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# A 322,000 kilometer (200,000 mile) Over the Road Test with HySEE Biodiesel in a Heavy Duty Truck

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## ABSTRACT

In July 1997, the Pacific Northwest and Alaska Regional Bioenergy Program, in cooperation with several industrial and institutional partners initiated a long-haul 322,000 km (200,000 mile) operational demonstration using a biodiesel and diesel fuel blend in a 324 kW (435 HP), Caterpillar 3406E Engine, and a Kenworth Class 8 heavy duty truck. This project was designed to: develop definitive biodiesel performance information, collect emissions data for both regulated and non-regulated compounds including mutagenic activity, and collect heavy-duty operational engine performance and durability information. To assess long-term engine durability and wear; including injector, valve and port deposit formations; the engine was dismantled for inspection and evaluation at the conclusion of the demonstration. The fuel used was a 50% blend of biodiesel produced from used cooking oil (hydrogenated soy ethyl ester) and 50% 2-D petroleum diesel. The demonstration vehicle traveled 326,235 km (202,160 miles) in actual commercial operation averaging 5.27 miles/gallon.

The biodiesel feedstocks used were ethanol and used hydrogenated soy oil produced as a by-product during the manufacture of French fries by the J.R. Simplot Company. The used French fry oil produced a biodiesel referred to as HySEE. Total glycerol of the fuel averaged 0.402% and yield as a mass percent of raw oil averaged 85.4%.

At the conclusion of the operational demonstration, Caterpillar conducted an extensive engine inspection and analysis. Suffice it to say, "Nearly all aspects of this test passed evaluation with flying colors." The only exceptions were transient emissions – significant increases in PM and related reductions in $NO_x$ were observed. The emission changes were attributed to a decrease in injection pressure and a delayed or retarded fuel injection event. These changes were not attributed to the biodiesel fuel. Wear measurement testing results expressed in miles suggest that this engine could be expected to exceed a vehicle mileage of 1.6 million km (1,000,000 operational miles.)

A second identical engine was performance and emissions tested at the Caterpillar Technical Center, Peoria, Illinois. The test engine was conditioned and tested to meet the requirements of Clean Air Act Section 211(b). As part of the testing, personnel from the Department of Environmental Toxicology, University of California, Davis collected samples for chemical analysis and bioassay studies of toxic (non-regulated) emission compounds.

The section 211(b) testing included neat HySEE Biodiesel, neat REE (Rapeseed Ethyl Ester), 50:50 blend of HySEE, and Phillips 211b reference diesel (2-D). The engine performance and emissions testing results, using a 1997 Caterpillar 3406E engine are shown in the following table.

Samples of both particle-phase and vapor-phase emissions were collected for determination of PAHs, Nitro-PAHs and mutagenicity.

For the particle phase, the HySEE fuel emissions rate for PAHs such as pyrene, benz(a)anthracene, chrysene, the emission rate was considerably lower than the PAH emission rate for diesel fuel. The emission rate for the vapor-phase PAHs from the HySEE fuel was lower than for the 50:50 blend and 100% diesel fuel samples. For example, the emission rates for phenanthrene were about 29.08, 47.4, and 76.3 μg/bhp-hr for the HySEE, 50:50 blend, and diesel fuel hot start cycles, respectively.

| Fuel Type | Power* | Fuel Usage* | THC** | CO** | NOx** | PM** |
|---|---|---|---|---|---|---|
| Hydrogenated Soy Ethyl Ester (HySEE) (Neat) | -7% | +13% | 0.026 | 0.632 | 4.78 | 0.038 |
| Rapeseed Ethyl Ester (REE) (Neat | -5% | +11% | 0.03 | 0.674 | 5.02 | 0.039 |
| 50:50 Blend HySEE and 2-D | -3.5% | +6% | 0.061 | 0.924 | 4.68 | 0.05 |
| Petroleum Diesel (2-D) – 211b reference fuel | | | 0.12 | 1.46 | 4.55 | 0.077 |

*Brake power and fuel consumption results are relative to 2-D baseline fuel.
**Composite average, Cold and Hot values in g/bhp-hr.

Filter and PUF samples were analyzed for 7 different nitro-PAHs. Only 9-nitroanthracene and 1-nitropyrene were detected and could be quantitated. The emission rate using the HySEE fuel was 0.06 and 0.12 µg/bhp-hr for 9-nitroanthracene and 1-nitropyrene, respectively. For the 100% diesel fuel, the emission rate was approximately 5 times higher for 9-nitroanthracene, and approximately 3 times higher for the emission of 1-nitropyrene.

Bioassay analyses were conducted on both the particle and vapor-phase samples. Both particle and vapor-phase extracts were mutagenic. The 100% diesel emission samples were the most potent having specific mutagenic activities (mutagenic activity per mass of particulate matter or per µL of PUF extract) at least 2 times the activity of the HySEE sample. The HySEE mutagenicity emission rates were approximately 6-times lower than the diesel emission rates with metabolic enzymes added and approximately 5 times lower without metabolic enzymes added. The mutagenicity emission rates for the 50:50 blend were higher than the 100% HySEE emission rates, but lower than the 100% diesel emission rates.

## INTRODUCTION

The Pacific Northwest and Alaska Regional Bioenergy Program, in cooperation with several industrial and institutional partners initiated a long-haul (200,000-mile) operational demonstration using a Caterpillar 3406E Engine[1] (435 HP) and a Kenworth Tractor (truck). This three-phase project was designed to:

- develop definitive Biodiesel performance information when used in heavy-duty commercial applications - engine performance data and fuel compatibility, emissions characteristics and levels, and general fuel performance in varying operational conditions. In addition, this demonstration included identifying - fuel properties and characteristics; fuel performance within the combustion chamber; and engine compatibility and durability.
- develop Biodiesel emissions (regulated and non-regulated gases and particulate compounds) profiles in large heavy-duty engines. Collect and expand operational data as required under Section 211 (b) and (e) of the Clean Air Act (New Fuel and Fuel Additives) (1). Assess Biodiesel health and environmental advantages over petroleum diesel fuels. For example, Biodiesel offers significant improvements in biodegradability, reduced fuel toxicity, and reductions in PAHs[2] and other air toxic compounds, while also showing reductions in regulated emissions.
- assess comparative Biodiesel operational performance between two classes of diesel engines (Cummins 5.9 Turbo Diesel Engine and the Caterpillar 3406E Diesel Engine) - emissions, toxic compound reduction, power, torque and deposit formation.
- collect heavy-duty operational engine performance and durability information - demonstrated the use and performance of a 50% Biodiesel/petroleum diesel blended fuel in an actual heavy-duty over-the-road application. This 200,000-mile demonstration assessed engine and fuel performance, lubrication oil dilution and systems impacts over the operational period. To assess long-term engine durability and wear; including injector, valve and port deposit formations; the engine was dismantled for inspection and evaluation at the conclusion of the demonstration.

The over-the-road demonstration engine was fueled with a 50:50 blend of HySEE Biodiesel and petroleum (2-D) diesel. This demonstration vehicle was used in actual commercial operation. Project participants were:

- U. S. Department of Energy, Office of Transportation Technologies, Pacific Northwest and Alaska, and Northeast Regional Programs;
- Caterpillar Inc;
- Kenworth Truck Company, and Trebar, Inc.;
- J. R. Simplot Company;
- Idaho Department of Water Resources;
- Western States Caterpillar;
- University of Idaho, Department of Biological and Agricultural Engineering; and
- University of California, Department of Environmental Toxicology

[1]The mention of company and trade names is for convenience in description and does not imply an endorsement of the products mentioned nor a discrimination against similar products not mentioned.

[2] Polycyclic Aromatic Hydrocarbon (PAH) - a group of highly reactive organic compounds.

Two separate but identical Caterpillar 3406E engines were used for the project.

- Engine One - Operational Over-the-Road Demonstration - included scheduled engine oil and filter changes and analysis; operational performance monitoring and chassis dynamometer testing; production and supply of Biodiesel (HySEE) fuel. The demonstration was designed to log 200,000 miles during a two-year period on the test engine. At the conclusion of the test the engine was evaluated for mechanical impacts or changes, if any, resulting from the use of the 50% blend of HySEE Biodiesel. This phase included final performance testing including a reassessment of regulated emissions; disassembly of the engine including inspection, and analysis of components; and engine rebuild.
- Engine Two - Engine Break-in and Engine Emissions Testing - addressed engine preparation, performance and emissions testing (for both regulated and non-regulated emissions), sampling and analysis following the EPA Heavy-duty Engine Dynamometer protocol including provisions of the Clean Air Act section 211(b).

The HySEE biodiesel fuel used in this project was processed at the J.R. Simplot alcohol plant in Caldwell using hydrogenated soybean oil from the potato processing plant and alcohol from ethanol made from waste potato products. This is conceivably the only location in the world where both used vegetable oil and alcohol are available at the same location.

After processing, the next step was to deliver the fuel to the test vehicle. Since HySEE has a cloud point of 9° C (48° F) (2), special precautions were taken to assure the fuel would be liquid when being pumped into the truck's fuel tank. The fuel specified for the on the road part of the project was a 50% blend by volume of HySEE and diesel fuel.

In most previous demonstrations with blended fuels, the mixture was prepared by either pre-mixing diesel with Biodiesel or by a blending system in the vehicle to achieve the desired blend of fuel (1,3). Since it was desired to minimize modifications to the truck and because drivers, who were unfamiliar with the project, needed access to the fuel day or night, a method to dispense blended fuel on demand was developed. This consisted of installing a HySEE nurse tank near the J.R. Simplot transportation diesel nurse tank and feeding the two fuels to the truck through a blending valve. Thus the drivers could fuel the truck as they normally would with no intervention required for mixing the two components.

The engines selected were 1997, 3406E 345 kW (435hp) truck engines. The engine specifications are listed in Table 1. Two separate identical engines were used in order to maintain the time frame in this project. One engine was used for the emissions testing while the second engine was installed in the test vehicle. For emissions testing, the engine remained in stock configuration (i.e. the control software and engine hardware were not changed from certification on 2-D). The on-road truck engine represented a challenge for evaluating the engine wear. If the engine was left as received from the OEM (Original Equipment Manufacturer), and operated on biodiesel the power output level would have been less than that of 2-D. Therefore, Caterpillar engineers decided to powerset the engine to the power levels achieved with 2-D while using a 50:50 blend of HySEE biodiesel and diesel. This was done so Caterpillar could compare engine wear rates at equivalent power levels.

Table 1: Caterpillar 3406E Engine Specifications

| | | |
|---|---|---|
| Cylinders | : | In-line 6 |
| Bore x stroke | : | 137 x 165 mm (5.4 x 6.5 in.) |
| Aspiration | : | Wastegate turbocharger |
| Displacement | : | 4.6 L (893 cu. In.) |
| Dry Weight | : | 1301 kg (2867 lb) |
| Valves | : | 4 |
| Fuel System | : | Caterpillar mechanically operated, electronically controlled unit injectors |

Caterpillar inspected, tested, powerset, and shipped one of the 3406E diesel engines to Kenworth Truck in Seattle, WA for installation in a Class 8 on-highway truck. Kenworth truck assembled and pre-tested the truck. It was then delivered to J.R. Simplot Transportation for on-road operation. At the conclusion of the durability demonstration the engine was returned to Caterpillar for post-test inspection and testing.

Personnel from the University of California, Davis provided technical analysis of the non-regulated toxic emissions as identified under 40 CFR 79 Section 211b. The class of compounds considered was the polycyclic aromatic hydrocarbons (PAHs) and their nitro-substituted derivatives (nitro-PAHs). The PAHs and nitro-PAHs are known to be potent mutagens and carcinogens. Once released into the atmosphere, some of the PAHs are also precursors to more potent and toxic compounds (4,5). Another important goal was to use bioassay analyses of the complex mixture of emissions. This approach allows the development of relative indices of the emissions based on damage to genetic material (DNA). Both complex mixtures of the particle and vapor-phases could be evaluated in this manner. A Salmonella microsuspension assay previously reported (6,7) was used in these studies.

The chemical and bioassay analyses of mutagenic activity emissions from HySEE and a 50:50 HySEE:diesel blended fuel were evaluated, relative to the emissions from 100% diesel fuel. An evaluation of the potential exposure or reduction in exposure to toxic compounds present in the exhaust from these fuels is an important component in assessing potential risk and benefits to the public and ecological health.

## OBJECTIVES

The objectives of the project were :

1. To produce sufficient HySEE biodiesel from used French fry oil and ethanol for both emissions and on-road tests. Production of the fuel included regular fuel characterization tests to assure quality control according to Caterpillar recommended specifications.
2. Evaluate the test vehicle's performance as determined by oil analysis, maintenance records and regular chassis dynamometer power tests.
3. Determine changes in transient engine emissions of Oxides of Nitrogen (NOx), Hydrocarbons (HC), Carbon Monoxide (CO), and Particulate Matter (PM) with both diesel and HySEE fuels during the 322,000 km (200,000-mile) on-road test.
4. Determine differences in steady-state engine performance of power, fuel consumption, and fuel rate between pre-test and post-test results on an engine PTO dynamometer.
5. Determine the projected engine life from wear measurements of critical engine components.
6. To evaluate the performance and emissions characteristics of a heavy-duty diesel on-highway truck engine utilizing HySEE biodiesel fuels in EPA 211(b) testing. These performance characteristics include steady-state operation measurements such as torque output, power, and fuel consumption. Regulated and unregulated emissions levels were also measured.
7. Quantify the PAH and Nitro-PAH emissions resulting from use of HySEE fuel.
8. Determine the toxicological nature of HySEE emissions from particle and vapor-phases based on damage to genetic material (DNA).

## REVIEW OF LITERATURE

There have been many projects demonstrating the use of biodiesel in various diesel vehicles. Abstracts have been compiled by the National Renewable Energy Laboratory concerning biodiesel demonstrations and research from 1992 to 1997 (8). A partial listing of vehicle demonstrations includes (but is not limited to) the following: a steamboat powered by biodiesel on the Ohio River; a biodiesel bus at the 1996 Atlanta Olympic games; a South Carolina truck running on soydiesel; a Virginia 43-foot work boat running on 20% biodiesel blend; and a Wisconsin bus running 20% biodiesel blend.

Personnel at the University of Idaho have conducted endurance studies on several types of biodiesel fuels and blends of biodiesel and diesel. Some of the light-duty truck demonstrations include the Yellowstone National Park's Truck-in-the-Park running 149 492 km (92,890 mile) burning 100% Canola ethyl ester (9); a 160 934 km (100,000-mile) test on a 20% blend of rapeseed ethyl ester in diesel 3); and a 160 934-km (100,000-mile) test of 100% REE in a 1994 Dodge pickup (10).

Peterson et al. (3) described a 1992 Dodge 2500, 3/4 ton truck running a 27.9/72.1 (volume/volume) blend of rapeseed methyl ester (RME) and diesel fuel for over 160 934 km (100,000 mile). An on-board mixing system was installed with the intent to mix a 20/80 blend (20RME) for the pickup truck. The mixing system included float switches to control the delivery of the desired volumes of diesel and biodiesel. A fuel heating system was installed on the test vehicle to assure cold weather operation in the winter months. Viton fuel lines were installed to reduce the risk of rubber deterioration. The pickup's fuel filter was changed 13 times due to power loss problems over the course of the four-year test. Rust formation and filter plugging prompted a change from mild steel fuel and mixing tanks to stainless steel tanks. The pickup was tested on a chassis dynamometer seven times during the demonstration to monitor and compare power produced while using 100% RME, 20RME, and diesel fuels. The researchers reported that 20RME averaged 1.47% less power and RME averaged 4.96% less power than the low sulfur diesel fuel used in the dynamometer tests. The power differences were attributed to the differences in heat of combustion of the RME, 20RME and diesel fuels.

Taberski (9) reported the results of running a 1995 Dodge 2500 four wheel drive pickup 149 408 km (92,838 mile) on 100% Canola and rapeseed ethyl esters. Overall, 23 076 liters (6,096 gal) was produced for the project. Fuel was stored in a 37 850-liter (10,000-gal) tank with portions stored inside a maintenance shop in case of cold weather. Chassis dynamometer testing showed that power from the ethyl esters was comparable to diesel fuel. Valve opening pressure was observed to decrease over the course of the test but was "not abnormal for the mileage of the engine." Engine oil analyses for wear metals were found to be normal.

Peterson et al. (2) tested HySEE in blends with number two diesel in a 1000-hour EMA engine test. Three blends were tested in stationary Yanmar 3-cylinder diesel engines including (percent volume HySEE / percent volume Diesel) 25/75, 50:50, and 100/0. They observed that the 25, 50, and 100% blends of HySEE had cloud points of 3, 5, and 9° C (37, 41, and 48°F), respectively and pour points of –33, -9, and 6°C (-27, 16, and 43°F), respectively. Fuel filter plugging was a problem in the winter months when HySEE would crystallize. They reduced the filter-plugging problem by heating the fuel tanks and the fuel filter with band heaters. They observed that the 100% HySEE engine was cleaner internally than the other two engines. They concluded that cold weather operation required fuel heating; burning neat HySEE resulted in a cleaner looking engine than the others; the analysis of the engine oil for the neat HySEE engine showed equal or reduced wear metals than the other engines; and the injector pressure and compression was unchanged for each engine.

No report was found in the literature about projects using HySEE as a feedstock in a large-scale, long-term endurance test. The precedents have been made for testing biodiesel and biodiesel blends in boats, busses, and light-duty trucks. The next step would be a controlled endurance demonstration using a heavy-duty tractor truck.

To our knowledge this is the first, and only, operational biodiesel demonstration utilizing advanced diesel engine fuel injection technology – Caterpillar's Electronic Unit Fuel Injectors – thus providing technical performance data related to the concerns expressed by the diesel fuel injection equipment manufacturers. Fuel produced and used in this project met Caterpillar's recommendation and requirements for esterified vegetable oil fuels. Several studies have reported results of regulated emissions tests with biodiesel (11,12). In general these studies showed reduced HC and CO. In PTO engine tests $NO_x$ is generally increased and PM reduced; for chassis dynamometer tests the reverse has been found to be true.

No PAH, Nitro-PAH or mutagenicity studies with biodiesel were found in the literature. However, similar data is being completed by Kado (13) from a biodiesel study in Yellowstone National Park.

## MATERIALS AND METHODS

### FUEL PRODUCTION

The used hydrogenated soybean oil for each batch was obtained from the J. R. Simplot Processing Plant. Ethyl alcohol (ethanol) was obtained from the J. R. Simplot Ethanol Plant. The catalyst used was 85% flake caustic potash (KOH) in 22.7 kg (50 lb) bags (Van Waters and Rogers Company, Nampa, ID). Ninety-nine percent tannic acid (Sigma Chemical Company, St. Louis, MO) was used in the acid wash step to neutralize residual KOH from the reaction step. Ninety-nine percent normal octanol (Sigma Chemical Company, St. Louis, MO) was used in both water washing steps to reduce the possibility of emulsification. The Biological and Agricultural Engineering (BAE) Analytical Lab at the University of Idaho, using materials normally available to the lab, prepared titration solvent, alcoholic KOH, and indicator for the free fatty acid analyses according to ASTM method D974-80 (14).

The alcoholysis reactions were performed in the distillation room of the Ethanol Plant with a 2839 L (750) gallon stainless steel reactor, Figure 1. Supplementary batches were produced at the University of Idaho. Hydraulic equipment was used in all steps of fuel production to meet the hazardous location standard for the Ethanol Plant's Distillation room. A mixture of steam and potable water was used in the water washing routines at the Ethanol Plant.

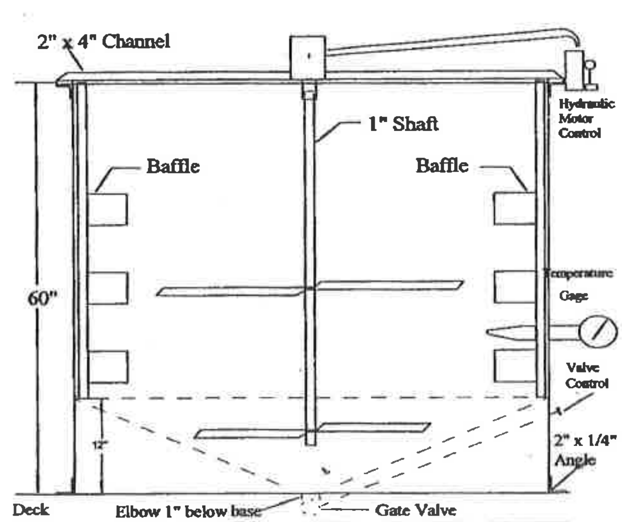



**Figure 1.** Schematic of the reactor used for 1893 L, (500 gal) batches of HySEE production.

Used oil was pumped from heated oil storage tanks at the Processing Plant into an 1893-liter (500-gal) metal tank and was transported to the reactor. Oil temperatures in the metal tank ranged from 71°C to over 93°C (160 to 200°F). Absolute ethanol for each batch was stored in 208-liter (55-gal) barrels in the distillation room and was used as needed. A Mettler DL37 Karl Fischer Coulometer tested the ethanol before each batch for percent water. The KOH bags were delivered in 10-bag lots, stored in a storage facility at the J. R. Simplot Company, and used as needed.

The BAE method for producing biodiesel has been published in earlier papers, for example Peterson et al. (15,16,17,18,19). The KOH was dissolved in the ethanol and agitated at a high rate for at least 15 minutes to ensure that the KOH was completely dissolved. The ethanol and KOH mixture was added to the oil while the oil was being stirred. Agitation lasted for at least four hours to ensure complete reaction. After the water washing steps, the final ester was removed from the top of the reactor and pumped through a high-efficiency diesel fuel filter (Caterpillar Inc., Peoria, IL) to remove any remaining particulate. Each batch required four days from start to finish.

After analysis for quality control, the HySEE was stockpiled in 1040-liter (275-gal) plastic totes (Schütz, St. Peters, MO). The fuel was subsequently pumped into an insulated 37,805-liter (10,000-gal) stainless steel tank. The tank was equipped with a centrifugal pump and a two-inch stainless steel recirculation pipe routed from the bottom to the top of the tank. The recirculation action and heat from the pump was observed to adequately heat the HySEE in the tank through the winters of 1997-98 and 1998-99. HySEE temperature was maintained between 26 and 49° C (80 and 120° F).

FUEL COST

The cost to produce the fuel was determined from an invoice of materials prepared by the Ethanol Plant. The materials for HySEE production and their associated costs were itemized. Profit, taxes, operating costs, labor, and capital recovery costs were not included in the cost to produce the fuel.

QUALITY ASSURANCE

The participants of this project agreed upon a HySEE quality assurance plan. The list of the specifications required by Caterpillar Inc. is in Table 2. Each batch was to be evaluated for process sensitive characteristics including viscosity, free glycerol, total glycerol, alcohol content, acid number, density, and potassium.

The other fuel characteristics in Table 2 would be evaluated for samples taken of the first 3785 liters (1000 gal) and other samples taken of the fuel in storage each year during the demonstration. Total glycerol weight percent is considered to be an indicator of the completeness of the HySEE reaction. The fuel's ethanol weight percentage, measured at the Ethanol Plant, was also used to determine if the fuel required further washing.

HySEE samples were sent to Williams Laboratory Services (Kansas City, KS) to quantify free and total glycerol using the Christina Plank method for impurities in Biodiesel Methyl Esters in a gas chromatograph (20). The resulting analyses from Williams Laboratory were correlated with free and total glycerol results from method AOCS Ca 14-56 (21) performed at the BAE Analytical Lab at the University of Idaho in Moscow. The ethanol content of each HySEE batch was found using a Shimadzu LC-10AD liquid chromatograph with a Shimadzu RID-6A refractive index detector operated by Ethanol Plant personnel.

Water and sediment; sulfate ash; cetane number; iodine number; Conradson carbon residue; and sulfur content were determined by Phoenix Chemical Laboratory (Chicago, IL). The BAE Analytical Lab (Moscow, ID) determined the remainder of the HySEE properties listed in Table 2.

## DIESEL FUEL

The diesel fuel used in the emissions tests was Phillips 211(b) reference fuel (Phillips Chemical Company, Specialty Chemicals, P. O. Box 968, Borger, TX 79008). The diesel fuel used for the blended fuel used in the over-the-road test was commercial diesel fuel as supplied to the J. R. Simplot, Inc. Transportation fueling depot.

## THE TEST VEHICLE

A Class 8, T800 truck supplied by Kenworth was powered by a Caterpillar 324 kW (435 hp), 3406E engine (serial number 6TS06750). Caterpillar Inc. programmed the engine to deliver 324 kW (435 hp) of power while burning a 50% blend of HySEE and 50% diesel fuel. The truck was equipped with a retractable third rear axle to accommodate heavy loads. The truck was linked to a 28 390 liter (7,500 gal), 16 m (53-ft) long, triple axle liquid transport trailer. Figure 2 shows the truck and trailer in use.

The truck was equipped with an "Arctic fuel package" that included insulated fuel lines, a heated fuel filter, and a tank heater using engine coolant to maintain a warm fuel tank temperature. The truck was parked inside a heated shop when not in use during cold weather conditions (below 10° C (50° F)). The truck and trailer transported molasses solution from a Caldwell feed plant to a Simplot feedlot in Grandview, Idaho, two to three times per day, depending on the molasses demand at the feedlot. The trailer was unloaded in Grandview and hauled back to Caldwell empty at an empty weight of 6532 kg (14,400 lb). Round trip mileage was between 225 and 290 km (140 and 180 mile) depending on the route chosen by the truck operators.

Engine and truck data were obtained from the truck's dashboard gages and through an electronic interface called the Caterpillar Information Display (CAT-ID). The information display was used to track the activity of the truck's engine whenever the ignition of the truck was "on". Vehicle operation statistics were stored in the CAT-ID's memory including engine hours, miles traveled, gallons of fuel used, average truck speed, average miles per gallon, average miles per hour, idling time, and idling fuel used. An hour gage was installed in the cab of the truck to read truck hours. The truck's odometer was used to indicate how many miles the test vehicle traveled for fuel economy calculations and for comparison with the CAT-ID.

**Table 2.** Caterpillar Inc. Specification Used for the Over -the-Road Project

| Fuel Specific Properties | Units | Maximum HySEE Limits | Test Methods (*ASAE Standards*, 1996) |
|---|---|---|---|
| Density at 15° C | g/mL | 0.86 – 0.90 | ASTM D-1298 |
| Viscosity | $mm^2/s$ | 4.0 – 6.0 | ASTM D-445 |
| Cold Filter Plugging[1] *Summer *Winter | ° C | 0<br>6 deg. Below Ambient | ASTM D-4539 |
| Sulfur Content | wt. % | 0.01 maximum | ASTM D-2622 |
| Carbon Residue (100% sample) -Conradson | wt. % | 0.050 maximum | ASTM 4530<br>ASTM D-189 |
| Cetane Number | | 45 minimum | ASTM 613-86 |
| Sulfate Ash | wt. % | 0.02 maximum | ASTM D-874 |
| Water and Sediment | vol. % | 0.05 maximum | ASTM D-1796 |
| Cloud Point[1] | ° C | | ASTM D-2500 |
| Copper Strip Corrosion (3 hr @ 50° C) | | No.1 | ASTM D-130-88 |
| Oxidative Stability | $g/m^3$ | 20 | ASTM D-2274 |
| Acid Number | mg KOH/g sample | 0.5 maximum | ASTM D-664 |
| Ethanol Content | wt. % | 0.2 maximum | LC method |
| Free Glycerin | wt. % | 0.2 maximum | GC method |
| Total Glycerin | wt. % | 1.2 maximum | GC method |
| Iodine Number | $cg\ I^2/g$ | 120 maximum | DIN 53941/IP 84/81 |

[1]The cloud point, pour point, and cold filter plugging temperatures are a function of the feedstock and cannot be guaranteed to meet a particular limit.

## THE FUELING STATION

A single-walled, 10-gauge, 304-l stainless steel, 1893-liter (500-gal) tank mounted to a metal skid (Isom Brothers, Caldwell, ID) was installed at the Simplot Transportation fueling depot. The fuel vault was equipped with a 0.25-kW (1/3-hp) submersible pump (F.E. Petro) and control box, a 1200-Watt immersion heater with a thermostat set to 32° C (90° F). A 28-liter (7.5-gal) metal spill containment compartment and metal cover surrounded the fill cap. Figure 3 shows the fuel vault after it was installed.

After final installation, the fuel vault was insulated with spray-foam insulation to maintain the HySEE temperature above its cloud point. The insulation and immersion heater was provided to ensure the delivery of liquid fuel during winter months.

Connections were made from both the HySEE nurse tank and a diesel nurse tank to a Schlumberger J 50:50 mixing valve set to mix equal volumes of HySEE and diesel. The piping was then routed to a Liquid Controls M5-A-1 meter and a 25.4-mm (1-in.) curb hose connected to a high flow diesel nozzle. The diesel fuel used in this project was supplied from Simplot Transportation's 3785-liter (1000-gal) diesel nurse tank, which also supplied diesel fuel for the Simplot trucking fleet based in Caldwell. A switch mounted to the fuel vault support platform simultaneously activated the diesel and HySEE fueling pumps. This system assured that the Kenworth driver could simply turn on the switch, place the fuel nozzle into the fuel tank, and pump 50:50 fuel without additional monitoring or human error in mixing the fuel.

Calibration of the fuel blend was determined by weighing samples of diesel, HySEE, and 50:50 blend in separate tared 500 mL volumetric flasks at a common temperature of 20° C (68° F). The blend composition was determined and reported by the BAE Analytical Lab.

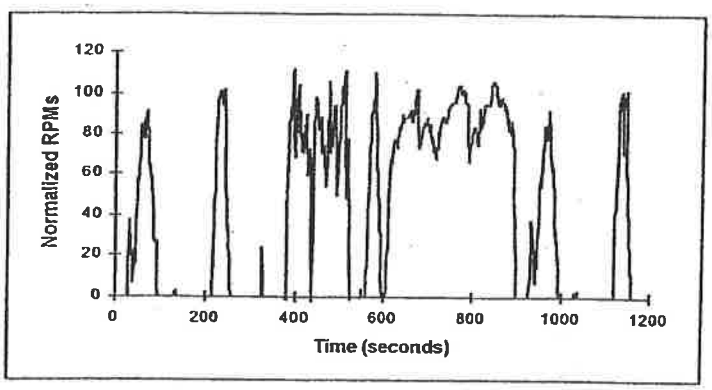
**Figure 2.** The test vehicle for the over-the-road demonstration.

**Figure 3.** The fuel vault with the blending valve and fuel meter in the foreground and the diesel nurse tank to the left in the background.

CHASSIS DYNAMOMETER TESTING – WESTERN STATES CATERPILLAR

For the demonstration project, the truck was to complete 322,000 km (200,000 miles). The truck was to be periodically tested on a chassis dynamometer to monitor the engine's horsepower and to monitor the 50:50 fuels' effects on the engine. The dynamometer tests were to occur at 0, 40 250, 80 500, 161 000, 241 500, and 321 869 km (0; 25,000; 50,000; 100,000; 150,000; and 200,000 mile, respectively). The dynamometer testing of the truck was performed at Western States Caterpillar in Boise, Idaho, with a Superflow 501 chassis dynamometer. Each dynamometer test consisted of three runs through a power curve at engine speeds of 1200, 1400, 1600, 1800, and 2090 rpm.

Engine injector cutout tests were performed at 1600 rpm to track injector fouling. The dynamometer operator turned off each engine injector and, at steady state engine operation, recorded the corrected wheel power. If a particular injector failed, a reduction in the change in power relative to a normal injector would be observed.




. **Figure 4**: EPA Heavy-duty Diesel Engine Transient Test Cycle -- (CFR40, Pt. 86, Subpt. N)

**Engine Oil Analysis** - Samples of engine oil were taken at approximate 6,000 mile intervals by Simplot mechanics and were analyzed by Caterpillar Inc. (Peoria, IL) and Chevron Lube Watch™ (Spokane, WA) to monitor the engine's wear and detect any lube oil deterioration. Oil changes were conducted at 25,000-mile intervals

**ENGINE TESTING METHODS – CLEAN AIR ACT SECTION 211(B) EMISSIONS TESTS**

TEST PROTOCOL

The testing in this program was conducted according to the requirements set forth in the Code of Federal regulations, 40 CFR parts 79 and 86. Part 79 of the CFR deals with Fuels and Fuel additive registration regulations, while Part 86 subpart N describes methods of testing and emissions sampling of heavy-duty diesel engines (22).

ENGINE BREAK-IN (211 ENGINE)

A new production Caterpillar 3406E diesel underwent 125 continuous hours of break-in using 100% HySEE biodiesel. This engine remained in the original certified configuration with less than 12 hours on the engine chronometer [40 CFR 79.57 (a)] following normal Caterpillar manufacturing methods this engine underwent initial assembly line testing on 2-D diesel. The break-in required approximately 2700 gallons of HySEE. The test cycle used for the engine break-in was a Caterpillar developed schedule. This schedule consists of three steady state operation points. The break-in ran flawlessly to the completion of the 125 hours test. Once the engine completed the break-in period, the fuel injectors were completely disassembled and inspected by Caterpillar fuel systems experts. There were no signs of wear or degradation at the time of inspection. The injectors were then reassembled and installed in the engine in preparation for testing.

TEST MATRIX

The test schedule utilized in this program was the transient test cycle for heavy-duty diesel engines, Figure 4. This is the same test cycle used for on-highway diesel engine certification. According to 40 CFR part 79, a ratio of Cold to Hot test results must be maintained. This ratio is 1/7 the value of the Cold run results averaged with 6/7 of the value of the Hot run results. This weighted average must be collected over six separate days to represent an unbiased emissions sample.

PERFORMANCE TESTING

Caterpillar ET (Engine Testing) procedures were followed during engine performance testing. This Caterpillar protocol stipulates the recording of engine data while operating a steady state condition. Typically these points are taken at full load. Engine inlet air is maintained at 25°C. while inlet manifold temperature is set at a maximum of 46°C. Inlet air restriction is also set at a maximum of 96 kPa. Many engine parameters were measured during this part of testing. Some of these parameters include engine torque, power, speed, and fuel rate along with many different temperature and pressure points.

## EMISSIONS TESTING

Caterpillar is an accepted EPA engine test facility. Regulated exhaust emission constituents measured in this program include Oxides of Nitrogen (NOx), Total Hydrocarbons (THC), Carbon Monoxide (CO), Carbon Dioxide (CO2), Total Particulate Matter (TPM).

Unregulated emissions were also measured for this program. These included aldehydes and ketones collected using impinger/DNPH method, Soluble Organic Fraction (SOF) – sampled on a 90 mm filter and analyzed using gas chromatography, Sulfates – samples on a 90 mm filter and analyzed using ion chromatography, Hydrocarbon speciation (C1– C12 and C13 – C22) – analyzed using gas chromatography and mass spectroscopy; a method similar to CARB protocol using solid adsorbent tubes rather than Tedlar bags, was followed for C1 – C12 compounds. PAH and NPAH compounds were collected on 90 mm filters as well as polyurethane foam (PUF) filters

An auxiliary particulate sampling system was used to collect sufficient TPM mass for NPAH testing. This apparatus was a model BG-1 micro-dilution system from Sierra Instruments.

## ALDEHYDES AND KETONES

Transient emissions from the CAT 3406E were collected and analyzed for 13 aldehydes and ketones using an established impinger/dinitrophenylhydrazine method (impinger/DNPH). Emissions samples were taken using a specially designed sample cart.

Diluted exhaust from each of two channels was "bubbled" through two impingers in series (each containing DNPH dissolved in acetonitrile) at approximately 5 liters per minute. The impingers were contained in an ice bath to minimize volatility losses. After the cycle, the impingers and their contained DNPH were taken to the emissions Laboratory where the solutions were quantitatively transferred, diluted to volume, and transferred to HPLC autosampler vials for analysis. Each impinger, both primary and secondary, was analyzed separately. This process produced two independent samples (each from a primary and a secondary impinger) per Transient test.

Hewlett-Packard 1050 high performance liquid chromatography (HPLC) instrumentation was used through for the chemical analyses. The HPLC was equipped with a photodiode array detector, which allowed us to use computer spectral matching to validate the identity of the individual compounds, and to assure that the results are accurate and not caused by contaminants. Gradient elution of the mobile phase (consisting of acetonitrile, water, and tetrahydrofuran), through a Waters Nova-Pak™ C18 3.9 x 150 mm column allowed us to separate the 13 analytes.

Retention times and calibration curves were made using multicomponent aldehyde/ketone standards from Radian Corporation. One of more DNPH blanks were taken and run during each day that emissions samples were collected. Calibration check standards were run after every 4$^{th}$ sample to ensure proper calibration.

## HYDROCARBON SPECIATION

Qualitative speciation of the hydrocarbon emissions was conducted to identify the dominant compounds in the diluted exhaust of the Cat 3406E Engine. Transient hydrocarbon emissions were collected and concentrated on ¼" by 7" Carbotrap™ 300 thermal desorption tubes (Supelco No. 2-0370), from secondary dilution tunnel flow, through a heated (190°C) sample line connected to our specially designed sampling cart. With this apparatus, approximately 16 to 20 liters of dilute exhaust were sampled and measured during the 20-minute cycle. The total collection volume was recorded from a calibrated gas meter mounted on the cart. This was the same cart that was used to collect aldehyde emissions, which allowed simultaneously collection of samples for aldehyde analysis and for hydrocarbon speciation. Thus, hydrocarbon samples were collected from one of the carts four independent channels, aldehydes were sampled from two of the channels, and one channel was not used. The sorption tubes were desorbed with a Tekmar Aerotrap 6000 desorber. Then the analytes were transferred and cryofocused onto a Supelco VOCOL™ 60m x 0.32 mm ID x 1.8 µm film thickness column in a Hewlett-Packard 5890 Series II gas chromatograph (GC). The column was interfaced to a Hewlett-Packard 5989B MS Engine Mass Spectrometer (MS). Afterwards, based on the identified compounds, chemical standards were selected and run through the same instrumentation to provide the basis for quantification of the selected species.

## QUALITATIVE IDENTIFICATION

Identification of the compounds in an approximate C2- C12 range was aided by use of Hewlett-Packard MS Chemstation software. Additional compounds were present at lower levels, however these had signals too weak and noisy to positively identify. Those compounds whose identification would be speculative are not listed. For the most part, enough information was present to determine that these unidentified peaks consisted of various branched alkanes and of various alkyl benzenes.

To obtain quantitative information and based on the presence of compounds that appeared to be dominant in the chromatograms, standards were purchased and/or prepared for the following species: ethyl acrylate, benzene, toluene, n-nonane, ethyl benzene, m-xylene, o-xylene, n-decane, isopropylbenzene, 1,2,4-trimethylbenzene, n-undecane.

Mixed standards were purchased from Supelco for the aromatic compounds. The alkane standards were prepared from methylene chloride dilutions of the bulk alkane compounds. Ethyl acrylate standards were prepared from aqueous dilutions of ethyl acrylate purchased from Aldrich Chemical Company.

RANGE LIMITATIONS

The sorption media and the analytical conditions in the hydrocarbon speciation tests were optimized for the analysis of the more volatile C2 – C12 compounds. However, the upper size limit for hydrocarbons was not known. To determine this, a diesel range organic standard containing C10 to C28 n-alkanes was tested. Alkane hydrocarbons of larger than approximately C12 were the practical upper limit. It was found that the limit could be extended to at least C20 by increasing the MCS (moisture control system) desorb temperature of the Tekmar unit from our setting of 35°C to 300°C. However, the impact of this change on the more volatile compounds is not known and was not addressed. Therefore, analysis of compounds greater than C12 would need to be addressed at a later date.

## EMISSIONS TESTING– UNIVERSITY OF CALIFORNIA

Instrumentation, chemical standards and techniques for the PAH, nitro-PAH and mutagenicity tests were similar to that reported by Kado (23) and will not be given in detail in this paper.

The particulate and vapor-phase (primarily semi-volatile) emissions from the hydrogenated soy ethyl ester (HySEE), 50:50 blend of HySEE: diesel, and diesel fuels were analyzed for PAHs, nitro-PAHs, and mutagenic activity. The analyses for the particulate matter-associated PAHs were based on cold and hot cycles, with three consecutive cycles composited for each sample. The analyses for the vapor-phase PAH were conducted with duplicate samples, with each sample a composite of three consecutive cycles. The particle and vapor-phase nitro-PAHs quantitation was based on duplicate samples with each sample consisting of a composite of three consecutive cycles. The bioassay samples for both the particulate and vapor-phase were tested for duplicate samples with each sample representing three consecutive cycles. The complete experiment for both chemical and bioassay analyses included 6 cold and 36 hot cycles for the HySEE fuel and 3 cold and 12 hot cycles for the diesel fuel emissions.

Particulate matter was collected on 90-mm diameter Teflon-coated glass fiber filters (T60A20; Pallflex; Putnam, CT). The filters were pre-cleaned with methanol and DCM, dried in an HEPA-filtered hood, and were preconditioned for at least 24 hrs. in a humidity and temperature-controlled room before use in the sampling. The filters were weighed in the testing facility temperature and humidity controlled weighing room before and after sample collection. The temperature in the room was maintained at 21°C (+ 2°C) and the relative humidity was 50% (+ 10%). The filters were stored in layers of glassine paper with a box prior to weighing.

Samples were collected from the secondary dilution tunnel for the bioassay and PAH analyses. To acquire adequate sample mass for the nitro-PAH analyses, a medium volume dilution sampler was used and was provided by Caterpillar. The medium volume sampler had a nominal flow rate of 190 Lpm at a dilution rate of 4:1. After sampling, the filters were reconditioned in the temperature-humidity controlled weighing room for at least 2 hours before a final weight was taken. Filters were then individually wrapped in glassine paper, foil-wrapped, coded, and temporarily stored at 4°C until shipping the next day for overnight delivery (packed in blue ice) to the University of California Davis laboratory. Upon receipt, samples were immediately inventoried using the enclosed tracking sheet and transferred to a -20°C freezer for storage.

BIOASSAY ANALYSES

Bioassay experiments were conducted to determine the specific mutagenic activity of the particle and vapor-phase extracts. Filter samples were extracted with DCM and sonication. The extract was filtered through a pre-cleaned Teflon filter (Gelman, CR PTFE, 0.45µm.) PUF samples were extracted using supercritical carbon dioxide in a procedure reported previously (23). For all samples, the specific mutagenic activity is reported as the number of revertants per mg of particulate matter or for the PUF samples, as microliter of sample extract. The specific mutagenic activity is determined from the slope obtained from the linear portion of the dose-response curve. The bioassay used throughout is the microsuspension assay as previously reported (6,7). All extracts were resuspended in DMSO for addition to the assay.

Instrumental conditions and column selection – The filter and PUF extracts for the PAHs were analyzed using a Hewlett-Packard Model 5890 Series II gas chromatograph (GC) equipped with a Model 8290 autosampler interfaced to a Hewlett-Packard Model 5970A quadrupole mass selective detector (MSD). The GC was equipped with a split/splitless injector and an electronic pressure controller. The injector was run in the splitless mode and the electronic pressure controller was programmed for vacuum compensation and constant flow mode. The GC was equipped with a 2 m x .25 mm i.d. deactivated silica pre-column connected to a 30 m x 0.25 mm i.d. DB-5 fused silica capillary column (0.25 µm film thickness J&W Scientific). The MSD was run using both the selective ion monitoring (SIM) and electron impact (El) modes.

The PM and PUF extracts for the nitro-PAHs were analyzed using a Hewlett-Packard Model 5890 Series II Gas Chromatograph (GC) interfaced to a Hewlett Packard Model 5972 quadrupole mass selective detector (MSD). All samples were injected cool-on-column using both the selective ion monitoring (SIM) and electron impact (El) modes following the methods of Arey et al., (1994) A 1 m x .32 mm id deactivated silica pre-column connected to a 30 m x 0.25 mm id DB-5 fused silica capillary column (0.25 µm film thickness was used for this analyses. The temperature program started at 40°C with a 2 min. hold, followed by 15%/min to 200°C, followed by a 4°C/min to 320°C with a 4.5 min hold. The detector was set at 300°C.

## RESULTS AND DISCUSSION

### HYSEE PROCESSING

A total of 80 152 liters (21,174 gal) of HysEE was produced. About 70 636 liters (18,660 gal) were produced at the Simplot location supplemented by 9517 liters (2,514 gal) produced at the University of Idaho during 1997. The 53 Simplot batches were made between June 1997 and August 1998. Each batch was evaluated for process sensitive characteristics including viscosity, free glycerol, total glycerol, alcohol content, acid number, density, and potassium. Table 3 lists the fuel characterization data for fuel samples from the bulk fuel storage taken at three different times during the tests.

The used frying oil was between 0.72 and 2.14 wt% free fatty acids (FFA). The oil was rarely above two weight percent FFA. The delivered oil required no special processing to prepare it for reaction.

The volumetric percent yield of HySEE appeared to be correlated to the amount of free fatty acids in the feed stock, Figure 5.

All of the eleven University of Idaho (UI) batches were within the specified viscosity range of 4.0 to 6.0 $mm^2/s$. Five batches produced at the Simplot location were above the 6.0 $mm^2/s$ specification for viscosity. The viscosity values were 6.12, 6.04, 6.13, 6.31, and 6.07 $mm^2/s$, respectively. Because the five batches were within the remaining quality assurance specifications, the batches were blended with the existing HySEE inventory.

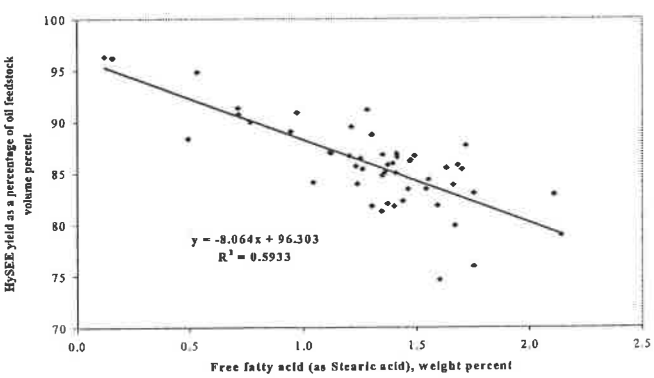



**Figure 5.** Graph of HySEE yield as a function of free fatty acids in the HySEE feedstock.

Total glycerol as measured for each of the batches of HySEE produced at Simplot is shown in Figure 6.

None of the Simplot and UI batches were above the free glycerol specification of 0.2 wt% and all of the UI batches were below the maximum 0.2 wt% specification for alcohol content. Two of the Simplot batches were above the alcohol specifications. The two batches were within the other fuel specifications and were blended with the existing inventory. the alcohol content of the inventory was always within specification.

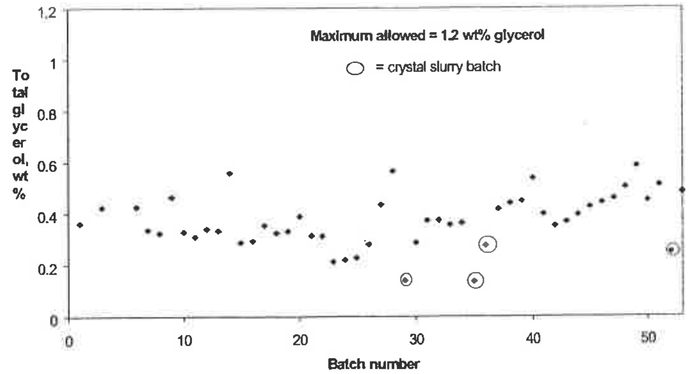



**Figure 6.** Graph of total glycerol for the Simplot HySEE batches.

**Table 3. HySEE Properties for Samples Taken from The HySEE Storage Tank**

| HySEE Specific Properties | Lot 1 | Lot 2 | Lot 3 |
|---|---|---|---|
| Density at 15° C, g/mL | 0.872 | 0.873 | 0.875 |
| Viscosity, $mm^2/s$ | 5.07 | 5.66 | 5.90 |
| Sulfur Content, wt. % | <0.005 | <0.005 | <0.005 |
| Carbon Residue (100% sample) -Conradson, wt. % | 0.05 | 0.05 | 0.07 |
| Cetane Number | 51.6 | 60.5 | 56.9 |
| Sulfate Ash, wt. % | 0.005 | 0.003 | 0.002 |
| Cloud Point, °C | 8 | 8 | 6 |
| Pour Point, °C | 6 | 6 | 6 |
| Water and Sediment, vol. % | <0.005 | <0.005 | <0.02 |
| Copper Strip Corrosion (3 hr @ 50° C) | 1A | 1A | 1A |
| Acid Number, mg KOH/g sample | 0.213 | 0.208 | 0.369 |
| Ethanol Content, wt. % | 0.145 | 0.095 | 0.084 |
| Free Glycerin, wt. % | 0.004 | 0.004 | 0.015 |
| Total Glycerin, wt. % | 0.391 | 0.476 | 0.383 |
| Iodine Number, cg $I^2$/g | 80.19 | 80.71 | 54.2 |
| Fatty Acid Composition, % | | | |
| Palmitic (16:0) | 11.12 | | 11.3 |
| Stearic (18:0) | 11.70 | | 11.9 |
| Unknown (18:1) | 34.69 | | 31.8 |
| Oleic (18:1) | 26.62 | | 28.9 |
| Unknown (18:2) | 10.03 | | 7.8 |
| Unknown (18:2) | 2.58 | | 2.1 |
| Linoleic (18:2) | 1.51 | | 2.3 |

All of the batches were within the acid number specification of 0.5 mg KOH/g sample and the density specification range of 0.86 to 0.90 g/mL and all of the batches were reported to be below the four parts-per-million detection limit for potassium.

Total glycerol averaged 0.402 % with a high of 0.582% and a low of 0.287% and thus was always well within the specified maximum of 1.2%. The free glycerol averaged only 0.0041%. The yield of HySEE as a mass percentage of delivered raw oil averaged 85.4%. Samples were taken from the HySEE biodiesel storage tanks.

## COST TO PRODUCE HYSEE

The unit cost to produce the fuel was $0.55 per liter ($2.08 per gallon). This cost is a materials cost for the used French fry oil, ethanol and catalyst based on 11 UI batches and 50 Simplot batches. The average cost of materials was 11.5 cents/lb for used oil (range of 11 cents/lb to 14.5 cents/lb), $1.40 per gallon for ethanol and KOH was $1.14 per lb. The cost does not include profit, taxes, operating costs, labor, and capital recovery costs. Ethanol recovery could reduce the cost to produce HySEE if an economical method is available to strip ethanol out of the glycerol layer. Recovery and sale of the glycerol could further offset the cost to produce HySEE.

## VEHICLE PERFORMANCE

### ON-ROAD TRUCK OPERATION

The engine was power-set (programmed) by Caterpillar Inc. to deliver a rated power of 324 kW (435 hp) while burning a 50% blend of HySEE and diesel. At the rated power, the engine was observed by Caterpillar Inc. to consume 6.5% more HySEE/diesel blend than the same type of engine power-set with 100% diesel. Based on data from the fuel meter and the truck, the test vehicle consumed 145 746 liters (38,502 gal) and ran 326,235 km (202,713 mile) for an observed fuel economy of 2.19 km/L (5.27 mile/gal). This falls within the range of 2.0 to 2.5 km/L (5 and 6 mile/gal) for a similar vehicle (different engine manufacturer) using 100% diesel on the same trucking route (24).

The truck consumed 145,746 liters (38,502 gal) of the blended fuel according to the fuel meter. The total amount of HySEE consumed by the test vehicle was 70,397 liters (18,597 gal) based on the fuel vault data. The composition of the fuel averaged 51.3% HySEE and 48.7% diesel, based on analysis of the blended fuel. The three analysis of volume percent HySEE / volume percent diesel were found to be 51/49, 51.98/48.02, and 50.8/49.2, respectively.

Figure 7 shows average corrected wheel horsepower values observed for the test vehicle during the dynamometer tests.

The corrected wheel power for the truck peaked at an average of 12.8 kW (17.1 hp) above the maximum tolerance values during the 44,651 km dynamometer test. The corrected wheel horsepower began and ended at nearly the same horsepower for the first and last dynamometer tests, with the exceptions of the 12.7 kW (17 hp) increase for 1198 rpm and the 6.7 kW (9 hp) decrease for 1800 rpm as shown in Figure 13. A steady decrease in power is evident in Figure 13 relative to the maximum power observed at 44 651 km (27,889 mi).

The average fuel rates for the dynamometer tests are presented in Figure 8.

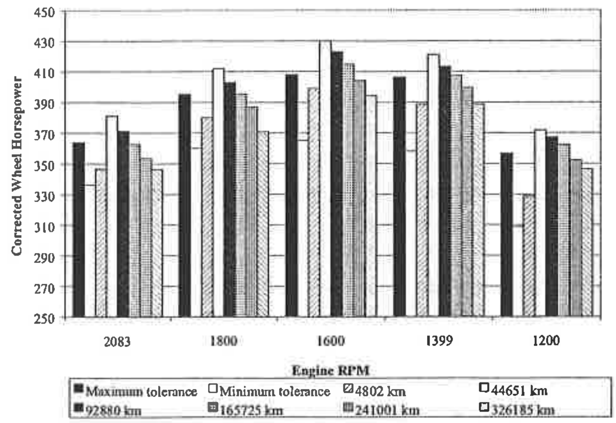



**Figure 7**. Chart of average corrected horsepower for the test vehicle. Maximum and minimum tolerance values were specified by Caterpillar Inc.

The fuel rates for the 44,651 km test averaged 7% higher than the engine manufacturer's expected fuel flow rates. The fuel rates during the last dynamometer test ranged from 0.11 L/h higher to 3.8 L/h lower (0.03 and 1.0 gal/h, respectively) than the fuel rates during the first dynamometer test. The 7.8 to 13% increases in corrected vehicle power between the 4802 km and the 44,651 km tests coincided with 4.8 to 9.5% increases in fuel rates. However, the fuel rate remained relatively constant during the next three tests while the corrected power gradually decreased, implying that the fuel rate was not the only factor effecting the corrected power of the test vehicle the during those tests.

Injector cutout tests were performed to track abnormal wear or fouling in the injectors. The relative horsepower for the injector cutout tests (the corrected wheel power when one cylinder is cut out relative to that when all cylinders are in operation) is illustrated in Figure 9. The relative power during the cutout tests was consistently between 75 and 80% of full horsepower at the constant engine speed. The lack of abrupt changes in relative power indicates that no injector degradation occurred.

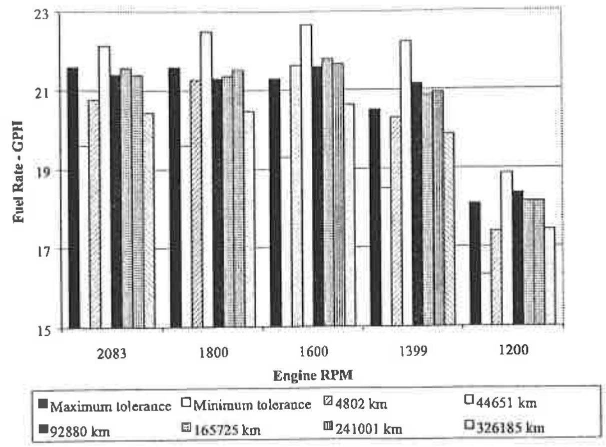



**Figure 8.** Chart of fuel rate for the test vehicle during the dynamometer tests. Maximum and minimum tolerance values were specified by Caterpillar Inc.

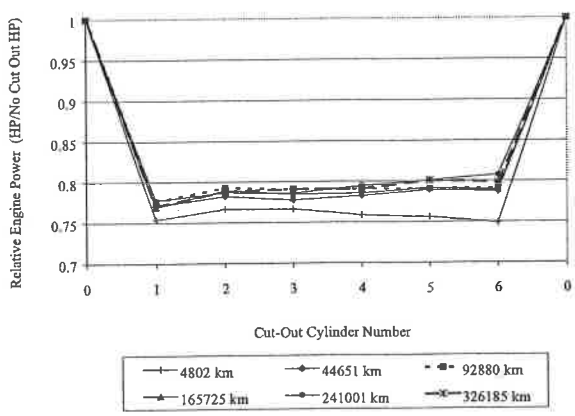



**Figure 9.** Injector cut-out test which shows relative power when the indicated injector is turned off.

The engine oil analyses showed that a cyclical buildup of wear metals occurred between oil changes. The analysis lab recommended that oil be changed when observed copper concentrations exceeded 50 parts per million (ppm). Figures 10 shows selected contaminants monitored by Chevron LubeWatch™. The figure shows early copper concentrations above 50 ppm. The copper concentration in the engine oil decreased as the engine continued operation, indicating the high initial concentrations were due to engine break-in and not due to abnormal engine wear. The average oil change interval was 40,174 km (24,963 mile). The viscosity of the engine oil is summarized in Figure 11. Between oil changes, the viscosity was observed to decrease as the life of the oil increased.

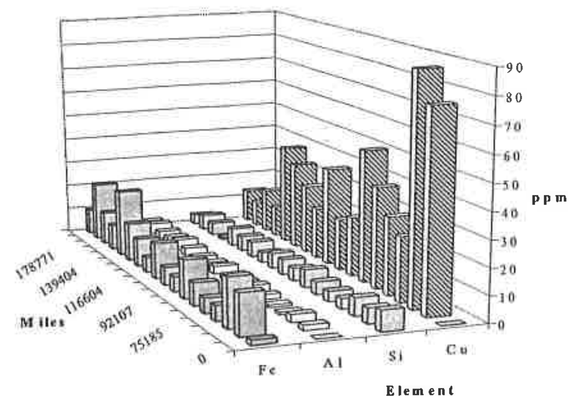



**Figure 10.** Selected contaminants monitored by Chevron LubeWatch™ during the engine oil analyses.

The f-soot level, an indication of carbon residue in the engine oil from the combustion process (9), was reported by Chevron Lube Watch™ for each engine oil sample. A sample of engine oil that had not been run in the engine was reported to have an f-soot value of 0.1. The reported f-soot levels were as low as the blank run with three exceptions of 0.2 as shown in Figure 12.

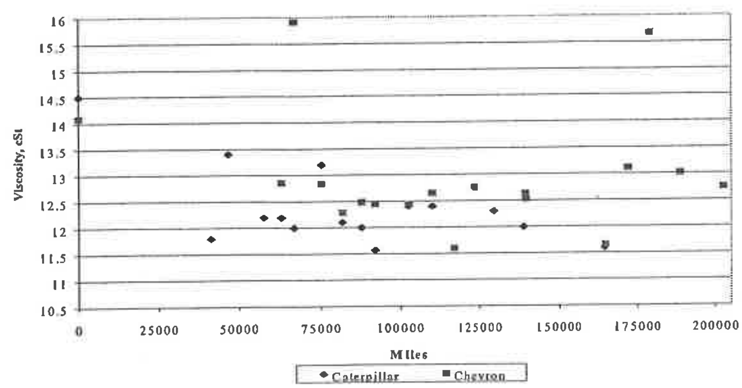



**Figure 11.** Viscosity of the engine oil measured at 100° C by Caterpillar Inc. and Chevron LubeWatch™.

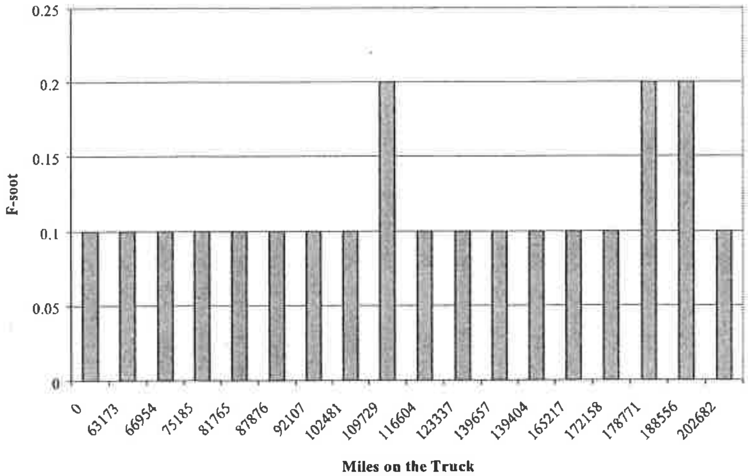



**Figure 12.** F-soot levels measured in the engine oil and reported by Chevron LubeWatch™.

## CATERPILLAR TECHNICAL CENTER ENGINE EVALUATION (ON-ROAD TRUCK ENGINE)

Nearly all aspects of this test passed evaluations with flying colors. The only exceptions were the Transient emissions. While remaining legal by EPA standards a significant increase in particulate emissions was measured. At the same time, a significant reduction in $NO_x$ was measured. Utilizing conventional diesel characteristics, this reduction of $NO_x$ correlates well with an increase in PM emissions. Biodiesel in general and certainly HySEE as well, do not seem to follow traditional diesel methodologies. But in this case the trade-off between the two emissions do correlate.

The changes in engine emissions were attributed to a decrease in injection pressure and a delayed or retarded fuel injection event. This was discovered after the testing was completed. The changes in the emissions data led to an investigation of the fuel system. A detailed inspection and testing at the Caterpillar Fuel System Plant revealed no significant build-up of carbon or other foreign material inside the injectors.

Transient Emissions - NOx emissions decreased 6.4% and 4.8% for 2-D and HySEE fuels respectively. This decrease was not expected and immediately led to further investigation of the engine and fuel delivery system. The emissions results are displayed in Table 4. These magnitudes of reduction are indicative of fuel injection delay or retardation and decreased fuel injection pressure.

Hydrocarbon emissions increased by 15% and 11% for 2-D and HySEE respectively. The unequal increase between the two fuels could be a function of the higher cetane number and the oxygen content in the HySEE fuel. Additional information is needed to verify this hypothesis.

**Table 4. Transient Cycle Emissions (FTP)**

| 3 Hot cycle avg. g/bhp-hr | Pretest/Post-test Fuel: 2-D | Pretest/Post-test Fuel: HySEE |
|---|---|---|
| NOx | 4.51/4.22 | 4.59/4.37 |
| HC | 0.13/0.15 | 0.09/0.10 |
| CO | 1.29/1.35 | 0.94/0.99 |
| $CO_2$ | 540/583 | 536/579 |
| PM | 0.072/0.089 | 0.051/0.056 |

Particulate matter emissions levels showed major differences for similar test conditions. HySEE PM emissions increased approximately half as much as 2-D PM levels. This again shows the major differences in fuel properties. Just as higher cetane number and the oxygen content possibly affected HC emissions, they also could have affected PM in the same manner.

Steady-State Engine Performance – Steady-state full load power with HySEE fuel dropped below normal deviation standards by approximately 1%. Normal deviation is +/-2% of nominal values. This result was not expected but at the same time is not that severe. Power changes of this magnitude, ~3%, are very difficult to detect in chassis but are more pronounced on an engine dynamometer, see Figure 13.

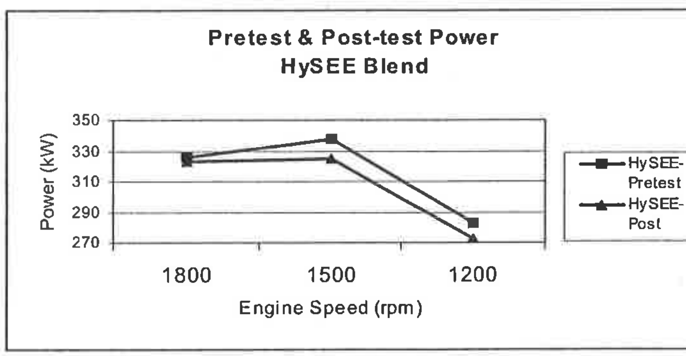



**Figure 13.** Pretest and Post-test full load power, Caterpillar 3406E engine fueled with a 50 % blend of HySEE biodiesel in a Kenworth truck operated for approximately 322,000 km (200,000 miles)

The brake specific fuel consumption (BSFC) increase was directly proportional to the rate of power decrease. This can be explained because the equation for BSFC, which is listed below, has a small value in the denominator.

$$BSFC = \text{Fuel rate (g/hr)} / \text{Brake Power (kW)}$$

BSFC data has been graphed in Figure 14.

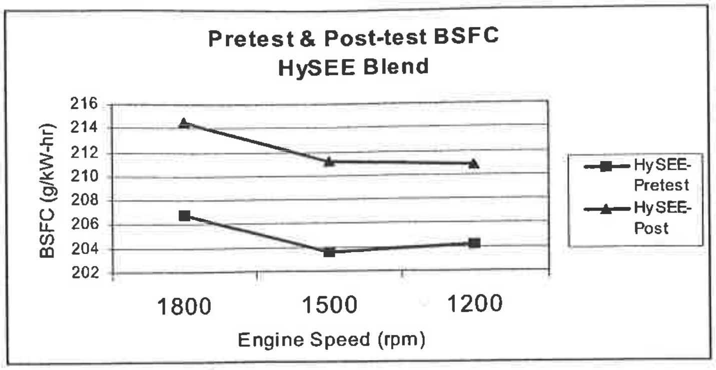



**Figure 14.** Pretest and Post-test BSFC, Caterpillar 3406E engine fueled with a 50% blend of HySEE biodiesel in a Kenworth truck operated for approximately 322,000 km (200,000 miles)

Engine fuel rates decreased slightly at the 1500 and 1200 rpm points. The fuel rate at 1800 rpm engine speed was higher by approximately 2.5%. This was attributed to a difference in the fuel temperature to the engine between the pretest and post-test results. The fuel temperature provided to the engine was higher for the post-test data. The system regulates fuel flow based on fuel density; hence, higher temperatures yield lower density and higher fuel rates. Fuel rates have been displayed in Figure 15.

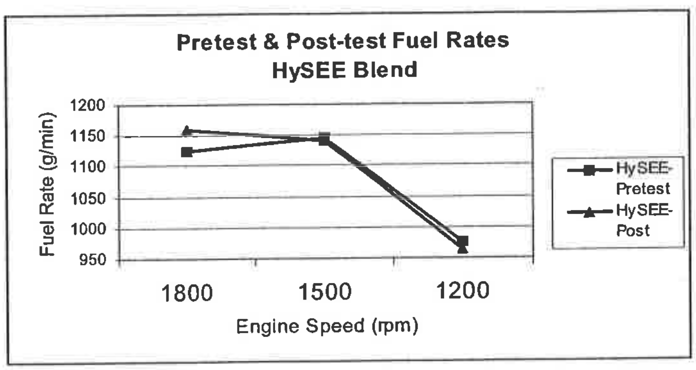



**Figure 15.** Pretest and Post-test fuel rate, Caterpillar 3406E engine fueled with a 50% blend of HySEE biodiesel in a Kenworth truck operated for approximately 322,000 km (200,000 miles.)

The wear measurement data can also be expressed in terms of miles. The life projections for the engine components listed above exceeded 20,000 hours of operation. This would equate to a vehicle mileage exceeding 1,000 miles under similar conditions. These results are equal to those from a diesel-fueled engine. A summary of the piston, ring, and liner (PRL) components has been listed in Table 5.

Valve Train - No signs of unusual wear were found from the inspection of the valve train components from the HySEE fueled engine. No significant differences were observed between this engine and comparable diesel fueled engines.

Projected Engine Life – The data displayed in Table 5 are the actual measurements recorded from the inspection of the engine components from the test engine. The projected life values have been calculated based on the wear trends established from 0 to 200,000 road miles or 0 to 4787 hours of engine operation.

## EPA SECTION 211(B) TEST RESULTS– (ENGINE 2)

Results from this section were measured with the second Caterpillar 3406E engine. Section 211(B) requires a new engine, to be broken-in for 125 hours on the test fuel, which in this case was HySEE biodiesel.

### PERFORMANCE

Table 6 summarizes engine performance with the various test fuels. The data shown represents lug curve performance at rated speed. The values of power and brake specific fuel consumption (BSFC) have been corrected using SAE J1995. The blended fuel was mixed on a volume basis. Engine power for each of the test fuels is shown in Figure 16 and fuel consumption in Figure 17.

**Table 6. Engine 2 Performance Data**

| | Neat HySEE | Neat REE | 50:50 HySEE /2-D | Neat 211(b) Reference 2-D |
|---|---|---|---|---|
| Engine Speed | 1800 | 1800 | 1800 | 1800 |
| Torque (Nm) | 1573 | 1599 | 1624 | 1695 |
| Power(kW) | 298.8 | 304.3 | 304.3 | 322.1 |
| Fuel Rate (g/min) | 1097 | 1102 | 1102 | 1050 |
| BSFC (g/kW-hr) | 220.2 | 217.3 | 217.3 | 195.6 |

### REGULATED EMISSIONS

The data shown in Table 7 represents the composite weighted Cold/Hot emission averages for each test period and test fuel. The test periods were not identical in duration for each of the fuels.

**Table 5. Projected Engine Life from Caterpillar Engine Teardown**

| PRL Component Wear and Life Projections based on 4787 hours (322K km (200K miles) | | | | |
|---|---|---|---|---|
| **Ring Face Wear (mm)** | Wear/1000 - Hours | | Life Projection - Hours | |
| | Average | Maximum | Average | Minimum |
| Top Ring | 0.0062 | 0.0083 | >20,000 | >20,000 |
| Inter Ring | 0.0049 | 0.0060 | >20,000 | >20,000 |
| Oil Ring | 0.0046 | 0.0062 | >20,000 | >20,000 |
| **Cylinder Bore Wear (mm)** | Wear/1000 - Hours | | Life Projection - Hours | |
| TRTA-8 Locations | Average | Maximum | Average | Minimum |
| | 0.004 | 0.0005 | >20,000 | >20,000 |
| **Ring/Groove Side Wear (mm)** | Wear/1000 - Hours | | Life Projection - Hours | |
| | Average | Maximum | Average | Minimum |
| Top | 0.0045 | 0.00055 | >20,000 | >20,000 |
| Intermediate | ------- | -------- | --------- | -------- |
| **Oil Ring Unit Pressure Loss Mpa** | Average | Maximum | Life Projection – Hours | |
| | 0.164 | 0.213 | | |
| % Loss | 9.7 | 11.6 | Acceptable | |
| **Pin Joint Wear (mm)** | Wear/1000 – Hours | | Life Projection – Hours | |
| | Average | Maximum | | |
| Rod Eye – Diametrical (mm) | <0.001 | <0.001 | Acceptable | |
| Pin Wear – mm | <0.001 | <0.001 | Acceptable | |
| Crown Pin Bore | <0.001 | <0.001 | Acceptable | |
| **Over Carbon Measurements** | Average | Maximum | | |
| Ring Projection – Top | -0.106 | -0.064 | Acceptable | |
| Inter | -0.320 | -0.275 | Acceptable | |
| **Ring Side Clearance Loss –** Top | None | | Acceptable | |
| Inter | None | | Acceptable | |
| **Piston Deposit Rating** | Average | Maximum | | |
| Top Groove Carbon | 30.4 | 31.5 | Acceptable | |
| $2^{nd}$ Groove Carbon | 10.6 | 13.5 | Acceptable | |
| Top Land Polished Carbon | 13.5 | 21.0 | Acceptable | |
| WD-1P Rating | 187 | 226 | Acceptable | |
| Note: The cooling galleries and undercrowns had lacquer but no carbon | | | | |

Table 7. Regulated Emissions – Engine 2

| (units: g/bhp-hr) | Neat HySEE | Neat REE | 50:50 HySEE/2-D | Neat 211(b) Reference 2-D |
|---|---|---|---|---|
| NOx | 4.78 | 5.02 | 4.68 | 4.55 |
| THC | 0.026 | 0.03 | 0.061 | 0.12 |
| CO | 0.632 | 0.674 | 0.924 | 1.46 |
| $CO_2$ | 552 | 545 | 551 | 552 |
| TPM | 0.038 | 0.039 | 0.05 | 0.077 |
| Test Duration, Days | 7 | 2 | 2 | 2 |

UNREGULATED EMISSIONS

In both the fuel and the soluble organic fraction (SOF), only three compounds each an ethyl ester, were found: 9-octadecenoic acid (Z), ethyl ester; octadecanoic acid, ethyl ester; and hexadecanoic acid, ethyl ester. The composition of the HySEE fuel and the particulate SOF were indistinguishable. This implies that small amounts of unburned fuel are condensed onto the exhaust particulate forming the SOF. Note that no hydrocarbons associated with lube oil were found on the SOF sample. Therefore, almost 100% of the SOF is unburned fuel.

Average hot Transient total aldehyde and ketone emissions levels among the three fuels, were 28.5, 21.5, and 28.1 milligrams per bhp-hr for 100% HySEE, 505 HySEE/50% Diesel, and Diesel fuels respectively. No statistical significance to the differences could be found.

Average aldehyde and ketone emissions within the dilution tunnel blanks were approximately 1 / 4 to 1 / 3 that of the Transient engine tests. (Mean 8.4, range 3.8 – 15.2 milligrams per bhp-hr.) This variability in the tunnel blanks accounts for the wide range in results from the test fuels and is responsible for the lack of statistical significance.

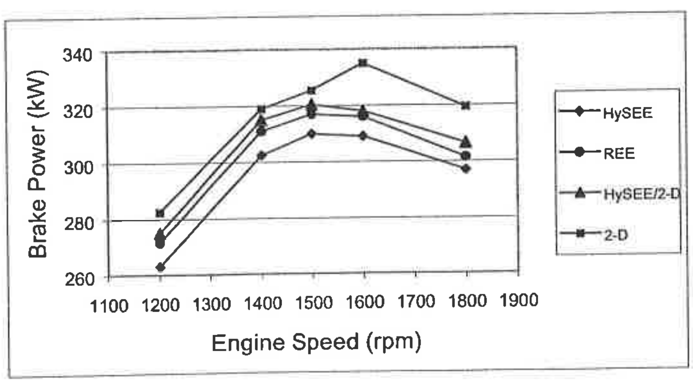



**Figure 16.** Engine Performance-Lug Curve Power – Caterpillar 3406 E, Section 211(b) Engine.

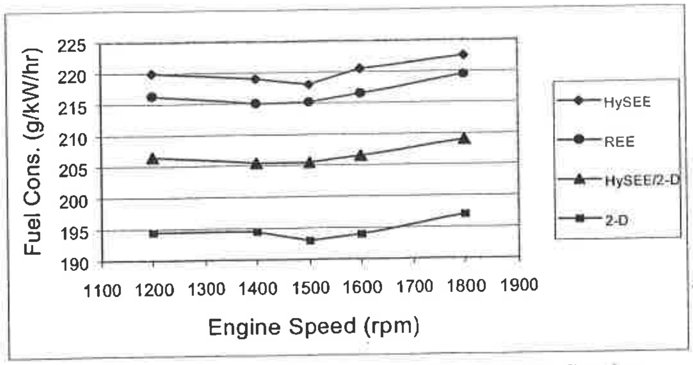



**Figure 17.** Fuel Consumption – Caterpillar 3406E, Section 211(b) Engine.

Formaldehyde and acetaldehyde make up the bulk of the total aldehyde and ketone emissions, account for roughly 75% of the total. During aldehyde and ketone testing, none were found with greater than four carbon atoms in size. Ethyl acrylate was emitted at concentrations averaging 2160 micrograms per bhp-hour during the hot Transient cycles while on 100% HySEE fuel.

Tunnel blanks could explain the presence of many of the hydrocarbons found during hydrocarbon speciation, particularly with the biodiesel fuels. The hydrocarbon speciation method used in this project (which was optimized for lower molecular weight compounds) had an approximate analysis range of C2 to C12. Adjusting several thermal desorption parameters to high temperatures can raise the speciation limit to at least C20. The particulate SOF, from 100% HySEE operation, is almost 100% unburned fuel.

**PAH AND NITRO-PAH ANALYSIS**

PARTICULATE MATTER - PAH

For the HySEE fuel, the emissions rates for PAHs from pyrene to benzo(g,h)perylene are lower than the diesel emissions. For example, the emission rates for HySEE benzo(a)pyrene and 0.03 and 0.04 μg PAH/bhp-hr for the cold and hot cycles, respectively, while the emission rates for diesel are 0.19 and 0.07 μg PAH/bhp-hr for the cold and hot cycles, respectively. The hot start 50:50 fuel PAH emissions are generally intermediate to the HySEE and diesel fuels. For example, for hot start pyrene emissions, the average rates (+S.D.) are 1.11 (+.22), 2.28 (+.15), and 5.74 (+.18) μg/bhp-hr for the HySEE, 50:50, and diesel fuels, respectively. This gradient of increasing emissions with the diesel fuel is seen for almost all the PAHs with molecular weights equal to or greater than pyrene. A comparison of the emission rates for HySEE and diesel for targeted PAHs are illustrated in Figure 18. The HySEE fuel emissions for chrysene + benz(a)anthracene, benzo(b)fluoranthene + benzo(k)fluoranthene, benzo(a)pyrene, indeno(1,2,3-cd)pyrene, and benzo(ghi)perylene are typically lower than the diesel emissions.

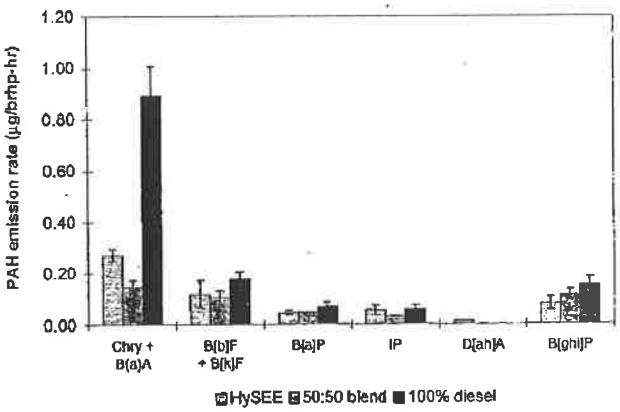



**Figure 18.** Emission rates for targeted particle-phase PAHs for the HySEE 50:50 blended, and 100% diesel fuels. Average emission rates (± S.D.). These PAHs are predominantly found on particulate matter. Chry = chrysene, B(a)A = benz(a)anthracene, B(b)F and B(k)F = benzo(a) and benzo(k)fluoranthene, B(a)P = benzo(a)pyrene, IP = Indeno (c,d)pyrene, D(a,h)A = dibenzo(a,h)anthracene, B(ghi)P = benzo (g,h,i)perylene.

VAPOR-PHASE (PUF) SAMPLES – PAH

For both the cold and hot start cycles for all PAHs through acenaphthylene, the HySEE fuel was lower relative to the diesel fuel. This trend of lower emissions of PAHs is seen for fluorene, 1-methyl phenanthrene, phenanthrene, and anthracene. The hot start emissions of fluorene, 1-methyl phenanthrene, phenanthrene, are approximately 25%, 205 and 505, respectively, of the diesel emission for these compounds. Again, as in the case with the particle samples, the 50:50 blend is intermediate between the HySEE and diesel fuel emission rates. For example, the hot start cycle emissions for phenanthrene were 29.08, 47.40, and 76.43 µg/bhp-hr for the HySEE 50:50 blend, and diesel fuels, respectively. A marked difference between the fuels is seen during the cold start cycle for the HySEE in comparison to the 50:50 and diesel emissions. While the cold start cycle is typically many fold higher for the PAHs from napthalene through 2,3,5-trimethyl-naphthalene, for the HySEE cold start cycle, the emissions of these PAHs appear to be slightly higher or similar to the hot start cycle. The cold start cycle of the HySEE fuel as a percentage of the hot start cycle has less of an increase compared to the 50:50 or diesel fuels.

Particle Phase Plus Vapor Phase

The emission rates as a sum of both the PUF and filter sample emissions are illustrated in figure III-3. The HySEE sample is dramatically lower than either the diesel or 50:50 blends. For almost all the PAHs starting from fluorene, there is increasing gradient of emissions seen from the HySEE to the diesel emissions.

Collection of the more volatile PAHs requires PUF sampling placed in series behind the filter sample. Some of the PAHs however, may not be captured on a single PUF and could be lost. To investigate this, a backup PUF was placed behind the primary PUF sample. If the primary PUF could not hold the entire sample, the PAHs not captured on the primary PUF would "breakthrough" and get trapped on the second or backup PUF. If both PUFs have equivalent amounts of PAHs, then the second PUF could not hold the compounds. There was appreciable breakthrough of compounds from naphthalene through acenaphthylene. Since the levels of most of these compounds appear similar in mass in both PUF, it may be that equilibrium is obtained during the sampling, but that the entire emission is not collected. Therefore, as an approximation, relative values between fuels can be used for comparison. If these compounds are to be quantitatively captured, other adsorbents need to be considered in any future study.

NITRO-PAHS

The results for the analyses of selected nitro-PAHs in the filter and PUF samples combined are summarized in Table 8. Of the targeted nitro-PAHs, the 2-nitrofluorene and 9-nitroanthracene are semi-volatile and therefore would be expected if present, to be in the vapor and particle phases. The other nitro-PAH 3-nitrofluoranthene, 1-nitropyrene are predominantly expected to be found in the particle phase. For the nitro-PAHs analyzed in the HySEE fuel emissions, 1-nitropyrene was the only compound significantly detected and quantitated. The 9-nitroanthracene was near the limit of detection, but was present in the HySEE sample. The emission rate for 1-nitropyrene was 0.12 µg/bhp-hr for the HySEE fuel and 0.06 µg/bhp-hr for 9-nitroanthracene. For the diesel sample, there were significant levels of 1-nitropyrene and 9-nitroanthracene. The emission rate for 1-nitropyrene in the diesel samples was 0.34 µg/bhp-hr and for 9-nitroanthracent was 0.27 µg/bhp-hr. The contribution of the tunnel blanks needs further investigation since the amounts of sample were limited.

**Table 8.** Total Nitro-PAH Emissions (µg/bhp-hr)*

| Compound | HySEE Total (µg/bhp-hr) | Diesel Total (µg/bhp-hr) |
|---|---|---|
| 2-nitrofluorence | <0.11 | <0.11 |
| 9-nitroanthracene | 0.06 | 0.27 |
| 3-nitrofluoranthene | <0.11 | <0.11 |
| 1-nitropyrene | 0.12 | 0.34 |
| 7-nitrobenz(a)anthracene | <0.11 | <0.11 |
| 6-nitrochrysene | <0.09 | <0.09 |
| 6-nitrobenzo(a)pyrene | <0.07 | <0.07 |
| Sum Nitro-PAH (µg/bhp-hr) | 0.18 | 0.60 |

*Emission rates are from the average of 2 particle samples, with each sample a series of 3 hot start cycles and a single three-cycle PUF sample.

## BIOASSAY ANALYSES OF THE PARTICULATE AND VAPOR-PHASE HYSEE AND DIESEL EMISSION SAMPLES

### PARTICULATE PHASE

Bioassays were conducted to determine the specific mutagenic activity of the HySEE, 50:50 blend and diesel particle extracts. Specific mutagenic activity refers to the number of revertants per microgram of PM extracted or per µl of extract solution. It is determined from the slope obtained from the linear portion of the dose-response curve. All filter extracts were then tested for mutagenicity using the microsuspension bioassay procedure with tester strain TA98, with and without the addition of metabolic enzymes.

All dose response curves were linear through the highest doses tested. Typically, mutagenic activity was higher when no metabolic enzymes are added (-S9) to the test system. The average (+ S9) specific mutagenic activity with S9 for the particles collected from the emissions from HySEE, 50:50, and Diesel are 1.35, 3.16 and 8.23 (for hot cycles), respectively. The potency of the diesel particulate matter here is approximately 6 times the potency of the HySEE particulate matter, Figure 19.

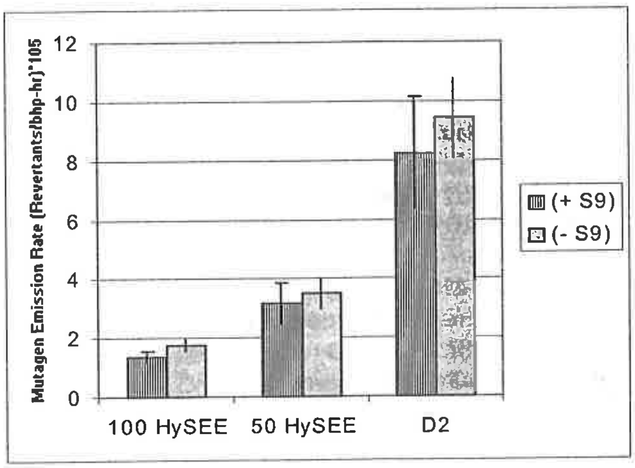



**Figure 19.** Mutagen Emissions from Particulate Matter from Combustion of Each Fuel. +S9 and –S9 are with and without metabolic enzymes.

### VAPOR PHASE

PUF samples were extracted using supercritical carbon dioxide and tested in the microsuspension assay. The tester strain TA100 was incorporated since activity was detected in the vapor-phase of diesel exhaust reported previously (Kado, 1996). The specific mutagenic activity of the HySEE fuel is about 25-45% lower than the 2-D fuel emissions with TA98, and about 60% lower with tester strain TA100. The emissions of mutagenic compounds are about 2 fold lower for the HySEE fuel emissions than the 2-D emissions when both tester strains are considered. The 50:50 blend rates were usually intermediate between the HySEE and 2-D.

## SUMMARY AND CONCLUSIONS

1. Fuel Production - A scaled-up production scheme produced 80 152 liters (21,174 gal) of HySEE biodiesel from used French frying oil feedstock and ethanol. The HySEE delivered to the test vehicle was within the fuel specifications developed by Caterpillar Inc. Total glycerol averaged 0.402% and yield of ester as function of delivered weight of oil averaged 85.4%.

2. Fuel Delivery - A blending valve connected to the diesel and HySEE nurse tanks provided on-demand fueling for the over-the-road truck. The blending valve delivered fuel averaging 51.3% HySEE and 48.7% diesel. Fueling the truck required no additional operator to monitor the blending of the fuel. The calibration of the fuel meter was accurate to within 0.87% of the volume registered on the fuel meter.

3. The estimated cost to produce the HySEE biodiesel was $2.08 per gallon for materials alone. No profit, capital costs, labor, or taxes are included.

4. Test Vehicle Performance -The arctic package installed on the truck along with parking the test vehicle inside when not in use allowed the truck to operate normally in all weather conditions. The truck consumed 70 397 liters (18,597 gal) of HySEE and a total of 145 746 liters (38,502 gal) of blended fuel during its 326,235 km (202,713 mile) of operation. The corrected road horsepower of the vehicle began and ended within the specified power tolerances. Injector cutout readings showed the corrected vehicle power was within 75 to 80% of the power with all injectors working. The injector cutout tests showed that all injectors performed normally. No accelerated engine degradation was detected according to engine oil analyses.

5. Transient Emissions – (On-Road Truck Engine) – There was a significant reduction in NOx emissions for both diesel and HySEE fuels over the course of the on-road test. The NOx reduction is not typical for this engine and was caused by a decrease in fuel injection pressure and retarding of the fuel injection timing. These changes were not attributed to the biodiesel fuel

   Engine hydrocarbon emissions from petroleum diesel fuel increased over the course of the on-road test greater than HC emissions with biodiesel fuel. The overall increase of HC with both fuels is attributed to the lowering of fuel injection pressure. However, the unequal increase of HC between the two fuels is attributed to differences in fuel composition.

   Carbon Monoxide engine emissions increased during the on-road tests with both fuels. The increase is consistent with lower fuel injection pressure.

An atypical increase in particulate emissions over the course of the on-road test was observed with both fuels. There was also an unequal increase between the two fuels that was greatly exaggerated by the differences in fuel properties.

6. Steady-State Engine Performance – Steady-state engine power decreased a small amount outside of normal deviation limits. Specific fuel consumption increased proportionally to the decrease in engine power. The increase was slightly above normal limits. Engine fuel rate was within normal deviation tolerance.

7. Projected Engine Life – Service life of the piston, rings, and liner is expected to exceed 20,000 hours or 1.6 million km (one million miles). No cracks were found in the piston crowns, piston skirts, connecting rods, wrist pins, or cylinder liners. The wrist pin joint wear was acceptable and parts are in excellent condition. Oil control and blowby were not measured but assumed acceptable based on piston deposits. Post-test over carbon measurements were acceptable. Piston deposit rating was acceptable. Valve lash measurements showed no indication of intake or exhaust valve recession. Valve condition was acceptable, showing no deposit, guide wear, or tip wear concerns. Light valve face wear was observed on the inlet manifold side cylinder #1 intake, but wear was not unusual for an engine with this mileage. Therefore, the wear was not a concern. Valve bridges showed no wear concerns. The camshaft and roller followers were in good condition. The rocker arm shafts and shaft bearings were acceptable. No rocker arm button to bridge interface wear was observed.

8. Regulated and Non-regulated Emissions (section 211(b) engine) – Observed engine operation with biodiesel fuels was very good. There were no obvious ill mannered engine characteristics observed while combusting biodiesel. Throughout the entire test program the engine ran flawlessly. The engine accumulated approximately 220 operational hours while consuming HySEE biodiesel. There were no signs of fuel injector component degradation after 125-hour break-in on HySEE. This was the first experience gained with HySEE in a high-pressure fuel injection system (138 MPa (20,000 psi or above)).

   Results obtained with neat HySEE and 50:50 HySEE/2-D fuels show considerably reduced particulate, CO and THC emissions over neat 2-D. NOx emissions were higher with the biodiesel fuels than with the neat 2-D.

   Blending HySEE fuel into 2-D at a ratio of 50:50 by volume caused measured TPM to drop at a rate disproportionately above that suggested by blend ratio alone. A very minor increase in NOx accompanied the TPM reduction.

   The SOF results variability mirrors the THC and TPM results variability for the HySEE fuel. Cold run %SOF levels were below hot run averages. There was no significant difference between cold and hot run gravimetric TPM results. Average BG-1 %SOF results were moderately below SDT values due likely to higher filter flow rates and filter pressure differential.

9. Based on background TPM data, the influence of the dilution tunnel operating history on measured emissions is very high. EPA's requirement that only "new" engines (not rebuilt or remanufactured, etc.) can be used for biodiesel emissions evaluation and bioassay testing is likely inappropriate in the absence of thorough cleaning and conditioning of the dilution system. This requirement should be more fully studied.

10. Aldehyde and ketone emissions with HySEE and blended HySEE fuels are no different than when diesel fuel is used. At the low emission levels of the tested engine, dilution tunnel blanks, and their variability, may have accounted for a large proportion of the aldehyde emissions; perhaps up to 1/3 of the total. Ethyl acrylate is formed from the partial combustion of HySEE fuels.

11. For the particle phase, the PAH emissions for the HySEE fuel and for PAHs such as pyrene, benz(a)anthracene, chrysene, the emission rate was considerably lower than the emission rate for diesel.

12. The emission rate for the vapor-phase PAHs from the HySEE fuel was lower than the 50:50 blend and 100% diesel fuel samples. For example, the rates for phenanthrene were about 29.08, 47.4 and 76.3 µg/bhp-hr for the HySEE, 50:50 blend, and diesel fuel hot start cycles, respectively.

13. In the Nitro-PAHs analysis only 9-nitroanthracene and 1-nitropyrene were detected and could be quantitated. The emission rate for 100% 2-D fuel for these compounds was approximately 5 times higher than HySEE for the first and approximately 3 times higher than HySEE for the second.

14. Bioassay analyses were conducted on both the particle and vapor-phase samples and both were found to be mutagenic. The 100% 2-D samples were the most potent having specific mutagenic activities at least two times the activity of the HySEE sample. The HySEE mutagenicity emission rates were approximately 6 times lower than the diesel emission rates when metabolic enzymes were added and approximately 5 times lower without the metabolic enzymes. The mutagenic activity of the 50:50 blend was intermediate between the HySEE biodiesel and 2-D.

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