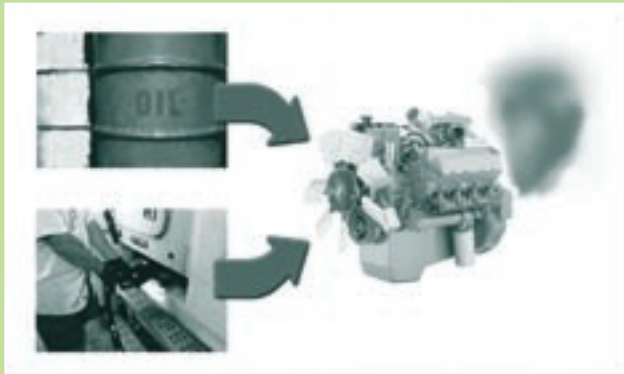


# Advanced Petroleum-Based Fuels— Diesel Emissions Control Project (APBF-DEC)

## Lubricants Project, Phase 1 Summary

July 2004



U.S. Department of Energy

**Energy Efficiency  
and Renewable Energy**

Bringing you a prosperous future where energy  
is clean, abundant, reliable, and affordable

A government/industry partnership studying  
the effects of fuel & lubricant compositions  
in engine systems to reduce diesel emissions

*APBF-DEC Lubricants Project,  
Phase 1 Summary*

## **APBF-DEC Reports**

Advanced Petroleum-Based Fuels—Diesel Emissions Control Project (APBF- DEC)  
Lubricants Project, Phase 1 Summary  
July 2004

This is a summary of the Lubricants Project, Phase 1 for the APBF-DEC project. Other information about APBF-DEC is available on the World Wide Web at [www.nrel.gov/vehiclesandfuels/apbf.html](http://www.nrel.gov/vehiclesandfuels/apbf.html).

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## **The Advanced Petroleum Based Fuels—Diesel Emission Control Project is supported by:**

- U.S. Department of Energy's FreedomCAR Program
- American Chemistry Council
- American Petroleum Institute
- California Air Resources Board
- Engine Manufacturers Association
- Manufacturers of Emission Controls Association
- South Coast Air Quality Management District

## Acronyms

<b>ACC</b>	American Chemistry Council (ACC)
<b>APBF-DEC</b>	Advanced Petroleum-Based Fuels—Diesel Emissions Control project
<b>API</b>	American Petroleum Institute
<b>bhp-hr</b>	brake horsepower hour
<b>Ca</b>	calcium
<b>CARB</b>	California Air Resources Board
<b>CCV</b>	closed crankcase ventilation
<b>CO</b>	carbon monoxide
<b>CO<sub>2</sub></b>	carbon dioxide
<b>DECSE</b>	Diesel Emission Control-Sulfur Effects Program
<b>DOE</b>	U.S. Department of Energy
<b>DPF</b>	diesel particle filter
<b>EGR</b>	exhaust gas recirculation
<b>EMA</b>	Engine Manufacturers Association
<b>EPA</b>	U.S. Environmental Protection Agency
<b>FEV</b>	FEV Engine Technology
<b>HC</b>	hydrocarbon(s)
<b>MECA</b>	Manufacturers of Emission Controls Association
<b>Mg</b>	magnesium
<b>Mo</b>	molybdenum
<b>NO</b>	nitric oxide
<b>NO<sub>2</sub></b>	nitrogen dioxide
<b>NO<sub>3</sub></b>	nitrate
<b>NO<sub>x</sub></b>	nitrogen oxide
<b>N<sub>2</sub>O</b>	nitrous oxide
<b>NPRA</b>	National Petrochemical and Refiners Association
<b>NREL</b>	National Renewable Energy Laboratory
<b>ORNL</b>	Oak Ridge National Laboratory
<b>P</b>	phosphorus
<b>PM</b>	particulate matter
<b>ppm</b>	parts per million
<b>S</b>	Sulfur
<b>SCAQMD</b>	South Coast Air Quality Management District
<b>SCR</b>	Selective catalytic reduction
<b>SO<sub>2</sub></b>	sulfur dioxide
<b>SO<sub>3</sub></b>	sulfite
<b>SO<sub>4</sub></b>	sulfate
<b>SOF</b>	soluble organic fraction
<b>SUV</b>	sport utility vehicle
<b>SwRI</b>	Southwest Research Institute
<b>THC</b>	total hydrocarbons
<b>TPM</b>	total particulate matter
<b>ZDDP</b>	zinc dialkyl-dithiophosphate

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## Advanced Petroleum Based Fuels— Diesel Emissions Control (APBF-DEC)

### Introduction

The Advanced Petroleum-Based Fuels—Diesel Emissions Control (APBF-DEC) project is a government/industry collaborative project to identify the optimal combinations of low-sulfur diesel fuels, lubricants, diesel engines, and emission control systems to meet projected emission standards for the 2004 to 2010 time period. Properties of fuels and vehicle systems could lead to even lower emissions beyond 2010. APBF-DEC consists of five projects that use a systems approach to enhance the collective knowledge base about engines, diesel fuels, lubricants, and emission control technologies. The five test projects are evaluating:

- Selective catalytic reduction/diesel particle filter (SCR/DPF) technologies
- Nitrogen oxide (NO<sub>x</sub>) adsorber catalyst/DPF technologies for passenger cars, light-duty trucks/sport-utility vehicles (SUVs), and heavy-duty applications (three different engine platforms)
- Lubricant formulations that may affect the performance and durability of advanced diesel emission control systems.

This summary describes the results of the first phase of the lubricants study—Phase 1—investigating the impact of lubricant formulation on engine-out emissions. Figure 1 pictures the two-phase approach of the lubricants project.

The APBF-DEC project is being sponsored and conducted by a broad collaboration of government and industry organizations including: the U.S. Department of Energy (DOE), the American Chemistry Council (ACC), the American Petroleum Institute (API), the Engine Manufacturers Association (EMA), the Manufacturers of Emission Controls Association (MECA), the California Air Resources Board (CARB), and the South Coast Air Quality Management District (SCAQMD). Providing technical assistance and support are the U.S. Environmental Protection Agency (EPA) and the National Petrochemical and Refiners Association (NPRA). Representatives from these and other agencies, associations, national laboratories, and private sector companies serve on the 20-member APBF-DEC Steering Committee and its working groups.

A systems approach is being used to simultaneously investigate how fuels, lubricants, engines, and emission control systems can provide clean and efficient transportation systems. APBF-DEC's five separate projects are evaluating how sulfur (S) and other compounds affect the performance and durability of advanced diesel emission control systems. The projects are summarized in Table 1. Other reports on diesel engines and emission control systems will be prepared as studies are completed.

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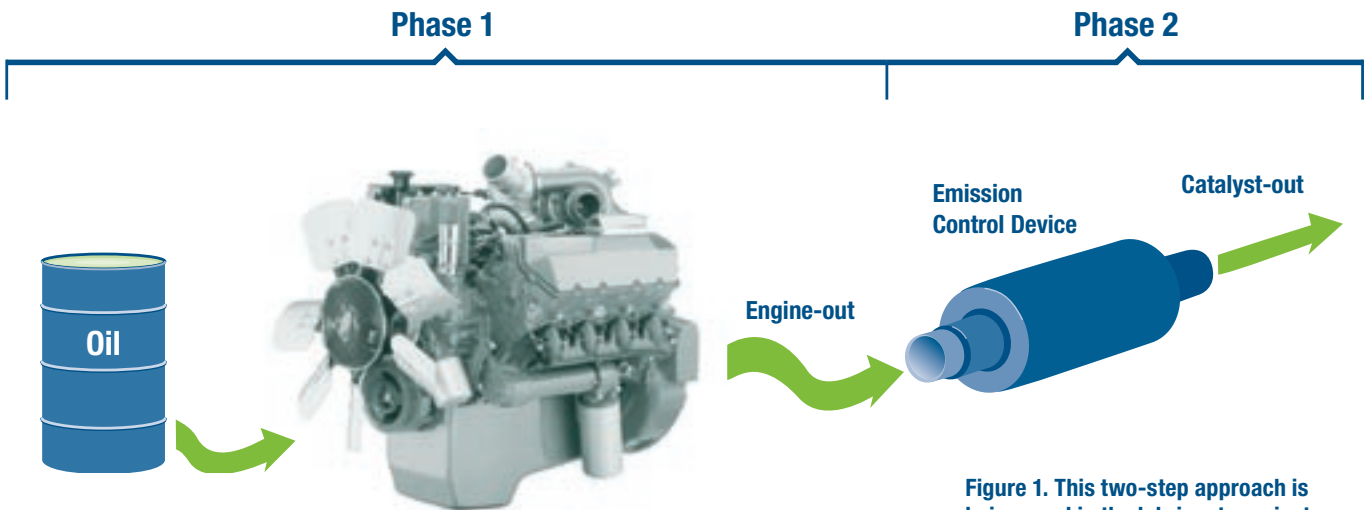


Figure 1. This two-step approach is being used in the lubricants project.

**Table 1: APBF-DEC; Five Test Projects**

Technology	Platform	Test Vehicle/Engine	Test Laboratory
NO <sub>x</sub> Adsorber Catalysts and Diesel Particle Filters	Light-duty passenger car	1.9L TDI Audi A4I	FEV Technology, Inc.
	Medium-duty truck	Chevrolet Silverado Isuzu/GM Duramax	Southwest Research Institute
	Heavy-duty engine	15L Cummins ISX DOHC engine	Ricardo, Inc.
Urea Selective Catalytic Reduction and Diesel Particle Filters	Heavy-duty engine	Caterpillar C12 engine	Southwest Research Institute
Lubricants	Medium-duty engine	International T444E Cummins ISB	Automotive Testing Laboratory (Phase 1) AEI (Phase 2)

## Background

New emission regulations for light- and heavy-duty engines that will be phased in later this decade will necessitate the use of advanced emission control technologies, including catalysts and filters. Some of the new technologies in development have demonstrated their sensitivity to fuel-borne sulfur, and regulations limiting the permissible levels of sulfur in diesel fuel have been put in place to enable their use. However, the sensitivity of these devices is extreme, and the durability requirements of heavy-duty commercial vehicles are very demanding. So a reduced fuel sulfur level may not be enough to guarantee the long-term performance of new emission control systems if other sources of catalyst poisons are found to exist.

Lubricants from diesel engines are known to be consumed in small, but not insignificant, quantities during the normal operation of the engine. While the quantities may be small, the sulfur content in engine oil is typically higher than the sulfur in fuel by an order of magnitude or more, which elevates the level of concern. Other constituents in the lubricant oil, such as wear control additives, have been found to be an issue for three-way catalysts used with gasoline engines and may cause similar problems in diesel emission control systems. These other constituents are expected to cause similar problems for diesel exhaust systems.

For these reasons, a separate project was planned within APBF-DEC to look specifically at the lubricant's effects on the durability of catalysts and other emissions effects. The results from the APBF-DEC lubricants study will provide valuable information to the developers of lubricants performance standards applicable to future engines.

Industry leaders, engine makers, and the oil and additive industries are actively developing a new category of diesel lubricants and a new specification for lubricating oil to be used in catalyst-equipped diesel engines. This specification, Proposed Category 10 (PC-10), is scheduled to be adopted by 2006 and may trigger the most important changes in oil formulation in many years. However, because of the limited experience with these new emission control technologies, little data currently exist to justify these new standards. Limits on sulfur and phosphorus, or the additives that contain them, could have a significant impact on the performance of the lubricant by compromising engine durability and oil drain intervals, both of which have a significant impact on the vehicle owner's economic bottom line. It is important that the effects lubricants have on emissions be well quantified and evaluated, so the appropriate lubricants can be developed to protect the emission control systems while continuing to provide superior engine protection.

## Project Overview

The objective of this project is to evaluate the effects of lubricant formulations on emissions from a multi-cylinder engine without a catalyst. This summary describes the results of Phase 1 of a two-phase project. The objectives of Phase 1 were to investigate the effects of lubricant additives and lubricant basestocks on engine-out emissions, and to understand how the rate and mechanism (combustion versus blow-by) of oil consumption might affect the relationship between oil formulation and oil-derived emissions. This phase also was to determine whether an appropriate method for accelerating oil consumption rates could be developed for use in a rapid catalyst aging protocol. All tests were conducted with 0.6 parts per million (ppm) sulfur (S) base fuel.

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Strategies were developed to study the impact of lubricant formulations on the performance and short-term durability of the emission control devices. Statistically significant differences in engine-out emissions attributed to lubricant formulations were also studied. Questions were devised to guide Phase 1's testing, data gathering, and data analyses. Several examples are listed in the box below.

Phase 1 also gathered information for Phase 2, which will study how these lubricant-derived emission species affect the performance of the diesel emission control system. It is believed that the effects, when present, are cumulative and may require long run times to reveal themselves. Therefore, accelerating aging protocols would allow for more tests in fewer hours and with less cost. The follow-on phase will, to a great extent, rely on the results of this initial phase and will include extended-duration engine tests with catalysts.

## Methods

Controlled laboratory tests, chemical and physical measurements, and statistical modeling were used to achieve the objectives of this project. Phase 1 of the lubricants project's mission involved the selection of the engine and test hardware, test fluids (fuels and lubricants), emissions measurements, and test matrices. The 1999 International 7.3L T444E-HT engine used for the test met the EPA emission standards for on-highway certification. Additional retrofit hardware was installed on the engine to allow cooled exhaust gas recirculation (EGR) and closed crank-

- ▶ Are there significant differences in engine-out emissions that can be attributed to oil properties?
- ▶ How much of an impact is attributed to the properties of the additive packages and how much is attributed to the base oil?
- ▶ Can the emissions of selected species (specifically metals) be predicted from the properties of the test oils and fuel?
- ▶ Can other indirect relationships between oil properties and engine-out emissions be identified?
- ▶ How do emissions change as a function of oil consumption rate for each oil type and acceleration method?
- ▶ How does oil type affect these changes?
- ▶ How does the oil consumption method (combustions, blow-by, or combination) affect these changes?
- ▶ Can the combined effects of these methods be predicted from the estimated effects of each method, that is, are there interactions between the acceleration methods?

case ventilation (CCV). Such systems are expected to be commonplace on engines meeting future EPA regulations.

The lubricants tested included a variety of additive packages and basestocks representative of modern commercial products as well as experimental products. A statistical approach was employed to select 13 test packages that would adequately span the range of properties of interest while meeting resource constraints. Base oils were selected from each of the four major base oil categories as defined by the API. They span the commercially available offerings in terms of sulfur content, saturation, viscosity index, and volatility. All test oils used the same viscosity index improver that was dissolved in a light fraction of the base oil. All tests were conducted with the ultra-low sulfur base fuel developed previously for the Diesel Emissions Control-Sulfur Effects (DECSE) project, the predecessor to APBF-DEC.

Particulate matter (PM), NO<sub>x</sub>, sulfur dioxide (SO<sub>2</sub>), hydrocarbons (HC), carbon monoxide (CO), and carbon dioxide (CO<sub>2</sub>) emissions were measured during four steady-state

test modes. The test matrices developed ensured that there would be sufficient high-quality data to meet statistical requirements for addressing the study questions without exceeding resource constraints. Mass balance analysis was used to predict system outputs (particulate and gaseous emissions) from system inputs (e.g., sulfur from fuel and oil consumption). The fuel and lubricants were sampled and their properties were analyzed throughout the testing process.

Figure 2 below illustrates the system used to accelerate oil consumption during Phase 1. Precisely measured amounts of lubricating oil were either blended directly into the diesel fuel supply (doping), so the oil was then burned in the engine along with the fuel, or the oil was injected under pressure into the exhaust manifold to simulate the blow-by of oil from the engine reservoir directly to the exhaust. In a third type of accelerated oil consumption test, both fuel-oil blending and exhaust manifold oil injection were used at the same time.

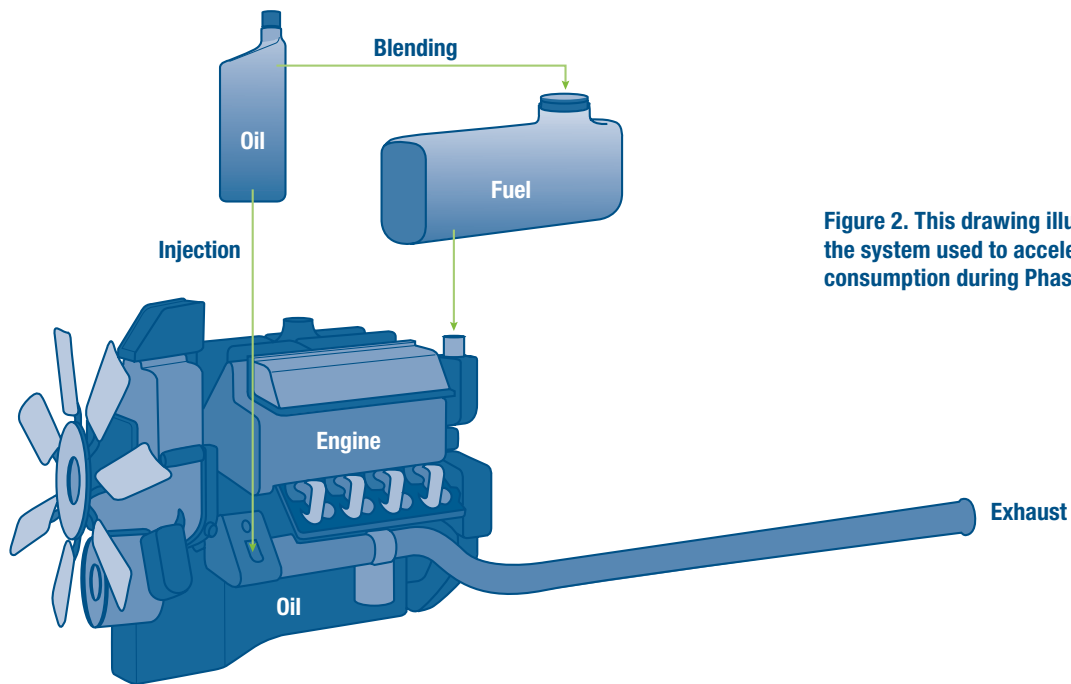
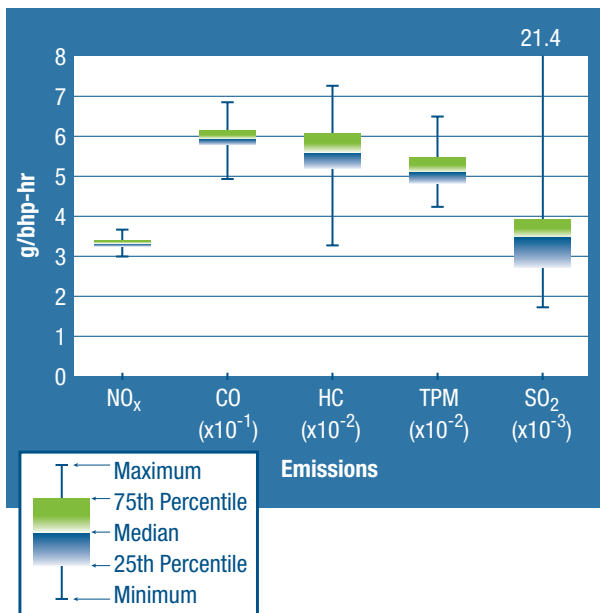
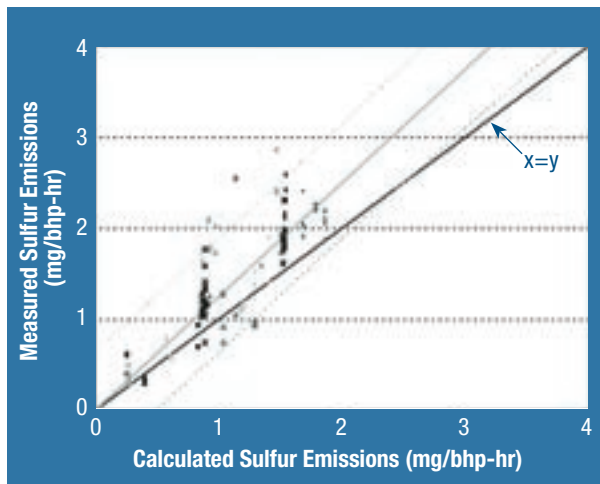


Figure 2. This drawing illustrates the system used to accelerate oil consumption during Phase 1.



**Figure 3.** Shown are the ranges and selected percentiles of mass emission rates of gaseous and PM emissions across all oil types tested.



**Figure 4.** This is a comparison of predicted and actual emissions of total sulfur (S), with estimated recovery regression line and 95% confidence interval.

## Findings and Conclusions

The significant findings and conclusions from Phase 1 are summarized below. The complete report containing results from Phase 1 of the lubricants project will be available on the U.S. Department of Energy Web site at <http://www.eere.energy.gov>.

- ▶ The oil formulation had statistically significant effects on nearly every emissions component. Figure 3 shows the range and selected percentiles of gaseous and total particulate matter (TPM) emissions that were observed from the oils tested.
- ▶ Both additives and base oils were found to affect emission levels. However, the effects of additives are not the same for every base oil. NO<sub>x</sub>, CO, HC, and TPM emissions vary by 10% to 20% across the oils tested, while SO<sub>2</sub> emissions vary by an order of magnitude.
- ▶ The emissions from lubricant-derived metals (S, calcium [Ca], zinc [Zn], phosphorus [P], and magnesium [Mg]) were highly correlated with emissions predicted from the composition of oil (and sulfur in the fuel). However, recovery rates varied considerably (ranging from 27% for Mg to 127% for S), and certain oils deviated significantly. Figure 4 is a comparison of predicted and actual emissions of total sulfur emissions. Oil i2 yielded sulfur emissions eight to 10 times higher than predicted by the mass balance. The mass balance analysis assumes that the composition of the consumed lube oil is the same as the lube oil in the crankcase (as determined by oil analysis). However, the variations in recovery rates suggest that this is probably not the case. The recovery rate indicates the actual composition of the consumed lube oil. Several factors ultimately determine the fate of a given species, including volatility, surface activity, and tendency to break down at elevated temperatures. Certain metals, such as calcium, may tend to collect in the lubricant filter, whereas others, such as magnesium, may tend to be retained in the oil due to its higher volatility.
- ▶ Although the sulfur content of the oil is the primary predictor of SO<sub>2</sub> emissions (59% correlation), the results demonstrate that oils containing higher levels of Zn and molybdenum (Mo) produce lower levels of SO<sub>2</sub> emissions. Adding Zn and Mo to the SO<sub>2</sub> prediction model



increases the correlation from 59% to 74%. This finding offers additional evidence that the source of sulfur in the lubricant has a greater impact on the resultant emissions of SO<sub>2</sub> than does the total sulfur content. The oils with higher zinc content have a larger portion of the sulfur coming from zinc dialkyl-dithiophosphate (ZDDP) relative to other possible sulfur sources (detergents, base oil, etc.). This would imply that the sulfur coming from the ZDDP is not as prone to producing SO<sub>2</sub> in the exhaust and may not be as detrimental to the emission control systems, compared with the sulfur coming from other sources. This also suggests that chemical limits may not be generally appropriate for lubricant specifications aimed at prolonging the life of an emission control system.

- ▶ The method of accelerating oil consumption can have a dramatic effect on gaseous and PM emissions. Emissions of HC, CO, and PM increase by 175%, 15%, and 40%, respectively, when oil consumption is doubled as it is injected into the exhaust stream. However, if oil consumption is doubled by blending oil with the fuel prior to injecting it into the engine, the impact on HC, CO, and PM emissions is negligible. NO<sub>x</sub> emissions are not significantly affected by accelerated oil consumption, regardless of the acceleration method. Oil composition has minimal impact on the changes in HC, CO, NO<sub>x</sub>, and PM emissions when oil consumption is accelerated. The relative increase in SO<sub>2</sub> emissions when oil consumption is doubled depends on the composition of the oil (increases range from 1% to 55%), but is relatively independent of the method of acceleration (Figure 6).
- ▶ The relative recoveries of targeted metals (S, Ca, P, and Zn) under accelerated oil consumption are affected by the acceleration method as well as oil composition. Relative recoveries (recovery of elements from “added” oil) ranged from 15% to 85%, while baseline recoveries (recovery under normal oil consumption) ranged from 30% to over 1,000%, depending on the oil used. Relative recoveries of Ca, P, and Zn are generally higher when oil is blended with the fuel (45% to 70%) compared to when oil is injected in the exhaust (15% to 35%). The relative recovery rates for S range from 25% to 85% depending on the particular combination of the test oil and the acceleration method (see Figure 6).

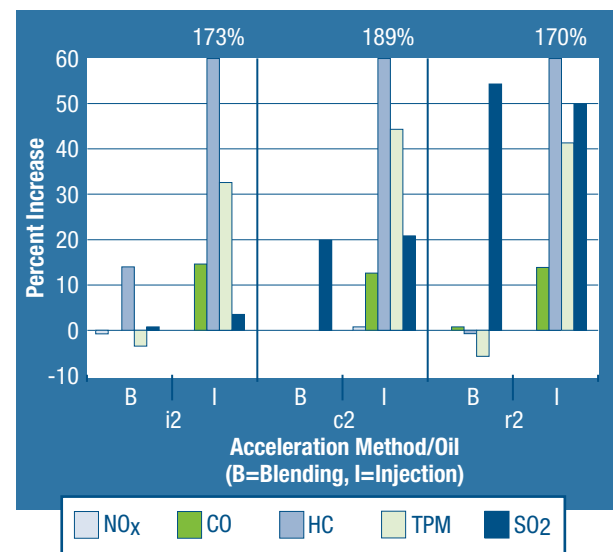


Figure 5. Estimated relative increases in gaseous and particulate emissions resulted from a 100% increase in oil consumption rate—by test oil and acceleration method.

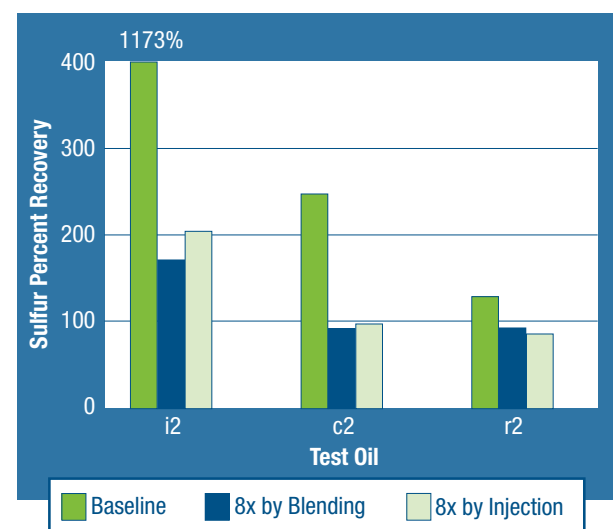


Figure 6. Shown is the estimated sulfur recovery rate at baseline (normal) and at eight times the baseline oil consumption rates—by test oil and acceleration method.

## Phase 2 Work

The primary objective of Phase 2 is to provide data to industry that will increase the collective knowledge base relative to the effects of lubricant on the performance and durability of diesel emission control systems. Specifically, Phase 2 will focus on the lubricant impacts on NO<sub>x</sub> adsorber catalyst systems. Studies specific to lubricant effects on PM control technologies are being addressed by other programs. Resource constraints require that this study focus on only one technology even though other NO<sub>x</sub> control technologies (e.g., urea selective catalytic reduction) are also being considered for use in future engines.

The fluid matrix to be examined in Phase 2 will not be as extensive as the matrix tested in Phase 1, because of the time and expense involved in durability testing. Instead, a set of oils will be blended that vary in S, P, and ash content by varying the levels of ZDDP and detergent (calcium sulfonate, calcium salicylate, and calcium phenate) additives.

A Cummins ISB engine with EGR has been selected for use in Phase 2 testing. An emission control system—including a NO<sub>x</sub> adsorber catalyst—will be integrated with the test engine. For each test, a new NO<sub>x</sub> adsorber catalyst will be installed. Each test will take 400 hours and conduct emission evaluations at 100-hour intervals. As in Phase 1, all Phase 2 tests will be conducted with the 0.6-ppm S base fuel. Certain test oils will be selected for duplicate testing to characterize repeatability.

Results from Phase 2 are expected to be available in the second half of 2004.



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**Produced by the Center for Transportation Technologies and Systems  
at the National Renewable Energy Laboratory (NREL),  
a DOE National Laboratory**

**NREL  
1617 Cole Blvd.  
Golden, CO 80401**

**DOE/GO-102004-1948  
July 2004**



Printed with a renewable-source ink on paper containing at least 50% wastepaper, including 20% postconsumer waste