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### **STORAGE, HANDLING AND COMBUSTION OF A NOVEL HEATING FUEL: EL**

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### **ABSTRACT**

Ethyl Levulinate (EL) is being developed as a cellulosic, renewable fuel for use in boilers and furnaces. In this paper, the technical aspects of using this fuel in this market are discussed. Positive attributes of EL include very low net greenhouse gas emissions, excellent cold flow properties, acceptable flash point, and low viscosity. This candidate fuel is not generally miscible with petroleum fuels. It is miscible with biodiesel but in such blends the cold flow advantages are reduced. Use of EL alone is seen as a practical deployment pathway. EL has not been found to be compatible with elastomers commonly used in petroleum designed equipment and alternative seal materials have been identified and integrated into burner systems for testing. With an oxygen content of 33% the heating value of EL, on a volumetric basis, is 30% lower than for petroleum heating fuel. At an equal energy input rate, the NOx emissions with EL have been found to be 40% lower.

#### **INTRODUCTION**

Renewable biofuels, derived from non-food and waste sources, offer the potential to achieve net reductions in greenhouse gas emissions (GHG) by displacing fossil fuel use from a range of applications. This study is focused on the use of biomass-derived ethyl levulinate (EL) as a fuel for use in stationary boiler and furnace applications. Relative to transportation applications, these have a lower global fuel consumption but are also seen as less technically challenging for biofuel conversion.

Ethyl levulinate (EL) fuel production is based here on the Biofine Process [1, 2] which starts with the mild acid hydrolysis of cellulosic feedstock. Primary products of these steps include levulinic acid (LA), formic acid (FA), and furfural. The levulinic acid is then esterified to produce ethyl levulinate. Figure 1 provides a simplified scheme representing the multi-stage chemical cascade occurring in a typical dilute acid hydrolysis reaction resulting in LA. Under typical conditions of aqueous dilute mineral acid (3%) and elevated temperature (200 °C.) cellulose is broken down by hydrolysis to form monomeric glucose. This isomerizes to fructose followed by dehydration to the unstable intermediate hydroxymethyfurfural (HMF). HMF then hydrolyzes to form the stable endproducts LA and FA.



**Figure 1.** Simplified chemistry scheme of Biofine process

At each stage of the reaction, decomposition products collectively known as humins are formed via kinetically favored higher order side-reactions and represent local free-energy minima. In the production of LA formation of humins represents significant yield loss when the reaction is conducted in simple batch mode. The low yield has, in the past, rendered production of levulinic acid via this route commercially unattractive despite the abundant availability of low-cost cellulosic feedstock [3-5]

The Biofine process addresses the problem of excessive humin production by carrying out the conversion reaction in a continuous back-mixed mode. The mixing regime in the reactor causes the average concentrations of reactants to be diluted thus reducing the dominance of the higher order humin-forming reactions. This increases the forward flow of carbon resulting in increased levulinic and formic acid yields. With this reactor configuration, for a wide range of cellulosic feedstocks tested, the molar yield of levulinic acid from the cellulose fraction is consistently found to be at least 70%. The Biofine process thus allows a potentially economic route to produce low cost levulinic acid from biomass. This opens up the possibility of economic production of a range of chemicals and bio-fuels using levulinic acid as the intermediate platform feedstock [1, 2]

Given the near-term availability, at reasonable cost, of roughly 12 quads energy equivalent of biomass material**,** the Biofine process therefore has the potential to contribute significantly in combatting global carbon dioxide emissions via displacement of fossil-derived heating fuel. The Biofine process for production of biofuels and chemicals is shown schematically below:



**Figure 2.** Block diagram of the Biofine process

The Biofine conversion process is presently operated at semi-commercial scale in a pilot demonstration plant owned and operated by the University of Maine Forest Bioproducts Research Institute (UMO FBRI) in Old Town, Maine. This plant is nominally capable of processing one dry ton per day of wood or other biomass feedstock producing around one dry ton per week of levulinic acid or ethyl levulinate. The plant has been used to produce tonnage quantities of ethyl levulinate for testing purposes.

## **Important Co-products from Cellulose Conversion**

As indicated in the above diagram, LA production via the Biofine route also produces important co-products: Formic acid, produced in the main reaction, is a commodity chemical with a present global market of over 500,000 metric tons per year. It is produced commercially at present using fossil-derived fuel oil. In addition to its commodity market uses, formic acid shows promise as a convenient method of hydrogen storage as it can be easily split using metallic catalysts such as ruthenium into gaseous hydrogen and carbon dioxide.

Furfural is produced in the acid hydrolysis reaction from pentosan polymer occurring in many biomass feedstocks. Furfural is also a commodity chemical with a similar sized market to formic acid. As an alternative to commercial sale, furfural can be converted to levulinic acid via furfuryl alcohol using well-known technology. This would represent a significant additional output of levulinic acid.

The process also produces bio-char which is a combination of lignin in the feedstock and humins. This is produced as a black hydrophobic powder with an energy content of around 28 MJ per Kg (12,000 BTU per lb). Biofine's bio-char can be dewatered, pelletized and sold as boiler or stove fuel or can be burned in the process to produce electric power for internal use and for export to the grid. Ahmad et al. [6] and Badgujar et al.[7] recently published broad reviews of methods of producing EL from biomass which provides insight into other production technologies.

Table 1 provides a comparison of the basic fuel property data for No. 2 petroleum-sourced heating oil, biodiesel, and ethyl levulinate.

		No. 2 heating oil	Propane	<b>Biodiesel</b>	Ethyl Levulinate $(EL) C7H12O3$
Composition	C wt%	86	89	75.5	58.3
	$H w t\%$	14	11	12.6	8.3
	$O wt\%$	0		11.9	33.3
Density	g/cm3	.85	0.49	0.88	1.02
<b>Higher Heating</b>	MJ/kg	45.6	48.1	40.7	26.3
Value	MJ/L	38.7	25.1	36.0	26.8
<b>Flash Point</b>	$\rm ^{\circ}C$	38 min	$-155$	93 min	91
Viscosity	$mm^2/s$ @ $40^{\circ}$ C	$1.9 - 4.1$	$4.98 \times 10^{-6}$	$1.9 - 6.0$	1.5

**Table 1.** Comparison of Basic Fuel Properties

Due to its high in oxygen content, the heating value of EL is considerably lower than No. 2 heating oil and biodiesel, but roughly equivalent to that of propane. Studies published to date have focused on the use of EL as a blend stock or additive in diesel fuel, No. 2 heating oil, or blends also involving biodiesel.

Christensen et al. explored the use of EL and butyl levulinate (BL) in blends with diesel fuel in engines[8]. They report that saturated EL contains 8.5% water by weight. This group also explored separation of EL/diesel blends at reduced temperatures. They found that for a 10% blend of EL in diesel, fuel separation begins at 5°C.

Joshi et al. [9] studied the use of EL as an additive to improve the cold flow properties of biodiesel fuel. Reductions in cloud point as high as 5°C were found with 20% EL in the blend.

The use of EL as a blend component in commercial heating fuels, including petroleum No. 2 and blends of petroleum No. 2 and biodiesel, offers some logistical advantages. With blends, a heating fuel marketer with a large number of end customers could distribute just one fuel, the EL blend, to all customers, avoiding the need for segregation of their storage and delivery systems. In an early, informal field test, the Dead River Company in Maine distributed a blend of 10% EL by volume in petroleum to 11 home sites for two years. In the present study, blends of EL, at different levels, with petroleum No. 2 heating fuel and biodiesel were explored. The primary focus in this work was on simple separation over a range of temperatures. These blend tests also included an evaluation of one candidate co-solvent reported to be at least somewhat effective with these types of blends.

Based on the results of the blend testing with No. 2 heating oil as described in the following sections, a decision was made to focus on 100% EL, unblended, as the target fuel for this application. Combustion studies, also described in the following sections, were done with the unblended fuel.

The liquid fuels heating industry provides heating oil, biodiesel, and kerosene to over 5 million homes and business in the United States. Comparatively, utility gas supplies gas to 58,702,794, and electricity 48,468,860. In total, heating oil supplies approximately 5 percent of all homes in the United States [10]. However, that modest percentage changes as we focus on the populated and colder areas of the country. In New York, there are 1,393,560 oil heated housing units, or 19 percent of all units in the state [11]. In Maine, there are 344,869 housing units using heating oil, or 60 percent [12].

The northeastern states where oilheat is prominent have also adopted greenhouse gas reduction strategies. Most of them are seeking a 30-40 percent reduction in emissions by 2030 and 80 percent or higher by 2050. The thermal load's contribution to greenhouse gases is characterized as onethird by NYSERDA. Thus, reductions of the magnitude sought cannot be achieved without efforts in the thermal sector.

The heating oil industry has attempted to provide low carbon alternatives through the adoption and use of biodiesel. However, concerns with supply, operability in cold climates, and greenhouse gas scores have prompted the industry to continuously examine other biofuels. One of these is ethyl levulinate.

A key aspect of ethyl levulinate is how its use would impact emissions of greenhouse gases from liquid fuels. Earthshift Global, a provider of life cycle analysis and sustainability services, completed an analysis of the fuel and process of Biofine in early 2019. In this analysis they examined two streams for potential conversion into ethyl levulinate, forest residue and postconsumer wastepaper. They did the analysis based on the process flow prepared by Biofine, and the co-products that would be produced in the production of EL. The study concluded that as a heat source, EL could reduce emissions from ultra-low sulfur diesel (ULSD) used as heating fuel between 110 and 120 percent [13]. Thus, this fuel could be very attractive to the heating industry as it can essentially provide a zero carbon fuel for heating oil consumers.

### **EXPERIMENTAL**

#### **Blend Tests**

A total volume of 100 ml was prepared for each target blend as listed in Table 2.

Initially, blends were observed for visual separation for 24 hours at room temperature. Blends which did not separate at room temperature were moved to a glass-front, controlled temperature chamber to observe visual separation at lower temperatures. Chamber setpoint temperature was lowered in 2.8 °C (5 °F) intervals until separation was observed or a temperature of -9.4 °C (15 °F) was reached. At each temperature setpoint the sample was observed for 30 minutes.

Five of the sample blends did not show separation at -9.4  $\degree$ C (15  $\degree$ F). The Cloud Point of these samples was then measured at Iowa Central Fuel Testing Laboratory following ASTM D 2500.

	Blend Composition (volume %)			
<b>Blend Number</b>	EL	Petroleum No. 2	Biodiesel	Isoamyl
		<b>Heating Fuel</b>		Alcohol <sup>1</sup>
	10	90		
$\mathcal{D}_{\mathcal{L}}$	20	80		
	30	70		
	50	50		
			95	
6	10		90	
⇁	20		80	
8	50		50	
9	33.3	33.3	33.3	
10	45	10	45	
11	20	75		
12	30	65		

Table 2. List of Blends of EL, No. 2 oil, Biodiesel, and Isoamyl Alcohol<sup>1</sup>.

<sup>1</sup>Isoamyl Alcohol used as a candidate cosolvent

### **Elastomers and Filter Elements**

Testing was done to determine EL's compatibility with some of the elastic materials found in an oil heat system. The two materials evaluated were Nitrile and Viton. Swell testing was done following UL 157 Section 11.4.2. In this standard, an elastomer exposed to fuel must have swell within the range -1% to 25% in the fuel to pass the test. For both materials, two different sized Orings were evaluated. Each material was immersed in the desired fuel for 70 hrs. The O-rings were measured with a micrometer before and after this swell period and the change in volume was calculated. Testing was done with multiple different fuels, including No. 2 oil, biodiesel, EL, and multiple blends of the fuels include 95/5 No. 2 oil:EL, 90/10 No. 2 oil: EL, 78/22 No. 2 oil:EL, 50/50 Bio:EL, and 1/1/1 No. 2 oil:Biodiesel:EL.

After testing was done on the materials common to the oilheat industry, other potential materials were evaluated. Many different types of materials were evaluated for their compatibility with EL, but the two that will be focused on here are silicone and Teflon. In the same manner as discussed above, silicone and Teflon O-rings were evaluated following UL 157 11.4.2. These o-rings were immersed in No. 2 oil, Biodiesel and EL. After 70 hrs of immersion, the change in volume of each o-ring was again determined.

Based on reports gathered from the field, the compatibility of EL with various fuel filter materials was also evaluated. The filter types evaluated were a cellulose "microfiber" filter, a felt filter, a wool string filter, and a spin-on filter cartridge. Each of these filters was completely immersed in EL. They were then left to be observed to see if any visible changes occurred over time.

# **Combustion Testing**

Combustion testing was done in two heating boilers, one typical of those used for the production of heat and domestic hot water in single family home applications and one typical of those used for larger commercial buildings.

The residential-scale testing was done in the NORA lab in Plainview, N.Y. using a 3-section cast iron boiler with an integral tankless coil heat exchanger for domestic hot water (Model Peerless WBV-03, PBHeat LLC). Water on the heating side of the boiler was continually circulated without active heat extraction during these tests. The desired thermal load on the boiler was controlled through adjustment of cold water flow through the internal domestic hot water coil. The boiler temperature for all tests was steady and in the range of 70 to 75°C. A swirl, pressure-atomized, fixed firing rate burner was used in all tests (Model EZ-1, Carlin Combustion Technologies). The fuel pump with integral pressure regulator used was modified to have all elastomer static seals compatible with EL. This included some silicon and some Teflon seals. The pump shaft Nitrile lip-style seal was replaced with a Teflon-graphite composite seal. This pump was used with both petroleum heating oil and EL.

To directly measure fuel flow in the residential boiler tests, the fuel supply container was mounted on a scale with a resolution of 0.023 kg (0.05 pounds). Fuel mass flow measurement was integrated over a 30 minute steady period for each fuel. Flue gas oxygen was measured in all tests using a wet electrochemical cell analyzer (Model DC 710, Test Products International, Inc.). Flue gas NO<sub>x</sub> and CO concentration was measured using an NDIR analyzer with external  $NO/NO<sub>2</sub>$  converter. (Model ZRE, California Analytical Inc.). Boiler temperature, flue gas temperature, gas composition and other basic operating parameters were recorded using a LabView-based data acquisition system.

Based on the results of blend testing, described in the next section, combustion tests focused on EL not blended with petroleum or biodiesel. Unblended EL is seen as the most practical commercial fuel. Testing of the residential boiler was done using No. 2 petroleum heating oil, 100% EL, and a blend of 91.5% EL and 8.5% water by volume. The water/EL blend represents EL which has been saturated by EL and this is seen as an extreme, limit case. It is highly unlikely that this would occur in the field.

Firing No. 2 petroleum heating oil, the heat input rate, based on measured fuel mass flow was 28.7 kW. Nozzle nominal size was  $0.65$  gph<sup>1</sup> and the burner operating pressure was adjusted to 150 psi. Firing EL the fuel mass flow was raised using a larger nozzle size (1.0 gph nominal) to produce

 $1$  Burner fuel nozzles of this type are commonly rated in flow of petroleum No. 2 heating oil at 100 psi in gallonsper-hour (gph). At higher pressure, volume flow rate can be closely estimated using the square root of the pressure ratio.

the same heat input rate. This same nozzle and pressure condition was used with the water/EL blend, producing a heat input rate 8.5% lower than with EL.

The larger commercial boiler tests were done tests in the development lab of Carlin Combustion Technologies, a major burner manufacturer in North Haven, Connecticut. Tests were conducted in a Weil-McLain 688 Cast Iron Section boiler configured with direct cold water at the boiler inlet. In the field, boilers of this type operate with much higher temperatures at the boiler inlet but with large boilers of this type it is common practice to test with cold water in, simply to reduce the water flow rate required to enable the burner to run continuously without cycling. The burner installed in the boiler for this test was a Model 601 CRD manufactured by Carlin Combustion Technologies. This burner also uses a pressure-atomized arrangement. Flue gas CO, Oxygen, and NOx were measured using an analyzer with wet electrochemical cells (Ecom Instruments). As with the residential boiler testing, the pump used for these tests was converted to have elastomer seals compatible with EL. For petroleum No. 2 heating oil, an 8.0 gph nominal rate nozzle with an operating pressure of 150 psi was used. For EL testing, a nominal 9.5 gph nozzle with an operating pressure of 250 psi was used. Testing with a water/EL blend was not done with the commercial boiler. With both tested fuels, the input rate was 410 kW.

For all fuels in both the residential and commercial boiler tests smoke number was measured using the Shell Bacharach method (ASTM D2156). In all cases reported here the measured smoke number was zero.

#### **Steel Corrosion**

Questions have been raised about the affects EL or EL contaminated with water could have on steel material. This is particularly important as many of the fuel storage tanks found throughout the industry are made of steel. To test this, small 2 inch by 1 inch sized coupons of steel were immersed in EL samples with various amounts of water. The EL samples evaluated here were EL with no water, EL with 1% water, and EL with 8% water. These samples were evaluated daily to visually check if any corrosion was forming on the metal.

### **RESULTS**

### **Blend Tests**

The results of mixing the various blends and leaving them at room temperature are available in Table 3. As you can see from the table, most samples did not have any issues with separation. The only samples that had issues were blends number 3, 4, and 12, as well as blends of No. 2 oil and EL at the 70/30 and 50/50 blend level. Separation was immediately apparent after shaking of the samples. Blend 12, with the addition of isoamyl as a cosolvent, also showed separation. This testing points to a compatibility issue with blending No. 2 oil and EL. Because of the separation at room temperature, these sample were removed from further testing. No other samples showed any separation at room temperature after 24 hrs.

<b>Blend Number</b>	Separation (Y/N)
	N
$\overline{c}$	N
3	Y
4	Y
5	N
6	N
7	N
8	N
9	N
10	N
11	N
12	

**Table 3**. Blend Separation at room temperature.

Next, testing in the freezer at various temperatures was done. Observations of any separation and the temperature this occurred at are shown in Table 4. From this testing, blends 1, 2, 10 and 11 showed separation below room temperature. With the separation of blends 1 and 2, it becomes clear that blending No. 2 oil and EL on their own can have serious issues. The addition of isoamyl alcohol as a cosolvent also seemed to have little to no effect. Blends of biodiesel and EL appear to have no issues with separation, though many of the samples appeared to be clouding at the lower temperatures of the test. It should be noted, though, that Blend 9, a blend of equal parts No. 2 oil, EL and biodiesel, did not have any issues with separation in the temperatures tested. Before moving to the next round of testing, all samples that showed separation were removed.

Finally, the cloud point of the various blends that did not separate were assessed using ASTM D2500. In addition, the cloud point of the No. 2 oil and biodiesel used to make the blends was tested. The cloud points of each of the fuels are listed in Table 5. As seen in the table, the cloud point of the blend with EL improved. However, these changes are small and the measured cloud points of these blends are much greater than that of unblended EL.

	Blend Number   Separation (Y/N)	Freezer Temperature at Separation (°F)
	V	15
2	V	55
6	N	
	N	
		20

**Table 4**. Separation of blends in freezer.

<b>Blend Number</b>	Cloud Point $({}^{\circ}F)$
5	30.2
6	28.4
	28.4
8	26.6
q	23.0
Base No. 2 oil	12.2
<b>Base Biodiesel</b>	33.8

**Table 5**. Cloud point of various blends.

#### **Elastomers and Filter Elements**

The change in volume of each of the Nitrile and Viton o-rings can be found in Table 6. For both Nitrile and Viton o-rings, a large amount of swell was observed with any fuel blend containing EL. Only the Nitrile O-ring 2 immersed in 95/5 No.2 oil:EL was below the 25% swell limit. Interestingly, it appears that Viton swells more in EL than Nitrile, though both changes in volume are unacceptably high.

**Table 6**. Volume Change of Nitrile and Viton O-rings in Various Fuels After 70 Hours of Exposure



The change in volume for both the Silicone and Teflon o-rings is listed in Table 7. The Teflon oring performed very well and saw almost no change when immersed in EL. The main issue with this Teflon o-ring is stiffness. This material may not be usable in all parts of a heating oil system. The Silicone O-ring was performed well with EL and is flexible enough to be used in all parts of a fuel system. It should be noted, however, that the Silicone O-ring showed a 44.9% swell in No. 2 oil. Additional fluorocarbon elastomer types were tested and found to be compatible with EL.

Fuel	Silicone O-ring Volume Change (%)   Teflon O-ring Volume Change (%)	
No. 2 oil	44 9	09
<b>Biodiesel</b>		

Table 7. Volume Change of Nitrile and Viton O-rings in Various Fuels.

Of the various filter materials tested, only one showed a noticeable change in the time period tested. The felt filter, the wool string filter, and the spin-on, pleated paper filter cartridge all showed no obvious change after being immersed in EL for two weeks. The one filter that did show a very rapid degradation is described by the manufacturer as a microfiber filter element. Only a few minutes after being placed in the fuel, the filter began to change, almost appearing to melt. After 30 minutes, the filter had completely dissolved. Pictures of the filter before immersion and after 30 minutes of immersion can be seen in Figure 3. This common type of filter is clearly incompatible with EL. The specific element tested is a Westwood "PureOil Element" – other manufacturers offer similar products.



Figure 3. A) Microfiber filter element before being immersed B) filter after being immersed in EL for 30 minutes.

### **Combustion and Emissions**

With all fuels tested in the residential and commercial boilers, ignition was smooth, and the burners operated over a wide excess air range with a stable flame. In the case of the residential boiler, flue gas CO was measured to be less than 10 ppm under all conditions and this level is considered negligible. Figure 4 provides a comparison of the measured NO<sub>x</sub> emission factors for all fuels in the residential boiler.



**Figure 4.** Residential boiler - NOx emission factor, all fuels tested

In the commercial boiler tests, flue gas CO was found to be higher for EL and these results are presented in Figure 5. NOx emission factor results for the commercial boiler are shown in Figure 6.



**Figure 5.** Commercial boiler- flue gas CO, all fuels tested



**Figure 6.** Commercial boiler- NOx emission factor, all fuels tested

The results in Figure 5 show higher CO for EL than for petroleum No. 2 heating fuel. However, these levels of CO are considered well within acceptable levels, particularly between 30 and 50% excess air, the common operating range. The smoke number was also found to be zero across this testing. Figures 4 and 6 show that the NOx emission factor will be significantly lower with EL, falling approximately 40% lower than for petroleum No. 2 in the residential boiler case and 30% lower in the commercial boiler case. The NOx emission factor with water saturation is 24% lower than for EL alone in the residential case.

The configuration of the commercial boiler allows direct viewing of the flame from the rear surface, opposite from the burner. Figure 7 provides these images for both petroleum No. 2 and EL at the same input rate and excess air level.



**Figure 7.** Comparison of the appearance of the petroleum No. 2 flame (left) and the EL flame (right). Both at  $\sim 6.5\%$  flue gas oxygen and a nominal input rate of 410 kW.

## **Steel Corrosion**

Visual inspection of the steel coupon did yield results. The sample of EL with no water and the sample of EL with 1% water show no signs of corrosion after 2 months of observation.

The sample of EL with 8% water, however, did begin to show signs of corrosion after 13 days. This can be seen in Figure 8. Additional testing over a range of water levels and using corrosion inhibitors is planned. It should be noted that 8% is not a typical operating condition for the heating fuels.



**Figure 8**. Steel coupon immersed in EL blended with 8% water after 13 days.

# **CONCLUSIONS**

In this work, Ethyl Levulinate has been shown to hold significant promise as a renewable fuel for stationary boilers and furnaces. Life Cycle Analysis studies which have been done show that the calculated greenhouse gas emissions with this fuel can be less than zero. Because this fuel can be made from waste wood products the resource size is exceptionally large.

At low blend levels the fuel can be used in combination with petroleum No. 2 oil although with increasing blend levels and reducing temperatures separation becomes a significant concern. EL can be blended with No. 2 petroleum heating oil and biodiesel together with improved separation aspects. With such a blend, however, the cold flow properties are dominated by the biodiesel fraction and there remains a significant concern in colder climates. A particularly important advantage of EL in this application is the extremely good cold flow properties. For these reasons, this work focused on 100% EL as a candidate fuel for this application.

EL has been found to not be compatible with Nitrile and Viton elastomer seal materials used in current No. 2 petroleum-fired boiler and furnace fuel systems. These materials need to be replaced with silicon, Teflon, and Teflon/graphite composite materials have been found to be suitable.

The corrosion of steel found with EL mixed with high amounts of water is concerning. More work is needed to confirm the concentration of water where corrosion will most likely happen. Work is also planned to look at metal deactivators as a potential way to resolve the corrosion issue. It should be noted that the EL sample mixed with 1% water has shown no corrosion reaction so far.

Using fuel system components with acceptable elastomers, combustion testing has been done with EL and water-saturated EL. In both cases good combustion performance was observed in a residential and a commercial boiler application. NOx emissions with EL were found to be 40-60% of those with petroleum No. 2 fuel under the same conditions.

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