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Possible Mechanism for Poor Diesel Fuel Lubricity in the Field

Stephen Cook, Jim Barker, Jacqueline Reid and Paul Richards
Innospec

ABSTRACT

Traditionally, diesel fuel injection equipment (FIE) has frequently relied on the diesel fuel to lubricate the moving parts. When ultra low sulphur diesel fuel was first introduced into some European markets in the early 1980's it rapidly became apparent that the process of removing the sulphur also removed other components that had bestowed the lubricating properties of the diesel fuel. Diesel fuel pump failures became prevalent. The fuel additive industry responded quickly and diesel fuel lubricity additives were introduced to the market. The fuel, additive and FIE industries expended much time and effort to develop test methods and standards to try and ensure this problem was not repeated. Despite this, there have recently been reports of fuel reaching the end user with lubricating performance below the accepted standards. Recent publications have also suggested that it is not uncommon for sodium hydroxide used in the fuel refining industry to be present in fuel entering the supply chain downstream of the refinery. Due to the chemical nature of some lubricity additives there is clearly the possibility of interaction.

This paper briefly reviews the need for diesel fuel lubricity improver additives, previous work on such additives and possible interactions. It then goes on to present new work performed to investigate how the presence of sodium compounds in the fuel may affect the performance of a range of lubricity additives of different chemistries. It shows that the presence of the sodium hydroxide can lead to reactions with and hence the depletion of certain types of lubricity additive. This could inevitably lead to reduced lubricity performance and fuels reaching the customer that do not meet specification.

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INTRODUCTION

The issue of diesel fuel lubricity, or the lack thereof, was highlighted [1] in the early 1990s when Sweden enacted legislation mandating the reduction of fuel sulphur and aromatic content to a maximum of 50 mg/kg and 20 % respectively for Class 2 diesel and to a maximum of 10 mg/kg and 5 % respectively for Class 1 diesel. Earlier work investigating the effect of fuel sulphur levels on engine deposits and wear [2, 3, 4, 5] had suggested that high sulphur fuels resulted in increased wear. It may therefore initially be supposed that removing sulphur from the fuel might reduce wear. However, the way in which fuel sulphur content was influencing wear rates was due to the presence of sulphur trioxide [3, 4] as a result of combustion rather than the presence of sulphur compounds in the fuel affecting the lubricating properties of the fuel. Poor housekeeping resulting in "stagnant water bottoms in fuel storage tanks which contained rust, sea water, and sulphate reducing bacteria" was also found to cause corrosion in aircraft fuel systems [6]. Probably due to the fact that diesel fuel in the 1940s and 50s

was predominantly straight run distillate; the intrinsic lubricating ability of diesel fuel was never questioned.

However, history had issued a warning. Awareness of the importance of fuel lubricity had been raised in the 1950s within the aircraft engine community. The high temperatures that prevailed in certain aircraft applications [7, 8] had driven the requirement for increased fuel stability and in many cases this was brought about by increased refining of the fuel. This increased refining commonly utilised hydrotreating [9, 10] which had been shown to be beneficial [8]. Hydrodesulphurisation had been introduced to "sweeten" the increasingly common high sulphur fuels that were being produced. It is commonly thought that removing the sulphur compounds reduces the fuels lubricity, however, the hydrotreatment not only removed sulphur compounds but it also removed other polar compounds containing nitrogen and oxygen plus heavier aromatic compounds. Work by Appeldoorn and Tao [11] concluded that heavy aromatic compounds, rather than sulphur compounds per se, were responsible for imparting lubricity to the fuel. Work by Nikanjam [12] had actually shown that adding sulphur compounds back into low sulphur fuel could have a negative

effect on fuel lubricity. Earlier work by Appeldoorn and Dukek [13] had concluded that "Certain additives such as corrosion inhibitors have a marked effect on lubricity. At very low concentrations these additives reduce friction and wear, while at high concentrations they reduce gear scuffing; that is, they improve load carrying capacity." These corrosion inhibitors were often organic acids [14, 15]. As regards diesel fuel, work by Wei and Spikes found that polyaromatics, particularly oxygen-containing components reduced wear [16]. Work by Vere suggested that it was the polar compounds that were important contributors to a fuels lubricity [17].

There was inevitably many studies performed around this time to determine the most appropriate method for assessing the lubricity of the fuel. Whilst field trails and fleet tests are the ultimate arbiter of performance these are obviously costly both in terms of finance and time. Rig tests utilising susceptible fuel pumps were also developed [18, 19]. Bench top test that simulated the wear mechanisms without having to utilise the actual fuel pump and thus requiring significantly lower quantities of fuel were clearly more desirable and many alternatives were developed. These have included the Ball On Cylinder Lubricity Evaluator BOCLE [20] and variations of this method such as [21] and the Scuffing Load Ball-On-Cylinder Lubricity Evaluator (SLBOCLE) [22]. Other test methods have included using the Cameron Plint TE77 [23], the Thornton Aviation Fuel Lubricity Evaluator (TAFLE) [24, 25] and the Ball on Three Disks (BOTD) [26]. The High Frequency Reciprocating Rig (HFRR) which had been developed for other engine tribological investigations was also investigated for assessing the lubricity of Diesel fuels [27]. After a significant programme of work [28] the HFRR is now widely accepted as a representative measure of diesel fuel lubricity and has become a standard test method [29]. Alternatives such as the roller on cylinder lubricity evaluator (ROCLE) have since been proposed [30].

It must be remembered that the whole issue of diesel fuel lubricity has come about through increased refining of the crude oil to produce the high quality diesel fuel that the modern customer and legislation demand. Particularly in the US part of this refining process is the use of caustic washing to remove the acidic compounds generated as part of the desulphurisation process. The caustic used in this process should obviously be removed before the finished fuel is released from the refinery. With the good housekeeping we expect from refineries in the US this is evidently so. This provides the possibility of interaction between the caustic and fuel additives resulting in fuel injector deposits. A fuller discussion of this phenomenon is outside the scope of this paper but is discussed more fully in [31].

If there is a possibility of sodium hydroxide entering the fuel supply chain, and inevitably documentary evidence of this is hard to come by, then the obvious question is how this caustic material will react with carboxylic acid materials that may be used to meet the lubricity specification required by the advanced fuel injection equipment employed on modern

engines. The work presented here is from a programme designed to investigate how the presence of different sodium compounds which could enter the fuel system, could interact with different lubricity improver additive chemistries. The potentially disastrous consequence of allowing sodium hydroxide to enter the fuel system when only carboxylic acid lubricity improvers are used is clearly demonstrated.

EXPERIMENTAL PROCEDURES

To test the hypothesis that the effectiveness of the lubricity improver additive could be compromised by the presence of sodium compounds within the fuel, a series of lubricity performance tests were conducted before and after the potential interaction with the sodium compound. Different lubricity improver samples and chemistries were tested and the sodium ions were added as either sodium hydroxide or sodium chloride and in both cases as an aqueous solution. As aqueous solutions are immiscible with diesel fuel a high shear blender was used to ensure good contact between the phases and some additional tests were performed after the samples had been allowed to stand for a short time. The various elements of the procedure are described in more detail in the following subsections.

TEST FUELS

Base Fuel

All the test work reported here was carried out using a European reference fuel; RF06. As this reference fuel must meet the European standard EN590, the fuel would normally contain a lubricity improver additive. The fuel must therefore be purchased from the producer before the lubricity improver additive is added or the fuel sample must be processed to remove the additive. For this work the base fuel was clay filtered to ensure that no lubricity improver additive was present. This approach also ensured that any other polar compounds that might confuse the results were removed. A copy of the certificate of analysis of the base fuel before clay filtering is included in [Appendix A](#). A copy of the procedure for clay filtering the fuel is included in [Appendix B](#).

Lubricity Improver Treat Rate

Two commercial carboxylic acid lubricity improver additives from different suppliers were used; designated acid LI (A) and acid LI (B). Both of these additives are tall oil fatty acid (TOFA) based products. An ester based additive was also tested to assess the effect of a different chemistry. The lubricity performance of diesel fuel is specified as a maximum wear scar diameter (WSD) of 520 μm in D975 and 460 μm in EN590; the repeatability of the HFRR method is given as 63 μm in ISO 12156-1:2000. The treat rate of the lubricity improver additives was thus chosen to give a target WSD of 397 μm to comply with both the D975 and EN590 limits even when allowing for the results being at the favourable limit of the repeatability range. A treat rate of 100

mg/L was thus chosen for the carboxylic acid lubricity improver additives and 300 mg/L for the ester based additive.

Sodium Hydroxide Addition

An aqueous solution of sodium hydroxide, as a 17.3 wt% solution, was prepared by accurately weighing out an excess of solid sodium hydroxide into a suitable, tared vessel and then adding to it the appropriate mass of de-ionised water. Such a solution contains 9.95 wt% sodium ions. This aqueous solution was then used at treat rates of either 3mg of sodium per litre of fuel or at 1mol equivalent per acid group on the lubricity improver. For the 17.3 wt% solution 3mg of Na gives 25mg of water. For non-acid lubricity improvers, such as esters or amides, the 1 mol equivalency has been determined as one equivalent per acid group of the parent carboxylic acid from which they may be regarded as being derived. For the 1mol equivalent tests the mass of sodium hydroxide solution required is calculated from the following formula:

$$M_{OH} = 4000 * N_{acid} / C_{OH} \quad (1)$$

Where M_{OH} is the mass of NaOH solution (mg), N_{acid} is the number of acid groups present in mmol and C_{OH} is the concentration of the NaOH solution (%). The number of mols of acid present in the fuel is determined from the following formula:

$$N_{acid} = (F_{vol} * LI_v * C_{LI}) / 1000 * LI_{fwt} \quad (2)$$

Where F_{vol} is the volume of fuel in the sample (cm^3), LI_v is the valency of the lubricity improver (e.g. 1 for a monoacid and 2 for a diacid), C_{LI} is the concentration of the lubricity improver (mg/L) and LI_{fwt} the formula weight of the lubricity improver (Da).

In cases where LI_{fwt} is not known, such as for a complex mixture, e.g. a TOFA, an effective formula weight per acid group (Da_{eff}) can be obtained from the acid value (V_{acid}) as expressed in mgKOH/g using the formula:

$$Da_{eff} = 56,000 / V_{acid} \quad (3)$$

When using this formula it must be remembered that LI_v in equation (2) above becomes unity.

In situations where lubricity improver additive is supplied as a concentrate in inert solvent then it must be recalled that:

$$C_{LI} = C_{add} * C_a / 100 \quad (4)$$

Where C_{add} is the concentration of additive solution in the fuel (mg/L) and C_a is the weight per cent concentration of lubricity improver in the concentrate.

The aqueous solution of sodium hydroxide was added to the diesel fuel and mixed using a Waring Blender Model

BHL120 with a two minute agitation time at a speed setting of "1" on the Waring Blender.

Sodium Chloride Addition

For sodium chloride additions a 23.4 wt% aqueous solution was prepared. This ensured an equal water addition for an equal sodium addition in comparison to the NaOH blends. For mol equivalency the NaCl solution was added to give the same mass of sodium as for the NaOH addition.

HFRR TESTING

After blending a sample of the blend was taken and subject to HFRR testing. The blend was then filtered using a Whatman® Glass Microfibre Filter GF/A, 47 mm Ø and another sample taken for HFRR testing. The remainder of the blend was allowed to stand for an hour before being re-stirred and another sample taken for HFRR testing. The stored blend was again filtered and a final sample taken for HFRR testing. Clean glassware was used throughout. A sample of the filtered fuel was then taken for further HFRR testing as required. All of the fuels were tested according to the standard procedure ASTM D6079 [29] with a fuel temperature of 60°C.

RESULTS

The results of adding 3 mg of sodium per litre of fuel of the 17.3% sodium hydroxide solution are shown graphically in Figure 1. The clay filtered RF06 fuel produced a WSD of 603 µm which is clearly outside the limit for both D975 and EN590. When treated with 100 mg/L of acid LI (A) the resulting WSD was 394 µm which is just below our target of 397 µm; the EN590 limit (460 µm) minus the repeatability of the method (63 µm). After the blend was filtered the WSD was reduced even further; to 255 µm. At present there is no explanation for this. When the sodium hydroxide was added at 3 mg of sodium per litre of fuel and the sample was tested after agitation the WSD had increased to 443 µm. This was still within the EN590 limit and within the repeatability of the method when compared to the result prior to the addition of the sodium hydroxide. After being filtered the result was 491 µm which, although within the D975 limit, was greater than the limit set in EN590 and, even allowing for the repeatability of the method, showed a deterioration on the performance of the fuel prior to the addition of the sodium hydroxide.

After being allowed to stand for an hour, to allow for any further reaction to take place, the fuel was again stirred in the high shear blender and retested after stirring. In this case the performance was 501 µm WSD with an insignificant further deterioration to 508 µm after filtration. These results suggest that there is a reaction between the acid lubricity improver additive and the basic sodium hydroxide solution resulting in depletion of the acid content of the fuel. The non-significant increases in WSD post filtration after either NaOH treatment could be due to experimental variability. Alternatively, it could be that some of the products of reaction are insoluble but may provide some lubricity benefit to offset the effect of

losing some of the acid lubricity improver. After filtration any such benefit could be lost. There is also the possibility that any insoluble products could promote filter plugging. The increases in WSD measurements after one hour's standing could be due to variability of to further reaction. However, it should be noted that all of these results are still below, i.e. better than that of the base fuel even when allowing for the repeatability of the method.

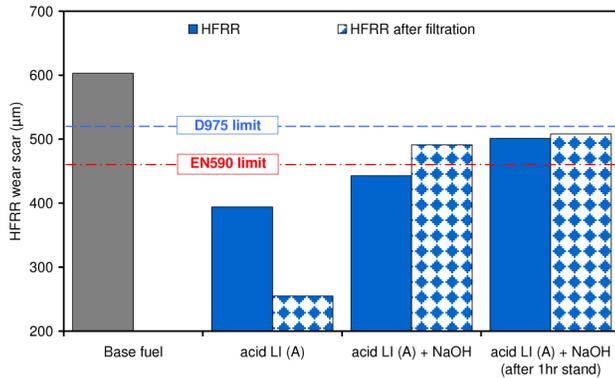


Figure 1. Acid LI (A) with 3mg of NaOH

The results from a repeat running of this series of tests are shown in [Figure 2](#). Although the absolute values of WSD are slightly greater the same general trend is observed. However, in this case the results after the addition of the sodium hydroxide are within the repeatability of the method when compared to the base fuel.

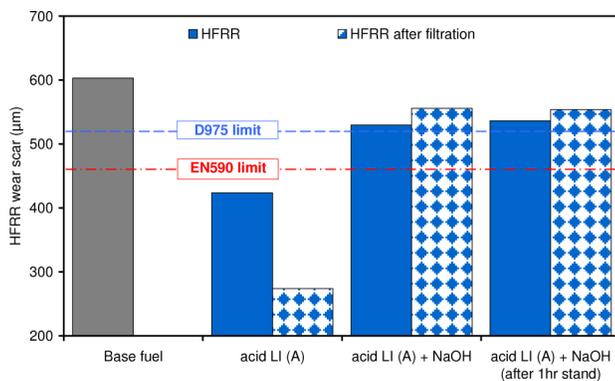


Figure 2. Acid LI (A) with 3mg of NaOH (repeat)

The results of the experiments when the level of sodium hydroxide was increased to the 1 mol equivalent are shown in [Figure 3](#). This is equal to ~ 8 mg(Na)/L and ~ 68 mg(water)/L in the fuel. In this case the addition of the lubricity improver additive reduced the WSD to 260 μm , which was lower than expected. However, after filtration the WSD was 413 μm which was higher than expected and also showed the opposite trend to the two test series shown above where the effect of filtration was to reduce the WSD. The addition of 1mol of sodium hydroxide resulted in a WSD of 610 μm , i.e. the result is effectively the same as the base fuel. This suggests

that all of the acid has in fact reacted with all of the basic sodium hydroxide and that this has essentially eliminated the effect of the lubricity improver additive. Filtering the fuel and leaving it to stand made no significant difference to its lubricity characteristics.

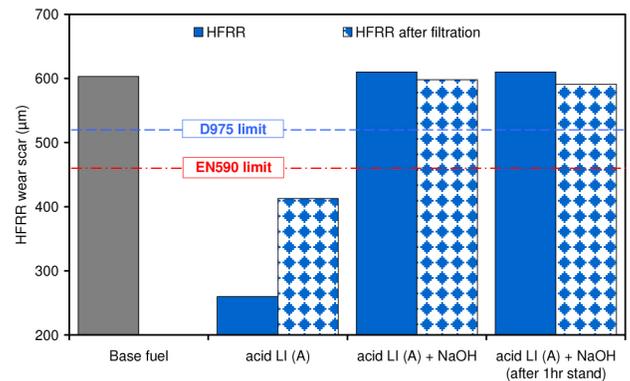


Figure 3. Acid LI (A) with 1 mol of NaOH

To confirm the suggestion that the acid was reacting with the base to remove the acid lubricity improver effect, tests were conducted with lubricity improver additive acid LI (B) and a stoichiometric addition of sodium hydroxide. The results are presented in [Figure 4](#). The addition of 100 mg/L of acid LI (B) reduced the WSD to 371 μm . The addition of the sodium hydroxide at 1 mol equivalent resulted in a WSD of 593 μm , i.e. the result was again equivalent to that of the untreated fuel. The testing after the fuel had been standing for an hour was not performed on this occasion.

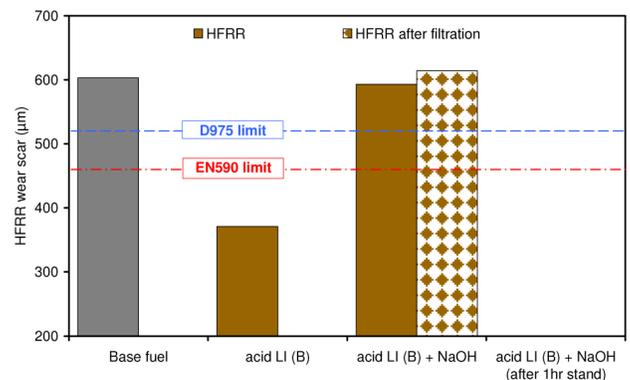


Figure 4. Acid LI (B) with 1 mol of NaOH

A corresponding series of tests were run with the ester based lubricity improver additive. The first set of tests was run with the addition of NaOH at a level of 3mg of Na per litre of fuel. The results are shown in [Figure 5](#). The addition of the ester based lubricity improver additive reduced the WSD value to 381 μm before filtration and 372 μm after filtration. After the addition of the NaOH the value of WSD was slightly reduced, but within the repeatability of the method, at 358 μm before filtration. After filtration this had increased to 402 μm . The fuel was left to stand for an hour

and then tested again. The value of WSD was again slightly lower but within the repeatability of the test at 338 μm . After filtration the value was again slightly higher than before filtration at 370 μm . All of the results with the ester lubricity improver additive were within the repeatability of the method and there was thus no indication that this level of NaOH was having a deleterious effect on the benefits bestowed by the ester lubricity improver additive.

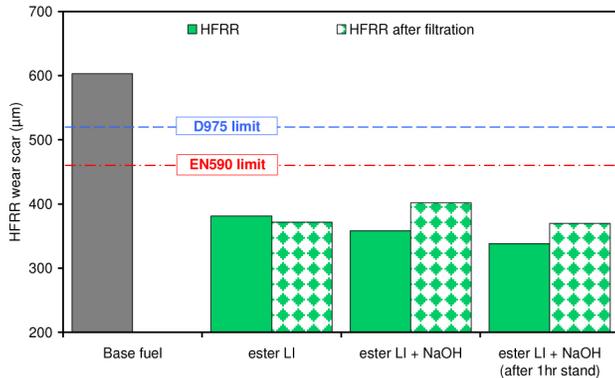


Figure 5. Ester LI with 3mg of NaOH

To substantiate this, a further set of tests was performed using the higher level of NaOH. The ester based additive was again added at 300 mg/L which resulted in a WSD value of 338 μm before filtration and 372 μm after filtration. The results are shown graphically in Figure 6. The sodium hydroxide was then added at the stoichiometric level of 1mol equivalent and mixed using the high shear blender. The WSD value for the resultant mixture was 351 μm before filtration and 264 μm after filtration. After the fuel had been allowed to stand for one hour the HFRR test was again performed before and after filtration. The resultant WSD values were 389 μm and 364 μm respectively. All of the values for the fuel containing the ester based lubricity improver additive are within the repeatability of the method; there is thus no indication of depletion of the additives benefits.

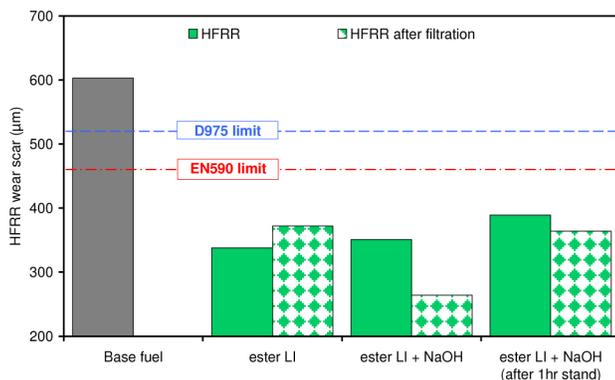


Figure 6. Ester LI with 1 mol of NaOH

Tests were also conducted using sodium chloride as an alternative source of sodium ions to the use of sodium hydroxide. The NaCl was made up as a 23.4 wt% aqueous solution and treated on the same sodium mass basis. The use of a 23.4 wt% NaCl solution gave the same mass of water addition as the 17.3 wt% solution of NaOH. Tests were performed with the mono-acid lubricity improver acid LI (A) and the ester based improver with the sodium compounds (NaCl and NaOH) added at the same sodium addition rates as the 1 mol equivalent of NaOH, i.e. $\sim 8 \text{ mg}(\text{Na})/\text{L}$ and $\sim 68 \text{ mg}(\text{water})/\text{L}$ in the fuel for both the NaCl and the NaOH for the acid LI and $\sim 23 \text{ mg}(\text{Na})/\text{L}$ and $\sim 188 \text{ mg}(\text{water})/\text{L}$ for the ester LI. The results are presented in Figure 7 in comparison to the results from the test with the NaOH addition. As expected the addition of the acid LI (A) and the ester LI reduced the WSD; to 359 μm (this is the average of the data presented in Figures 1, 2 and 3) and 360 μm (this is the average of the data presented in Figures 5 and 6) respectively. The addition of 1 mol equivalent of NaOH removed the effect of acid LI (A) but the benefit of the ester LI remained. With the addition of an equal amount of Na as NaCl the effect of acid LI (A) appeared to be slightly reduced giving a result of 412 μm but this is within the repeatability of the method and still below the EN590 and D975 limits. The result for the ester LI again remained the same within the repeatability of the method.

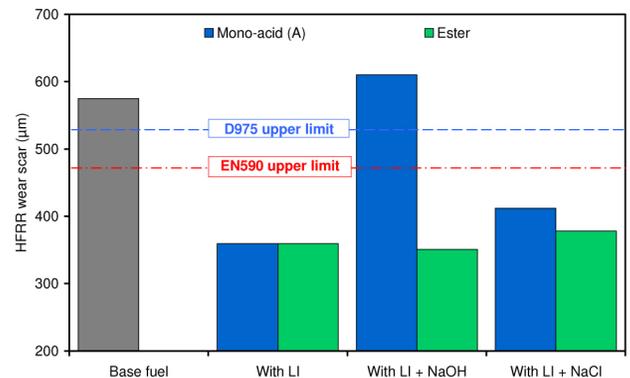


Figure 7. Comparison of the effect of NaOH and NaCl

To determine whether the water content of the blends had any influence on the effects a series of test were performed using 5 wt% aqueous solutions of NaOH and NaCl. These solutions were added to the fuel at the same Na concentrations equal to the 1 mol equivalent level for use with the mono-acid lubricity improver, i.e. $\sim 8 \text{ mg}(\text{Na})/\text{L}$ and $\sim 269 \text{ mg}(\text{water})/\text{L}$ for the acid LI/NaOH blends and $\sim 8 \text{ mg}(\text{Na})/\text{L}$ and $\sim 394 \text{ mg}(\text{water})/\text{L}$ for the acid LI/NaCl blends and $\sim 23 \text{ mg}(\text{Na})/\text{L}$ and $\sim 744 \text{ mg}(\text{water})/\text{L}$ for the acid LI/NaOH blends and $\sim 23 \text{ mg}(\text{Na})/\text{L}$ and $\sim 1087 \text{ mg}(\text{water})/\text{L}$ for the acid LI/NaCl blends. The results are shown in Figure 8.

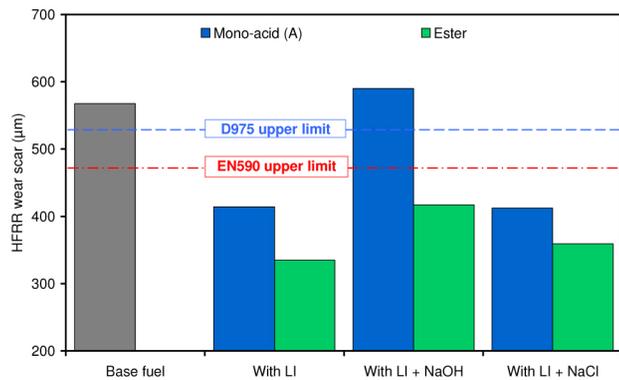


Figure 8. Comparison of the effect of 5 wt% solutions of NaOH and NaCl

It is clear from comparing Figure 7 and Figure 8 that, at least within the range explored here, the amount of water does not make any significant difference to the results. The addition of the 1 mol equivalent of NaOH appears to completely negate any benefit bestowed by the acid lubricity improver. The ester based lubricity improver performance is not significantly affected and the NaCl does not significantly affect the performance of either additive.

CONCLUSIONS

Due to environmental pressure to reduce the emissions from diesel vehicles, the permitted level of fuel sulphur has been incrementally reduced. The deep hydrogenation that is commonly used to produce petroleum diesel meeting the current ultra-low sulphur fuel specifications has been shown to remove trace components that bestowed inherent lubricity characteristics to the older high sulphur fuels. For commercial diesel fuels, the required lubricity performance has been achieved by the inclusion of fuel additives. There are many commercial fuel additives that will provide the necessary benefits with a corresponding number of different chemistries. However, most of these different chemistries can be classified as either acid or ester based.

Good refinery housekeeping should ensure that the fuel stream leaving the refinery is a hydrocarbon product with only trace amounts of other elements. However, contaminants such as NaCl may enter the fuel system between the refinery and the customers' engine. Contaminants can also be present in the fuel if there are lapses in the refinery practices, examples may be NaOH from caustic washing or NaCl from salt driers.

The work presented here has shown that if NaOH is allowed to enter the fuel chain then it can react with carboxylic acid lubricity improvers. This effectively depletes the lubricity enhancement that the acidic additives were intended to impart. This results in fuels reaching the engine with unacceptable lubricity performance which ultimately could result in engine failure. Such reactions do not appear to take place if ester based lubricity improvers are used and good fuel lubricity is retained. Contamination with NaCl

alone does not appear to result in negative effects on the performance of either acid or ester based lubricity improver additives.

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CONTACT INFORMATION

Steve Cook

Tel: +44 151 348 5835

steve.cook@Innospecinc.com

Innospec Limited

Innospec Manufacturing Park

Oil Sites Road, Ellesmere Port

Cheshire

CH65 4EY

England

<http://www.innospecinc.com>

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APPENDIX

APPENDIX A



**PETROCHEM
CARLESS**

CERTIFICATE OF ANALYSIS

PROPERTY (UNITS)		SPECIFICATION		RESULT	METHOD
		Min	Max		
MATERIAL: Carcal RF-06-03				BATCH NUMBER: 08/047	
PRODUCT CODE: 47094				CERTIFICATION DATE: 26/11/08	
				CERTIFICATE NUMBER: 2575	
Cetane Number *		52	54	53.2	D613
Density @ 15°C, g/ml		0.833	0.837	0.8339	D4052
Distillation, °C				D86	
IBP		TBR		192.5	
10% Vol Point		TBR		221.0	
50% Vol Point		245		272.5	
90% Vol Point		TBR		327.0	
95% Vol Point		345	350	348.5	
FBP			370	357.5	
Pensky Closed Flash, °C		55		83	D93
Cold Filter Plugging Point, °C			-5	-23	EN116
Viscosity @ 40 °C, mm ² /sec		2.5	3.3	2.77	D445
Polycyclic Aromatic, %mass *		3	6	4.8	IP391
Total Aromatics, %mass *		TBR		28.7	IP391
Sulphur Content, mg/Kg *			10	<1	IP490
Copper Corrosion, 3hr @ 100°C			1	1b	D130
Conradson Carbon Residue on					
10% Dist. Residue, % mass *			0.2	<0.01	D189
Ash Content, % mass *			0.01	<0.001	D482
Water Content, mg/Kg *			200	70	IP438
Neutralization (Strong Acid) Number (mg KOH/g) *			0.02	0.01	D974
Fatty Acid Methyl Ester			Prohibited	NIL	EN14078
Lubricity, µm *			400	316	ISO12156-1
Oxidation Stability, mg/100ml *			2.5	0.1	D2274
Cloud Point, °C *		TBR		-20	D2500
Water & Sediment, %v/v *		TBR		NIL	D2709
Fuel H/C Atomic Ratio		TBR		1.84	Calc.
Fuel O/C Atomic Ratio		TBR		<0.0003	Calc.
C/H Mass Ratio		TBR		6.47	Calc.
Carbon Content, %m/m *		TBR		86.62	D5291
Hydrogen Content, %m/m *		TBR		13.38	D5291
Oxygen Content, %m/m *		TBR		<0.04	Elem Anal
Carbon Weight Fraction		TBR		0.8662	Calc.
Net Calorific Value, BTU		TBR		18485	Calc.
Net Calorific Value, MJ/Kg		TBR		43.00	IP12
Gross Calorific Value, MJ/Kg *		TBR		45.84	IP12

The results marked with an asterisk were obtained from ITS UK Ltd

All other tests were carried out by Petrochem Carless.

HARWICH REFINERY
REFINERY ROAD
PARKESTON
HARWICH
ESSEX
CO12 4SS

ISSUED BY S. Calver
POSITION RCR Chemist
CHECKED BY S. Barrett
ISSUE DATE 15/12/08
ISSUING SIGNATURE

Petrochem Carless Limited Refinery Road, Parkeston, Harwich, Essex CO12 4SS, UK
Tel +44 (0)1255 502372 Fax +44 (0)1255 552181 www.petrochemcarless.com



Registered office: Cedar Court, Guildford Road, Leatherhead, Surrey KT22 9RX. Registered in England No. 429315

APPENDIX B

CLAY FILTERING OF FUELS

1. Scope

1.1. This method is used to prepare reference fluid base that gives 100 MSEP Rating

2. References

2.1. ASTM D3948 Appendix X1. - Preparation of reference fluid base

2.2. ASTM D4306 Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination

3. Summary of Method

3.1. A 20L sample of fuel is flowed at constant rate through a fresh column of granular clay and collected in a clean storage receiver.

4. Apparatus

4.1. Glass Column with sealed coarse fritted glass disk at bottom and with a 4mm metering type PTFE-fluorocarbon stopcock outlet at the bottom. Inside diameter is 55-65mm and the length above the disk is at least 1metre.

4.2. Separating funnel with lid - 2L

4.3. Receiver container - Borosilicate glass bottle as per ASTM D4306

5. Materials

5.1. Attapulugus clay - 30/60 mesh, LVM (calcined) grade or equal. Store the clay protected from atmospheric moisture and avoid handling that will cause particle size segregation.

5.2. Glass Wool

5.3. Toluene

5.4. Silicone Grease

6. Procedure

6.1. Mount the column vertically

6.2. If using large column place fist size amount of glass wool in bottom

6.3. Fill the column approximately 2/3 full of clay, tapping sides to settle.

6.4. Place a fist size amount of glass wool at the top of the column.

6.5. Place waste receiver container under column

6.6. Mount the separating funnel above the column so that the drain spout is sitting in the top of the filter column NOTE: Separating funnel only required if using small clay filter column - larger column will hold 2L without the need for using a displacement technique

6.7. Ensuring the stopcock is shut on the column pour enough fuel on top of the clay to cover the spout on the separating funnel

6.8. Ensuring the stopcock on the separating funnel is closed pour 1-1.5L of fuel and put lid in place (a small amount of silicone grease may be used to allow easy removal of lid)

6.9. Open the stopcock on the column then open the stopcock on the funnel the fuel will filter via displacement

6.10. Allow the fuel to filter at approx. 50-60mL/min, collect a few 100mls and pour filtrate back into filter column or discard

6.11. Place sample receiver container under column and begin collecting clay filtered fuel

7. Changing Clay Column The column must be changed regularly key signs the column needs changing are listed below

- 7.1. If 20L has been passed through the column
- 7.2. Column has been left to run dry
- 7.3. Signs of cracking in the clay
8. Cleaning the column
 - 8.1. Drain the column and separating funnel
 - 8.2. Dismount the column and holding upside down and with the stopcock open blow out the clay into a suitable waste container
 - 8.3. While column is still inverted rinse thoroughly with toluene then with acetone. The separating funnel can be cleaned in the same manner.
 - 8.4. Disassemble the stopcock and rinse again with toluene followed by acetone, air dry and reassemble
 - 8.5. Occasionally the column may need washing in hot water if column is not clean after the above washing cycle. If necessary rinse with acetone and air dry.