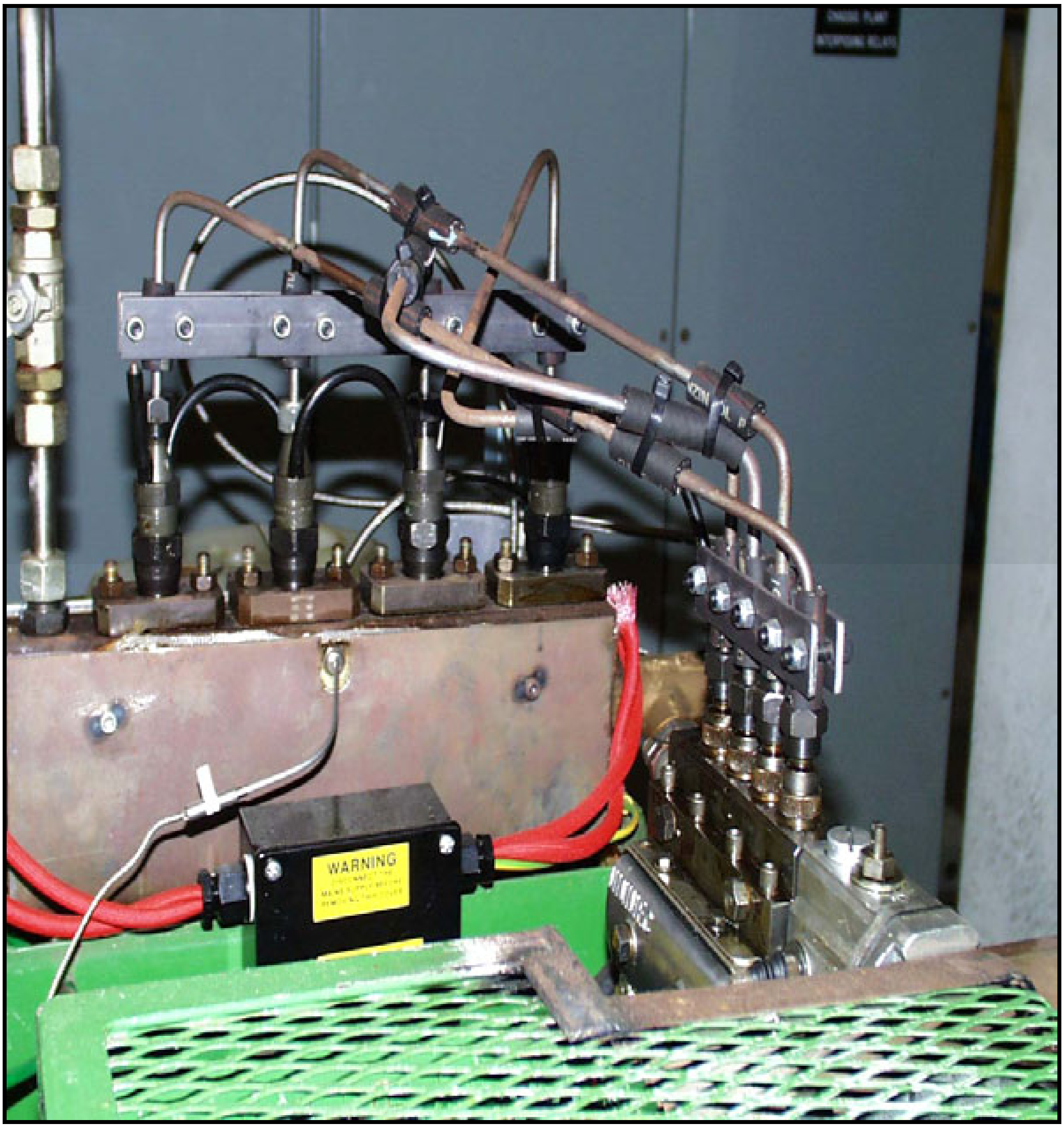
Material	Tensile Properties							
	Fluid 4 B20 Oxidised Soy Blend		Fluid 5 Basefuel		Fluid 6 B5 RME Blend		Fluid 7 B5 Soy Blend	
	Tensile Strength (MPa)	Elongation at Break (%)	Tensile Strength (MPa)	Elongation at Break (%)	Tensile Strength (MPa)	Elongation at Break (%)	Tensile Strength (MPa)	Elongation at Break (%)
NO674-70	9.7	300	15.2	400	11.3	265	12.0	280
NB104-75	3.7	135	10.6	275	10.7	270	2.6	100
KB162-80	14.8	175	19.3	200	15.6	165	11.9	145
VB153-75	8.2	265	11.1	310	9.4	280	10.0	300
V1164-75	11.1	270	11.6	265	10.5	250	11.5	275

Material	Percentage Change in Tensile Properties							
	Unaged		Fluid 1 B20 Soy Blend		Fluid 2 B20 RME Blend		Fluid 3 B5 Oxidised Soy Blend	
	Tensile Strength (MPa)	Elongation at Break (%)	Tensile Strength (MPa)	Elongation at Break (%)	Tensile Strength (MPa)	Elongation at Break (%)	Tensile Strength (MPa)	Elongation at Break (%)
NO674-70	-	-	-18.6	-37.2	16.3	-33.1	-22.7	-36.4
NB104-75	-	-	-80.1	-6.96	-70.5	-57.0	-85.3	-79.7
KB162-80	-	-	-32.1	-37.3	-41.3	-37.3	-23.4	-31.4
VB153-75	-	-	-2.9	-11.8	-3.9	-14.7	-12.7	-17.6
V1164-75	-	-	0.0	0.0	2.7	3.7	5.4	7.4

Material	Percentage Change in Tensile Properties							
	Fluid 4 B20 Oxidised Soy Blend		Fluid 5 Basefuel		Fluid 6 B5 RME Blend		Fluid 7 B5 Soy Blend	
	Tensile Strength (MPa)	Elongation at Break (%)	Tensile Strength (MPa)	Elongation at Break (%)	Tensile Strength (MPa)	Elongation at Break (%)	Tensile Strength (MPa)	Elongation at Break (%)
NO674-70	-43.6	-50.4	-11.6	-33.9	-34.3	-56.2	-30.2	-53.7
NB104-75	-76.3	-65.8	-32.1	-30.4	-31.4	-31.6	-83.3	-74.7
KB162-80	-32.1	-31.4	-11.5	-21.6	-28.4	-35.3	-45.4	-43.1
VB153-75	-19.6	-22.1	8.8	-8.8	-7.8	-17.6	-2.0	-11.8
V1164-75	-0.9	0.0	3.6	-1.9	-6.2	-7.4	2.7	1.9

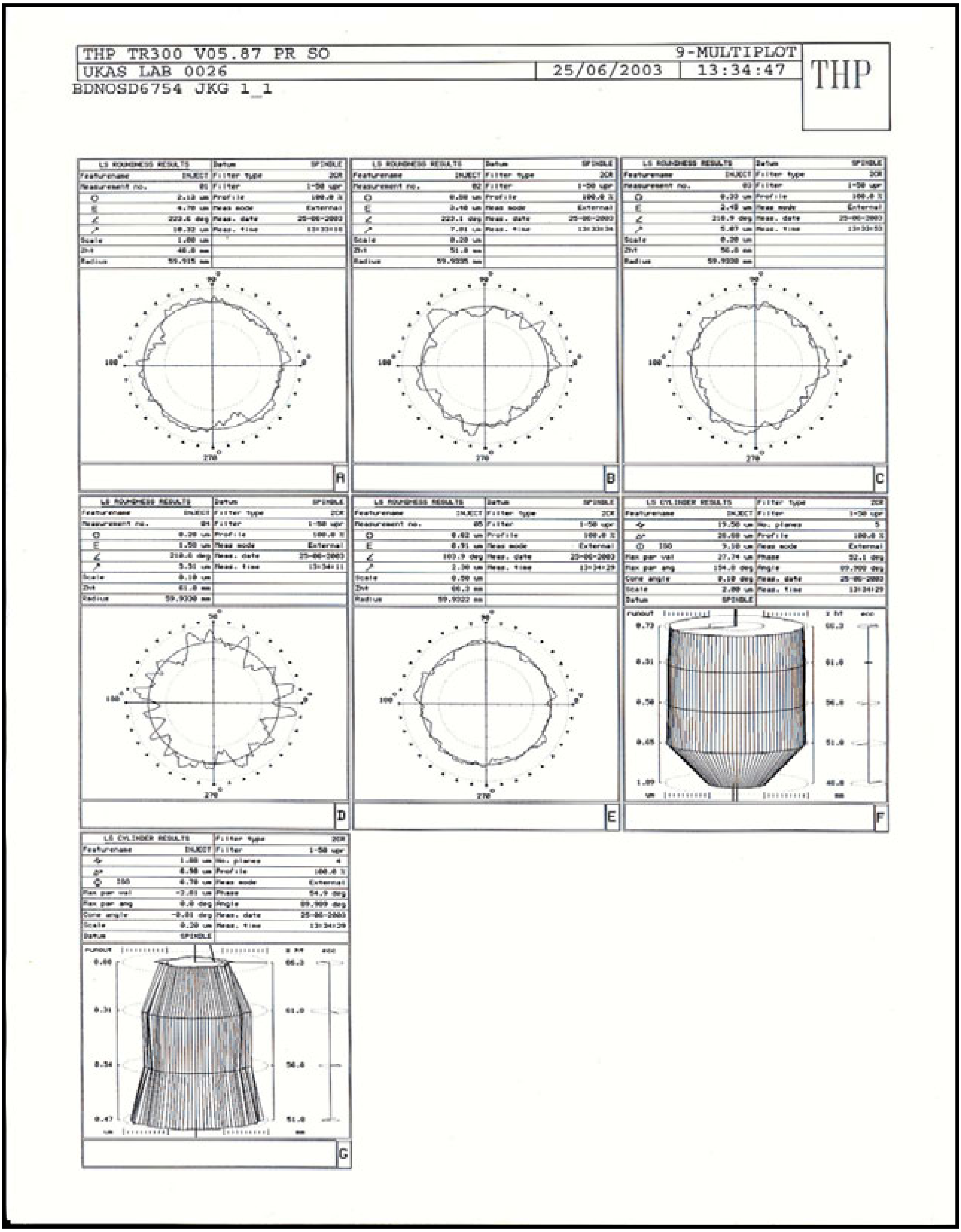
APPENDIX 10

Injector Wear Test Rig



APPENDIX 11

Injector Needle Measurements



APPENDIX 12

'Out of Roundness' Injector Measurements

Table 1 – Pre-Test 'Out of Roundness' Measurements, (µm)

Test Fuel	Injector I.D.	Plane 1		Plane 2		Plane 3		Plane 4		Plane 5	
		Pre test	Post test	Pre test	Post test	Pre test	Post test	Pre test	Post test	Pre test	Post test
Basefuel	**1-1**	2.13	1.43	0.50	0.45	0.33	0.33	0.28	0.35	0.62	0.62
Basefuel	**1-2**	1.32	1.46	0.44	0.54	0.54	0.39	0.47	0.31	1.23	0.39
Basefuel	**1-3**	0.88	0.70	0.56	0.47	0.36	0.31	0.85	0.55	0.99	0.97
Basefuel	**1-4**	1.81	1.27	0.72	0.45	0.43	0.33	0.57	0.30	0.91	0.41
B20 RME	**2-1**	1.45	1.38	0.55	0.59	0.41	0.42	0.48	0.43	0.76	0.65
B20 RME	**2-2**	0.28	1.00	0.80	0.46	0.10	0.31	0.15	0.41	0.28	0.89
B20 RME	**2-3**	0.35	0.92	0.37	0.41	0.11	0.38	0.13	0.40	0.21	0.65
B20 RME	**2-4**	1.07	0.99	0.70	0.58	0.40	0.35	0.49	0.54	0.72	0.65
B20 Soy Biodiesel	**3-1**	0.70	1.39	0.22	0.46	0.17	0.34	0.21	0.41	0.37	0.76
B20 Soy Biodiesel	**3-2**	4.18*	1.28	0.70	0.51	0.41	0.34	0.70	0.37	0.74	0.64
B20 Soy Biodiesel	**3-3**	1.10	0.85	0.69	0.73	0.80	0.50	0.46	0.50	0.88	0.77
B20 Soy Biodiesel	**3-4**	0.64	2.04	0.20	0.49	0.16	0.41	0.16	0.73	0.22	0.95
B20 Soy Biodiesel (oxidised)	**4-1**	1.09	0.94	0.74	0.74	0.60	0.43	0.78	0.37	1.53	0.46
B20 Soy Biodiesel (oxidised)	**4-2**	2.14	1.55	0.59	0.48	0.53	0.45	0.60	0.50	0.93	0.72
B20 Soy Biodiesel (oxidised)	**4-3**	1.53	0.96	0.63	0.58	0.52	0.46	0.43	0.34	0.65	0.52
B20 Soy Biodiesel (oxidised)	**4-4**	1.43	1.25	0.92	0.80	0.64	0.62	0.65	0.67	0.98	0.94

* Outlier determined by Dixons test

Table 2 – Pre-test distribution

	Pre test distribution ALL
Maximum	0.92
Minimum	0.10
Average	0.48
σ	0.22
Mean + 2 σ	0.92
Mean – 2 σ	0.05

Table 3 – Post Test distribution

	Base fuel	B20 RME	B20 SME	B20 SME oxidised
Maximum	0.55	0.59	0.73	0.80
Minimum	0.30	0.31	0.34	0.34
Average	0.40	0.44	0.48	0.54
σ	0.09	0.09	0.13	0.15
Mean + 2 σ	0.58	0.62	0.74	0.83
Mean – 2 σ	0.22	0.26	0.22	0.25

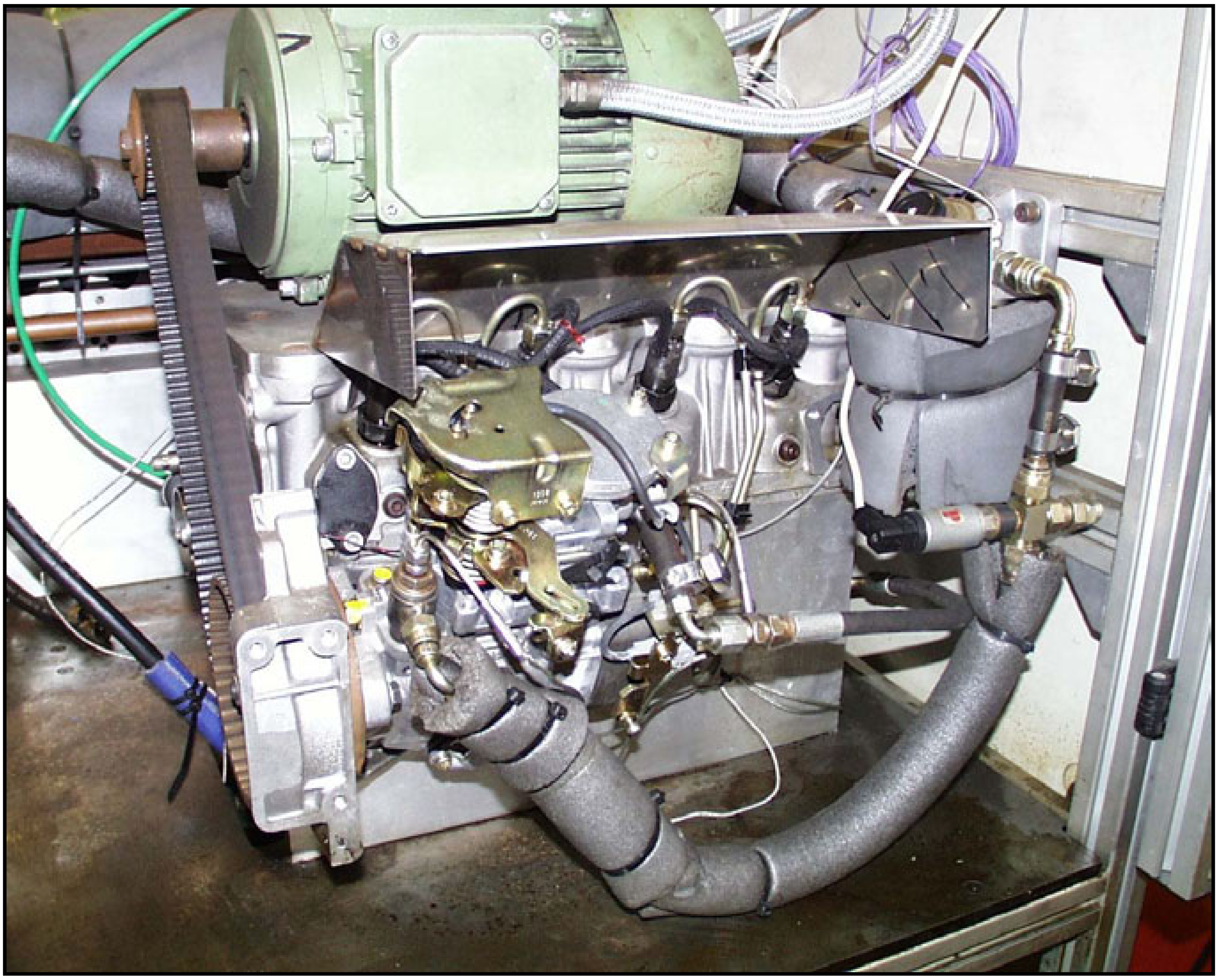
APPENDIX 13

Wear Rating Assessment of Bosch VE Rotary Test Pumps, EL-80

	Base fuel	B5 RME	B5 SME	B5 SME Oxidised	B20 RME	B20 SME	B20 SME Oxidised
Pump I.D	54050	60321	54035	60315	50352	60323	53970
Camplate							
Path	2	2	2	2	2.5	2	2.5
Centre	2	2	2	2.5	2	2	2
Claws	2.5	2	2	2	2	2	2
Plunger button	2.5	2	2.5	2	2	2	2
Rollers	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Roller bolts	3	3	2.5	2.5	2.5	2.5	2.5
Governor							
Fly weights	2	2	2.5	2.5	2.5	2	2
Washer ring	2	2	3	3	2.5	2.5	2.5
Supply pump							
Blades	2	2.5	2.5	3	3	3	2
Raceway	2	2	2	2	2	2	2.5
Overall Rating	2.5	2.5	2.5	3	3	3	Fail (2.5)

APPENDIX 14

Bosch VE Rotary Pump Wear Test Rig



APPENDIX 15

Wear Rating Assessment of Bosch Common Rail Test Pumps

	Base fuel	B5 RME	B5 SME	B5 SME Oxidised	B20 RME	B20 SME	B20 SME Oxidised (Batch 2)	B20 SME Oxidised (Batch 1)
Pump I.D.	6393	6437	6291	6218	6438	6433	3374	3159
Driveshaft Eccentric surface	1.5	1.5	1	1.5	1.5	1.5	1	1
Plunger feet	1	1	1	1	1	1	1	1
Plunger	1	1	1	1	1	1	1	1
Drive shaft cam	1	1	1	1.5	1.5	1	1.5	1.0
Drive shaft bearing surfaces	1	1	1	1.5	1.5	1	1.5	1.5
Valve chamber	1.5	1.5	1.5	1.5	1.5	3	1.5	1.5
Suction valves	3	1.5	3	3	3	3	3	2
Overall Rating	2	1.5	1.5	2	2	2.5	1.5	1.5

Rating Guide

Pump Part	Normal Size	Wear	Reduced Life	Wear	Early Failure	Wear
Driveshaft Eccentric surfaces	Polishing wear	<5µm	Scuffing and Pitting	>5µm - 100µm	Fatigue	>100µm
Plunger feet	Polishing wear	<1µm	Scuffing and Pitting	>1µm - 10µm	Fatigue	>10µm
Plunger pistons	Polishing wear	<1µm	Polishing wear and scuffing	>1µm - 2µm	Scuffing	>2µm
Drive shaft cam	Polishing wear	<1µm	Polishing wear and scuffing	>1µm - 10µm	Scuffing	>10µm
Drive shaft bearing surfaces	Polishing wear	<1µm	Polishing wear and scuffing	>1µm - 10µm	Scuffing	>10µm
Valve chamber	Polishing wear	<1µm	Polishing wear and scuffing	>1µm - 10µm	Scuffing	>10µm
Suction valves	Polishing wear	<1µm	Polishing wear and scuffing	>1µm - 10µm	Scuffing	>10µm

Common Rail Pump Wear Test Rig – Front View

APPENDIX 17

EL-79

Diesel Injector Nozzle Wear Test

Method N°	EL-79
Test Method	Diesel Injector Nozzle Wear Test
Rig / Engine	Test Rig

Status	Approved: T Donovan
Issue N°	1
Date of last modification	08-AUG-03

01 CONTENTS

Subject	Introduction
N°	EL-79 - 02
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Author	Approved by
I P Bradbury	T J Donovan

Record of Changes and Amendments

Issue N°	Date of Issue	Summary of Changes
1	08-AUG-03	new release

CONTENTS of section **02 - Introduction**

02.1 Foreword

With the reduction in diesel fuel sulphur levels there has been increased concern over the lubricity of the fuel and the affect that this may have on diesel fuel injection equipment. Diesel fuel injectors are totally lubricated by the fuel and so are susceptible to increased levels of wear when low sulphur fuels are used.

02.2 Area of application

This test method provides the means for evaluating the lubricity performance of fuel blends, fuel blending components and fuel additives in terms of their effect on the wear of light duty diesel injectors.

02.3 Test equipment

Any suitable motorised rig can be used for this test provided it can be operated according to the conditions specified.

02.4 Short description

A four cylinder in-line diesel injection pump is operated at a constant speed of 14 rev/min, for a total of 500 hours ± 10 hours, with the governor locked in position to provide a fixed rate of fuel delivery. Injector needles are measured and profiled before and after running the test. The differences in these measurements constitute the wear relating to the product under test. Reference products can be used to demonstrate acceptable levels of wear.

02.5 Definitions

Terms used throughout the method are defined as follows:

<u>Fuel Injection Equipment (FIE)</u>

This relates to any item of equipment used to inject fuel into a diesel engine. For example, the high pressure injection pump, the injectors, injection nozzles, supply or feed pumps, high pressure pipes.

<u>Light Duty Diesel Engine</u>

This relates to any diesel engine used in passenger cars or light commercial vehicle applications.

Subject	Health and Safety
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CONTENTS of section **03 - Health and Safety**

03.1 Health, Safety and Environment

Health and safety is the responsibility of each laboratory using this test procedure.

It has been assumed by the compilers of this test method that anyone using the method will either be fully trained and familiar with all normal engineering and laboratory practice, or will be under the direct supervision of such a person.

All rotating machinery and hot metal-work is to be properly guarded, fuel systems must be protected against fire risk and used oil and solvents must be disposed of in a responsible manner.

Suitable precautions must be taken in relation to the operation and testing of high pressure fuel injection systems.

Further hazards associated with this test are those normally associated with test rigs and the operation of rotating machinery, for which normal good engineering practice should be observed.

It is the responsibility of the company operating the motorised test rig to ensure that all local legislative and statutory requirements are met.

The test facility must only be operated in compliance with the pertinent European Community and local mandatory requirements governing the safe operation of rotating equipment.

Any emissions into the atmosphere, the noise and the health risk for the staff should be kept at the lowest possible level.

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CONTENTS of section 04 - Installation

04.1 Test rig

It is intended that any suitable rig could be used for testing provided it is capable of meeting the conditions specified. It would be necessary for the drive motor speed and torque to be matched to the requirements of the chosen injection pump. Additionally, suitable control and data logging systems must be made available.

04.2 EQUIPMENT PREPARATION

04.2.1 PREPARATION OF THE TEST RIG

The test rig should be equipped with a suitable fuel injection pump and heated injector block. The rig should be visually checked for integrity of the various drive and fuel supply components. The lubricant within the injection pump must be changed before the start of each test and subsequently every 100 hours along with each fuel change.

04.2.2 FUEL SYSTEM

The fuel supply to the injection pump is provided by a feed pump which transfers fuel from the storage tank. The main injection pump provides the high pressure fuel to operate the injectors. The injectors are mounted in a heated block to simulate normal operating temperatures within an engine. The fuel injected is collected and returned to the fuel storage tank. The delivery of each injection pump element is to be measured at the start and end of each test. This is to ensure that no appreciable wear of the injection pump has taken place. An in-line injection pump, with oil lubricated cam mechanism, is recommended as they are less susceptible to wear from poor lubricity fuels.

04.2.3 INSTRUMENTATION

<u>Speed</u>
The injection pump is operated at a constant speed and should be checked at the start and end of each test.

<u>Temperatures</u>

The injector block is heated to 150 °C ±10°C and must be monitored throughout the test. Suggested minimum logging frequency is once per hour.

The bulk fuel temperature is controlled to 40 °C ±5°C and should be monitored throughout the test. Suggested minimum logging frequency is once per hour.

The fuel temperature at the injection pump inlet is to be monitored throughout the test. Suggested minimum logging frequency is once per hour.

<u>Pressures</u>

Fuel supply pressure must be controlled and monitored. There is a nominal feed pressure of 0.5 bar gauge. If this falls below 0.3 bar gauge pressure then the test rig should be stopped automatically as there is a risk of the injection pump and injectors seizing. Suggested minimum logging frequency is once per hour.

Details of accuracy and calibration, for the above instrumentation, are given in chapter **05 Calibration**.

Subject	Calibration
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Record of Changes and Amendments

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CONTENTS of section **05 - Calibration**

05.1 Measuring Equipment

All measuring systems required for this test should comply with European Quality Assurance Requirements ISO 9000

Parameter	Function	Recommended measurement range	Measurement units	Measurement system minimum accuracy requirement
Temperature	Injector block	0 - 300	°C	± 2.5
Temperature	Fuel	0 - 300	°C	± 2.5
Pressure	Injector opening	10-250	bar (abs)	± 1.0
Pressure	Fuel supply	0 - 5	bar (abs)	± 0.1
Speed	Injection Pump	0 - 2000	rev/min	± 10

05.2 Calibration Frequency

The test rig should be totally re-calibrated at least once per year.

If an un-calibrated component is replaced in a measuring chain, then that chain shall be re-calibrated.

An implausible channel record should initiate a failure analysis, including a calibration check and re-calibration if required.

General note

Staff carrying out calibration must be competent personnel, adequately trained.

All prescribed channels should be calibrated, using equipment that is traceable to national standards. Current calibration records must be maintained for inspection. Historical calibration records must be kept for a period of not less than six years.

Subject	Test Preparation
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1	08-AUG-03	new release

CONTENTS of section **06 - Test Preparation**

06 TEST PREPARATION

06.1 Test rig preparation

The test rig should be prepared prior to the start of each test in the following manner:

The lubricating oil in the injection pump must be replaced using an appropriate quantity of Shell Rimula X 10W/30 diesel engine oil. The pump must be filled to the level determined by the level plug or using other information provided by the pump manufacturer.

The delivery volume from each of the injection elements should be measured at the start and end of each test. This is to ensure that no appreciable wear of the injection pump has occurred throughout the test. The fuel injection pump delivery should be 65 ± 5ml, from each element, measured at the normal rig operating speed of 1440 rev/min over a 3 minute time period. If the fuel pump delivery is incorrect then the pump must be examined for wear or damage and repaired or replaced accordingly.

The operating speed of the injection pump should be checked, as a minimum, at the start and end of each test.

06.2 Injector preparation

New injector nozzles must be used for each test. Prior to test, the needles from the test nozzles must be measured and profiled around their complete circumference and at a minimum of three positions along their length. All measurements must be made on the part of the needle which comes into contact with the nozzle body.

The injector nozzles are to be assembled and installed into the injectors using standard good practices for assembling diesel fuel injection equipment. The injectors must then be checked for opening pressures, spray pattern and leakage. The opening pressures must be adjusted to 130 bar ±5 bar. Calibration fluid as detailed in the reference fluids section must be used for all injector checks and adjustments.

After installing the injectors into the test rig, the fuel system should be bled to remove all air prior to start of the test.

06.3 Fuel system flushing

The fuel tank must be thoroughly drained and flushed with 20 litres of test fuel prior to installing the test fuel. Once flushed, 40 litres of test fuel is to be installed for the test along with a new fuel filter. Subsequent fuel changes within a test do not require the system to be flushed or the filter to be replaced.

Subject	Test Operation
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CONTENTS of section **07 - Test Operation**

07.1 Test Cycle

The rig is operated at a constant speed of 1440 rev/min ±25 rev/min for a total period of 500 hours ±10 hours.

The injector block is heated to a temperature of 150°C ±10 °C to simulate normal operating conditions of the injectors.

The bulk fuel temperature is controlled to a temperature of 40°C ±5 °C. It is permissible for the bulk fuel temperature to outside these limits for the first 3 hours after start up and after a fuel change has been conducted.

07.2 Fuel Changes

After completion of 100 ± 10 hours running, the rig is stopped and isolated. The fuel is then drained and replaced with 40 litres of fresh test fuel. This procedure is repeated after each subsequent 100 hours running until the completion of 500 hours ± 10 hours when the fuel is drained in preparation for the next test.

Along with each 100 hour fuel change, it is important that the injector pump lubricant is also changed.

07.3 Stoppages during the test

It is permissible to interrupt a test for a period of up to 72 hours, within each 100 hour testing interval between fuel changes.

07.4 Test Referencing

Acceptable levels of wear are determined using a reference fuel having known good performance. A borderline pass reference fuel suitable for this purpose is detailed in the reference fluids section.

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CONTENTS of section **08 - Test Evaluation**

08.1 Test Evaluation

At the end of the test the injectors are removed and checked for opening pressures, spray pattern and leakage. The opening pressures and any unusual observations to be recorded. The injectors are then dismantled and the nozzles and needles washed in n-heptane. The needles are then measured and profiled in the same manner as the pre-test measurements. The difference between the pre and post test measurements determines the amount of wear.

If the injector nozzles are to be stored before measuring, they must be coated with a suitable corrosion inhibitor; e.g. engine lubricating oil.

The test result for a particular fuel blend is the average, for all four needles, of the relevant wear measurements and surface profiles.

This result can be compared with a reference product of known field performance to determine comparative severity.

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CONTENTS of section **09 - Test Report and Validation**

09.1 Contents of Test Report

A complete test report should be produced for each test, however, dependant on customer requirements, a shortened version may be sufficient.

The test report is made up of three sections as follows:

09.2 General information

- Name and address of testing laboratory
- Name and address of client
- Unique identification of report
- Identification of each page and total number of pages of the report
- Date if receipt if the test item; i.e. test fuel / additive
- Date of issue of the test report
- Signature and legible name of the approved signatory
- Test fuel code
- Test additive code (where applicable)
- Description of test procedure
- Details of component preparation
- Test results
- Any other information relevant to the validity of the test as requested by the client; e.g. reference data.

09.3 Operational data

- Pre-test rig measurement and set up data
- Operational data obtained throughout the test
- End of test rig measurement and test data
- Report of all instances of operations outside specified limits
- Record of all unusual occurrences

09.4 Test Evaluation

- Injector needle measurements and profiles, pre and post test
- Calculated differences between pre-test measurements and post-test measurements
- Comparison with reference tests

Subject	Reference Fluids
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CONTENTS of section 10 - Reference Fluids

10.1 Fuel

A borderline pass reference fuel would consist of any diesel fuel meeting EN 590 specifications and having a lubricity value of between 400µm and 460µm as measured using the HFRR test ISO 12156-1.

10.2 Injection pump lubricant

Use Shell Rimula 10W/30 diesel engine lubricant.

10.3 Injector calibration fluid

The injectors must be tested and adjusted using calibration fluid conforming to ISO 4113.

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CONTENTS of section 11 - Referencing

11.1 Purpose of Reference Tests

Reference tests on a borderline pass fuel enable the normal or acceptable levels of wear to be established. A potential reference fuel is described in section 10. However, depending on the particular work programme, it may be more appropriate to use alternative referencing products.

11.2 Frequency of Reference Tests

Reference tests should be included in each test programme or after every 10 product evaluation tests where single tests are conducted.

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CONTENTS of section 12 - Documentation

12.1 Documentation of Calibration

The laboratory should prepare a calibration document that comprises all calibration data (see **Section 05**).

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CONTENTS of section 13 - Accreditation

13.1 Accreditation

This test has been developed by the Associated Octel Company Limited and is not accredited by any external organisation.

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CONTENTS of section 14 - Reference Data Base

14.1 Reporting of Results

The test results shall be reported for each injector needle as:

Pre and post test measurements and calculated average values for an individual injector needle

Any calculated differences between pre and post test measurements

Average values for all injector needles from a specific test, should also be reported.

14.2 Precision of the Method

The precision of the method is based on the average measurement, for all four injector needles, of a specific measurement e.g. ‘roundness’. Precision data should be based on using marginal pass or fail reference fuels so as to correctly reflect the test precision under such marginal conditions.

Currently no data are available regarding the precision of the method.

14.3 Table of Precision Data

Date	Base Fuel	Additive	Average roundness (µm)

APPENDIX 18

EL-80

Diesel Injection Pump Wear Test

Method N°	EL-80
Test Method	Diesel Injection Pump Wear Test
Rig / Engine	Test Rig and Bosch VE4 injection pump

Status	Approved: T Donovan
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01 CONTENTS

Subject	Introduction
N°	EL-80 - 02
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CONTENTS of section **02 - Introduction**

02.1 Foreword

With the reduction in diesel fuel sulphur levels there has been increased concern over the lubricity of the fuel and the affect that this may have on diesel fuel injection equipment. Some diesel fuel pumps are totally lubricated by the fuel and so are susceptible to increased levels of wear when low lubricity fuels are used.

Some companies have recognised that the simple HFRR lubricity test does not measure all the lubricity phenomena that affect a diesel injection pump. Furthermore, the HFRR test does not always provide a good correlation with field experience, especially for additised fuels. The pump lubricity test provides information which is much closer to real life and offers a more complete assessment of a fuel's lubricity performance.

02.2 Area of application

This test method provides the means for evaluating the lubricity performance of diesel fuel blends, fuel blending components and fuel additives, in terms of their effect on the wear and longevity of light duty rotary diesel injection pumps.

02.3 Test equipment

Any suitable motorised rig can be used for this test provided it can be operated according to the conditions specified. The test uses any four cylinder Bosch VE pump designed for use with indirect injection (IDI) engines and with mechanical governor mechanism.

02.4 Short description

A four cylinder Bosch VE diesel injection pump is operated under stop start cyclic conditions, for a total of 1000 hours. The frequent stops ensure that hydrodynamic lubrication, of the various components, is not maintained and so is more representative of real-life operation. At the end of the test, the injection pump is dismantled and critical components are rated for wear against a rating scale. The rating scale includes normal or acceptable amounts of wear which were based on the road trial experience of Robert Bosch GmbH.

02.5 Definitions

Terms used throughout the method are defined as follows:

Fuel Injection Equipment (FIE)

This relates to any item of equipment used to inject fuel into a diesel engine. For example, the high pressure injection pump, the injectors, injection nozzles, supply or feed pumps, high pressure pipes.

Light Duty Diesel Engine

This relates to any diesel engine used in passenger cars or light commercial vehicle applications.

Rotary Injection Pump

This refers to the design of the fuel injection pump which uses a common rotating shaft to operate the high pressure pumping element as well as distribute the fuel to the relevant injector. These designs of pumps are invariably fuel lubricated and so are more sensitive to changes in fuel lubricity.

In-direct Injection (IDI)

This refers to the design of the diesel engine where the fuel is injected into a pre-combustion chamber, rather than the main cylinder. These engines have increased amounts of air turbulence and so generally have lower injection pressures than the direct injection engines.

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CONTENTS of section **03 - Health and Safety**

03.1 Health, safety and environment

Health and safety is the responsibility of each laboratory using this test procedure.

It has been assumed by the compilers of this test method that anyone using the method will either be fully trained and familiar with all normal engineering and laboratory practice, or will be under the direct supervision of such a person.

All rotating machinery and hot metal-work is to be properly guarded, fuel systems must be protected against fire risk and used oil and solvents must be disposed of in a responsible manner.

Suitable precautions must be taken in relation to the operation and testing of high pressure fuel injection systems.

Further hazards associated with this test are those normally associated with test rigs and the operation of rotating machinery, for which normal good engineering practice should be observed.

It is the responsibility of the company operating the motorised test rig to ensure that all local legislative and statutory requirements are met.

The test facility must only be operated in compliance with the pertinent European Community and local mandatory requirements governing the safe operation of rotating equipment.

Any emissions into the atmosphere, the noise and the health risk for the staff should be kept at the lowest possible level.

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CONTENTS of section 04 - Installation

04.1 Test rig

It is intended that any suitable test rig could be used for testing provided it is capable of meeting the conditions specified. It would be necessary for the drive motor speed and torque to be matched to the requirements of the chosen injection pump. Additionally, suitable control and data logging systems must be made available.

04.1.1 Pump speed

It is recommended that the injection pump is driven by an electric motor covering a speed range from 0 rev/min to 3000 rev/min. To overcome the initial starting torque of the injection pump a 7.5 kW electric motor is recommended, although a motor of lower power output may be acceptable.
Speed control is probably best achieved using a frequency controlled device.

04.1.2 Pump load

The pump is operated with the fuel control lever locked in the maximum fuel position.

04.1.3 Fuel circuit

The amount of fuel circulating in the entire fuel system is 40 –5 litres.

The fuel injection circuit is designed to simulate a vehicle fuel system.

The fuel system must include a replaceable fuel filter.

The injectors recommended are of the indirect injection design with corresponding nozzles. VW injectors are one suggestion which use nozzles with part number: 068 130 201 RX. The injectors must be checked for spray pattern, leaks and opening pressure at the start of each test. The opening pressures must be set to 130 ±5 bar absolute.

The delivered fuel must be injected via injection pipes into a collection tank, from where the fuel is recirculated back to the fuel tank or reservoir. The injection pipes must be manufactured from steel pipe having a wall thickness of 1.5 mm minimum and to a length of 400 mm ± 50 mm. All pipes must be within 10mm of the same length.

The fuel has to be delivered to the injection pump air-free (without bubbles) and at a pressure of 0.3 to 0.5 bar gauge pressure. A suitable pressure monitoring system should be installed in the injection pump feed line to monitor this pressure. The rig should shut down in the event of low pressure, i.e. 0.2 bar or below. This will prevent damage to the test components in the event of a fuel supply failure.

04.1.4 Fuel conditioning system

The fuel has to be conditioned to a temperature of 60°C ±5 at the injection pump outlet by means of an indirect heating unit (heat exchanger) providing a maximum surface temperature of 75°C, in order not to cause any oxidation problems or thermal cracking of the test fuel.

The conditioning system must be capable of enabling the set value of 60°C to be reached within a maximum of 3 hours running time.

The fuel system components which come into contact with the fuel, including conditioning heat exchangers, pipe-work, fittings, etc must not contain any parts made from copper and copper alloys, e.g. brass, as these materials are known to react with and change the properties of some fuels. Stainless steel is recommended.

04.1.5 Fuel supply control

The fuel supply must be uninterrupted. It is essential to ensure that the injection pump is not allowed to rotate without a fuel supply. The pump rig must be stopped immediately (automatically) if any failure occurs in the fuel supply system.

04.1.6 Measurement, location, uncertainty

Parameter	**Location**	**Typical Range**	**Uncertainty** *See Section 5 for any special requirements*
Pump Speed	Automation system	0 to 3000 1/min	B
Fuel Outlet Temperature	Within 25mm of the fuel outlet connection.	0 to 100 °C	A
Fuel Inlet Temperature (optional)	Within 25mm of the fuel inlet connection.	0 to 100 °C	B
Fuel Feeding Pressure	In the fuel feed pipe before the injection pump inlet, but after any potential restrictions such as fuel filter, etc.	0-1 bar (gauge)	B

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CONTENTS of section **05 - Calibration**

5.1 Uncertainty of measurement

Two classes of measurement uncertainties have been defined for each parameter.

Class A Which demands the current cost effective technologies of measurement equipment has to be applied to all controlled parameters of the test.

Class B Which can accept a lower degree of sophistication of the measurement equipment and is applied to all test parameters specified as "record" or "check" values.

Other more specialised requirements must be included as per section 4

PARAMETER	UNIT	RANGE	Class A	Class B
Engine speed	rev/min	0 to 7500	± 10	± 10
Engine torque	Nm	0 to 100 100 to 200 200 to 400 400 to 1000 1000 to 2000 2000 to 4000	± (1 + 1 % M) *1) ± (2 + 1 % M) ± (4 + 1 % M) ± (10 + 1 % M) ± (20 + 1 % M) ± (40 + 1 % M)	± (1 + 2 % M) ± (2 + 2 % M) ± (4 + 2 % M) ± (10 + 2 % M) ± (20 + 2 % M) ± (40 + 2 % M)
Temperature	°C	- 10 to 200 200 to 375 375 to 1200	± 1.5 ± 2 ± 15	± 3 ± 4 ± 25
Atmospheric pressure	mbar	800 to 1200	± 5	± 10
Pressure	mbar mbar bar	0 to 3000 (abs) -1000 to 2000 (gauge) 2 to 10	± 15 ± 15 ± 0.1	± 25 ± 25 ± 0.2
Fuel flow	kg/h	0-100	± (0.1 + 1 %)	± (0.1 + 5 %)
Total consumption	kg or litre		± 1.0 %	± 5.0 %
Blow-by flow	l/min	0 to 300	± 5.0	± 10.0
Coolant flow	l/min	0 to 500	± (1 + 3.0 %)	± (1 + 5.0 %)
CO content	%	0 to 10	± 0.2	± 0.5

*1) M : maximum value during test

Notes: No units means uncertainty of measurement in the same unit as parameter, % means percent of reading

The limits of measurement uncertainty defined in the above table include all upstream calibration uncertainties associated with the traceability chain

5.2 CALIBRATION INTERVAL

Standard documents (ISO 9000 or EN29000) do not give any advice on what this interval must be. They only require that the calibration is done on a regular basis. The interval between two calibrations must be adjusted according to:

- The experience

- The quality of the measuring chains

- The frequency of use

Each time an unexpected measuring result is obtained, a re-calibration of the measuring chain must be done.

5.3 STANDARDS / DEFINITIONS

Standards

- ISO 9001 (§ 4.11) (equivalent to EN 29001) about equipment requirements,

- NFX 07-001 (equivalent to DIN 1984-945) for the vocabulary.

- UKAS M3003 The expression of uncertainty and confidence in measurement for calibrations.

Definitions

Uncertainty of measurement traceability (*)	Parameter, associated with the result of a measurement, that characterises the dispersion of the values that could reasonably be attributed to the measure and:

Notes :

The parameter may be, for example, a standard deviation (or a given multiple of it), or the half-width of an interval having a stated level of confidence.

Uncertainty of measurement comprises, in general, many components. Some of these components may be evaluated from the statistical distribution of the results of series of measurements and can be characterised by experimental standard deviations. The other components, which can also be characterised by standard deviations, are evaluated from assumed probability distributions based on experience or other information.

It is understood that the result of the measurement is the best estimate of the value of the measure and, and that all components of uncertainty, including those arising from systematic effects. Such as components associated with corrections and reference standards, contribute to the dispersion.

Traceability (*)	Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties.

Notes :

The concept is often expressed by the adjective traceable The unbroken chain of comparisons is called a traceability chain.

(*) extracted from NFX.07.001

Operational Range	The widest range of values (difference between the maximum & minimum values) anticipated to be seen for any given parameter throughout the duration of the test.
Sensor/Measuring Chain	All items hardware or virtual with the potential to affect the reported value in engineering units. For example: the sensor, cabling, signal conditioning, ADC circuitry & resolution configured range, display/logging resolution.
Sensor Calibration Range	The maximum recommended range over which each specific sensor should be calibrated. The defined sensor calibration range may be either single sided (i.e. all +ve or all -ve values), or may be bi-linear (i.e. output changes from -ve to +ve values).
Calibrated Range	The range (minimum & maximum values) over which each specific sensor/measuring chain is calibrated. The configured range of the measuring chain would not normally exceed the sensor calibration range.
Full Scale Deflection (FSD)	The maximum single sided value for each measuring chain having a calibrated range intercepting at or through zero.

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CONTENTS of section **06 - Test preparation**

06.1 Pump specification

This test is based on the Bosch VE four cylinder injection pump with mechanical governor mechanism, designed for use with indirect injection (IDI) engines. The test pump must be new and not reconditioned. Alternatively a rebuilt pump may be used provided all the rated test components are new and other components are in a serviceable condition.

06.2 Pump preparation

Prior to test, the pump fuel control lever must be locked in the maximum fuel position. A suitable pulley must be fitted to the drive shaft to enable the correct speed cycle to be achieved with the drive system employed.
If the fuel pump has been dismantled, it must be hydraulically tested and adjusted to meet the manufacturers' specifications for all parameters.

06.3 Injector nozzles

Prior to the start of each test, the injectors must be checked for opening pressures, spray pattern and leakage. The opening pressures must be adjusted to 130 bar ±5 bar. Calibration fluid, as detailed in the reference fluids section, must be used for all injector checks and adjustments.

After installing the injectors into the test rig, the fuel system must be cleaned and flushed as described in sections 6.4 and 6.5 below. Then the fuel system should be bled to remove all air prior to starting the test.

06.4 Fuel system cleaning prior to start of test

Care must be taken that all fuel system components such as heat exchangers are drained completely of all previous fluids.
The whole fuel system, without the injection pump being fitted, is to be flushed. The inlet and outlet pipes for the injection pump are to be connected in a short circuit to enable this.
The whole fuel system must be flushed through, without recirculation of the fuel, using approximately 40 litres of a non-additised diesel base fuel. This is to remove any additive from the metal surfaces of the system. All flushing fuel is passed directly to a 'waste' fuel container.
For the purposes of cleaning the fuel system, the fuel filter, used with the previous test fuel, may be discarded and the system cleaned without a filter in place. However, it must be ensured that the cleaning fuel is free of contamination.
At the discretion of the test engineer, the above cleaning procedure may be avoided for a particular series of tests. This is particularly appropriate if all fuels in a series are of similar compositions and are using similar base fuel stocks. In this instance the flushing procedure below is deemed to be adequate.

06.5 Flushing with test fuel

a) Install 20 litres of the test fuel.
b) Flush the system, without injection pump connected, pumping 20 litres of the new test fuel through the system to a waste fuel drum. The injection pump inlet and outlet connections need to be connected together to enable this.
c) Drain the fuel reservoir after flush run.
d) Drain the new injection pump of any remaining calibration fuel.
e) Prefill the injection pump with the test fuel.
f) Install the injection pump and connect to the fuel system.
g) Fill the fuel reservoir with 40 litres of test fuel.
h) Install a new fuel filter.

i) Disconnect the fuel return pipe at the fuel reservoir and connect it to a ‘waste’ container.
j) Operate the test rig to discharge 5 litres of test fuel into the waste container.
k) Top up the fuel reservoir to the initial amount of fuel circulating.
l) Reconnect the fuel return pipe to the fuel reservoir.

Note: Subsequent fuel changes, within a test, do not require the system to be flushed.

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CONTENTS of section 07 - Test operation

Test Cycle

The rig is operated according to the test conditions for a total period of 500 ±10 hours. Shorter or longer duration tests may be requested at the discretion of the engineer managing a specific project.

07.1 Test conditions

Test cycle				
Phase	Acc. Running Time [s]	Pump Speed [% of rated speed]	Fuel lever position [%]	Fuel Outlet Temp. [°C]
1	0	0	100	60
2	5	110%	100	60
3	7	110%	100	60
4	9	100%	100	60
5	118	100%	100	60
6	120	80%	100	60
7	170	80%	100	60
8	175	0	100	60
9	180	0	100	60
	Tolerances	+/-25 rev/min on determined speed	Fixed	±5
	Data Logging Phases	: once per hour at the end of phase 3 and 5		

07.3 Control of fuel circulation

After 2 hours running of the test, it is advisable to check the level of fuel in the storage tank.

07.4 Fuel temperature accumulation gradient

At start of test and after every 100 hours fuel exchange the fuel temperature must have reached its set value of 60 ±5 °C after a maximum of 3 hours running time.

07.5 100 hour fuel changes

Every 100 ± 10 hours of running time the whole 40 litres of test fuel in the system must be replaced with 40 litres of new fuel from the same test fuel batch.

The fuel change must include the full drainage of the fuel storage tank and the conditioning system. Reference should be made to the tolerance on the total test duration time when planning fuel changes.

07.6 Stoppages during the test

It is permissible to interrupt the test for up to 72 hours within each 100 hour test period.

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CONTENTS of section 08 - Test Evaluation

08.1 Dismantling of test pump

At the end of the test the test pump is removed and dismantled using appropriate special tools. The critical components are laid out for examination and visual rating.

08.2 Findings - surfaces, deposits, wear particles

Components without deposits

With correct test operation the interior of the pumps should be metallically bright and without deposits.

Causes for deposits:

- unstable fuel; e.g., unsuitable or insufficient antioxidant additive
- prescribed fuel changing intervals have been disregarded
- operating temperature during the test is to high
- unsuitable materials in the fuel circuit of the test bench (e.g., copper). It is recommended that the fuel tank and lines are made from high-grade steel (e.g., stainless 316) and that all copper and copper alloys are totally eliminated from the system.
- fuel contamination such as a defective fuel heat exchanger allowing water ingress.

If the pump interior contains surface deposits it is advisable that relevant components, with a large surface area of deposit, should be kept in suitable form and condition for a further analysis, i.e. do not clean or dry.

08.3 Wear measurements

As there is no geometrical pre-measurement of the pump components, wear can be determined only relatively; e.g., by measurement of the "step" between worn and unworn surfaces of a component.

There is no mandatory measurement of wear and any measurement serves only to support the visual rating or to clarify borderline pass/fail situations.

Scuffing marks are a reference that a critical wear situation (localised lack of lubrication) has occurred. These scuffing marks are more critical than the absolute amount of wear in relation to the service life of the pump. For example, a small amount of scuffing present on the roller bolts is likely to result in premature failure of the pump.

08.4 Rating

After dismantling, all pump components are examined for wear and/or damage. The components which are more susceptible to fuel related wear are rated, by a trained and competent rater, according to the rating scale below.

Each rated component is examined and the rating based on any seizure marks, score marks or other wear related damage.

Scuffing and fretting marks are early indications of failure as are signs of fatigue of hardened surfaces.

08.5 Rating table

Pump part	Normal wear Rating 1-3		Reduced life Rating 4-6		Premature failure Rating 7-10	
Camplate						
Cam path	polishing wear	<1µm	scuffing and pitting	>1-100µm	fatigue	
Camplate centre	fretting	1µm	fretting	>3-10µm	fretting	>10µm
Camplate Claws	polishing wear	<10µm	polishing wear and scuffing	10-20µm	scuffing	
Plunger washer	fretting	1µm	fretting	>3-10µm	fretting	>10µm
Rollers	polishing wear		scuffing and pitting	>1-5µm	scuffing and fatigue	
Roller bolts						
Point of contact with roller	polishing wear	<1µm	polishing wear and scuffing	>1-10µm	scuffing	
Point of contact with roller ring	fretting	<10µm	fretting	10-15µm	scuffing	
Governor						
Flyweights	polishing wear	<10µm	polishing wear	10-50µm	polishing wear	>50µm
Butting ring	polishing wear	<10µm	polishing wear	10-50µm	polishing wear	>50µm
Supply pump						
Blades	polishing wear	<10µm	polishing wear	10-200µm	polishing wear and scuffing	
Faceway	polishing wear	1-2µm	polishing wear	>2-100µm	polishing wear and scuffing	

08.6 Determination of overall wear

The overall rating has to take into account the likely affect of the ratings for the individual components. If a single roller bolt has a rating of say 5 then this cannot be negated by several components having ratings of 2. It is important that if the pump is likely to suffer reduced life, as a result of a single component, this should be reflected in the overall rating.

Judgement must be exercised regarding ratings which are on the borderlines between the different categories. The most important is the borderline between 'normal wear' and 'reduced life' as this is effectively the pass / fail point for the test. A rating of 3.5 is the mid point between 'normal wear' and 'reduced life'. It is generally accepted that a rating of 3.5 is still considered 'normal wear' and so a 'pass result'.

If only a single component is rated marginally within the 'reduced life' portion of the rating table, i.e. up to a rating of 4, with all other components rating within the 'normal wear' values then it the rater may determine the overall wear to be 3.5.

If a single component is rated above 4 then the overall rating must, at best, be one of 'reduced life'.

Similarly, if more than one component is rated with 'reduced life' then the overall rating must, at best, be one of 'reduced life'.

Subject	Test Report and Validation
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Record of Changes and Amendments

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CONTENTS of section **09 - Test Report and Validation**

09.1 Contents of the test report

A complete test report should be produced for each test, however, dependant on customer requirements, a shortened version may be sufficient.

The test report is made up of three sections as follows:

09.2 General information

- Name and address of testing laboratory
- Name and address of client
- Unique identification of report
- Identification of each page and total number of pages of the report
- Date of receipt if the test item, i.e. test fuel / additive
- Date of issue of the test report
- Signature and legible name of the approved signatory
- Test fuel code
- Test additive code (where applicable)
- Description of test procedure
- Details of component preparation
- Test results
- Any other information relevant to the validity of the test as requested by the client, e.g. reference data.

09.3 Operational data

- Pre-test measurement and set up data
- Operational data obtained throughout the test
- End of test measurement and test data
- Report of all instances of operations outside specified limits
- Record of all unusual occurrences

09.4 Test evaluation

- Individual component ratings as well as overall rating.
- Calculated differences between pre-test measurements and post-test measurements, where applicable.
- Comparison with reference tests, where applicable.
- Details of pass / fail limits, i.e. reference to ‘normal wear’ and ‘reduced life’.

Subject	Reference Fluids
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CONTENTS of section 10 - Reference Fluids

10.1 Fuel

It is difficult to specify lubricity reference fuels as small changes in fuel composition can lead to large differences in their lubricity performance. The EN 590 lubricity measurement using the HFRR test ISO 12156-1 specifies a maximum wear scar of 460µm. The inherent safety factor in the EN 590 standard means that a true borderline fuel is likely to have a HFRR wear scar of approximately 500µm. This information should be used with caution (see notes in section 2.1).

10.2 Injector calibration fluid

The injectors must be tested and adjusted using calibration fluid conforming to ISO 4113.

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N°	EL-80 - 11
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CONTENTS of section 11 - Referencing

11.1 Purpose of reference tests

Reference tests on good quality and also borderline pass fuels enable the normal or acceptable levels of wear to be established. Reference tests on borderline fail fuels enable the severity of the test rig to be determined. Potential borderline reference fuels are described in section 10. However, depending on the particular work programme, it may be more appropriate to use alternative referencing products.

11.2 Frequency of reference tests

Reference tests should be included in each major test programme or after every 10 product evaluation tests where single tests are conducted. Reference tests are intended to ensure that the severity of the whole test rig is maintained.

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CONTENTS of section 12 - Documentation

12.1 Documentation of Calibration

The laboratory should prepare a calibration document that comprises all calibration data (see **Section 05**).

Subject	Accreditation
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CONTENTS of section 13 - Accreditation

13.1 Accreditation

This test has been developed, from the CEC F-32-X-99 test method for diesel pump lubricity, by the Associated Octel Company Limited and is not accredited by any external organisation.

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CONTENTS of section 14 - Reference Data Base

14.1 Results

The test results shall be collated into a database for each test as:

Ratings for individual components
Overall rating
Duration of test in hours
Details of test fuel and additive

14.2 Precision of the method

The precision of the method is based on the average measurement, for all four injector needles, of the mean change in cross sectional area. Precision data are normally generated using reference fuels.

Currently no data are available regarding the precision of the method.

14.3 Table of precision data

Date	Base Fuel	Additive	Overall Rating