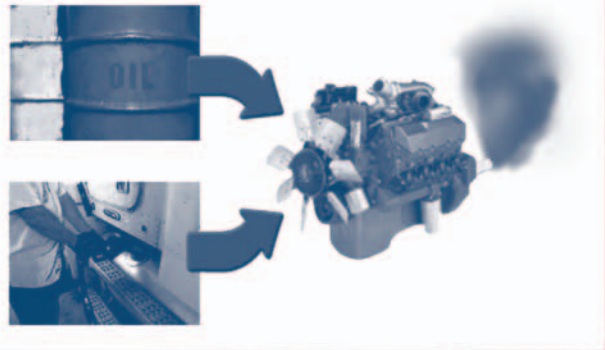# Advanced Petroleum-Based Fuels— Diesel Emissions Control (APBF-DEC)

Lubricants Project Phase 1

Final Report

U.S. Department of Energy

Energy Efficiency and Renewable Energy

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

The test program and subsequent data analysis represent a collaborative effort of a technical working group consisting of representatives from the government and industry organizations listed on the front cover of this report. The work group prepared this report using methods believed to be consistent with accepted practice. All results and observations are based on information available using technologies that were state-of-the-art at the time of this effort. To the extent that additional information becomes available, or factors on which analyses are based change, the finding could subsequently be affected.

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Abbreviations

Abbreviation	Definition
ACC	American Chemistry Council
ACEA	*Association des Constructeurs Europeens d'Automobiles* (European Association of Automobile Manufacturers)
APBF–DEC	Advanced Petroleum-Based Fuels–Diesel Emissions Control
API	American Petroleum Institute
ASTM	American Society for Testing and Materials
ATL	Automotive Testing Laboratories
B	Boron
bhp	Brake horsepower
Ca	Calcium
CARB	California Air Resources Board
CCV	Closed crankcase ventilation
CFR	Code of Federal Regulations
CO	Carbon monoxide
CO_2	Carbon dioxide
CVS	Constant volume sampler
DEC	Diesel Emissions Control
DECSE	Diesel Emissions Control Sulfur Effects
DOC	Diesel oxidation catalysts
DPF	Diesel particle filter
DRI	Desert Research Institute
ECS	Emission control system
EGR	Exhaust gas recirculation
EMA	Engine Manufacturers Association
EPA	U.S. Environmental Protection Agency
FID	Flame ionization detector
ft-lb	Foot-pound
H_2S	Hydrogen sulfide
HC	Hydrocarbon
HEPA	High-efficiency particulate air
HFRR	High-frequency reciprocating ring
Hg	Mercury
hp	Horsepower
kW	Kilowatt
MECA	Manufacturers of Emission Controls Association
Mg	Magnesium
Mo	Molybdenum
NDIR	Nondispersive infrared
Nm	Newton-meter
NO_x	Nitrogen oxides
NPRA	National Petrochemical and Refiners Association

Abbreviation	Definition
OICA	*Organisation Internationale des Constructeurs d'Automobiles* (International Organization of Motor Vehicle Manufacturers)
ORNL	Oak Ridge National Laboratory
P	Phosphorus
PAH	Polycyclic aromatic hydrocarbon
PAO	Polyalphaolefin
PC	Principal component
PCA	Principal components analysis
PC-10	Proposed Category 10
PM	Particulate matter
PMCC	Pensky-Martin Closed Cup
ppm	Parts per million
rpm	Revolutions per minute
S	Sulfur
SCAQMD	South Coast Air Quality Management District
SCFM	Standard cubic feet per minute
SCR	Selective catalytic reduction
SO_2	Sulfur dioxide
SO_4	Sulfate
SOF	Soluble organic fraction
SUV	Sport-utility vehicle
SwRI	Southwest Research Institute
TBN	Total base number
TPM	Total particulate matter
ZDDP	Zinc dialkyl-dithiophosphate
Zn	Zinc

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Executive Summary

The Advanced Petroleum-Based Fuels – Diesel Emission Control (APBF-DEC) project is a government/industry collaboration seeking 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. APBF-DEC consists of five projects that use a systems approach to enhance the collective knowledge base on 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_x) adsorber catalyst/DPF technologies for passenger cars, light-duty trucks/SUVs, and heavy-duty applications (three projects on different engine/vehicle platforms)

- Lubricant formulations that may affect the performance and durability of advanced diesel emission control systems.

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).

This report summarizes the results of the first phase of the lubricants project, which is investigating the impact of lubricant formulation on emissions and the emission control system's durability.

ES.1 Introduction

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 been demonstrated to have a 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 the devices is so extreme, and the durability requirements of heavy-duty commercial vehicles are so demanding, that 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.

Diesel lubricant is known to be consumed during the normal operation of the engine in small but not insignificant quantities. While the quantities may be small, the sulfur content in engine oil is typically higher than that of fuel by an order of magnitude or more, elevating the level of concern accordingly. Other constituents of the lubricating 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.

In anticipation of such challenges, engine makers and the oil and additive industry are actively developing a new specification for lubricating oil to be used in catalyst-equipped diesel engines. This specification, Proposed Category 10 (PC-10), is scheduled for adoption by 2006 and may trigger the most drastic changes in oil formulation in many years. However, because of the limited experience with these new emission control technologies, little data currently exists 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, compromising engine durability and oil drain intervals, both of which have a significant impact on the vehicle owner's economic bottom line.

It is therefore critical that the effects that lubricants have on emissions be well quantified and evaluated so that appropriate lubricants can be developed to protect the emission control systems while continuing to provide superior engine protection.

ES.2 Project Overview

The objective of this study is to evaluate the effects of lubricant formulation on emissions from a multi-cylinder engine (without a catalyst). This lubricants study serves as the first phase of a two-phase project to evaluate the impact of lubricant formulation on the performance and short-term durability of diesel emission control devices. As shown in Figure ES-1, Phase 1 is concerned with the effects of lubricants on engine-out emissions. The follow-on phase will rely, to a great extent, on the results of this initial phase and will include extended-duration engine tests (with catalysts).
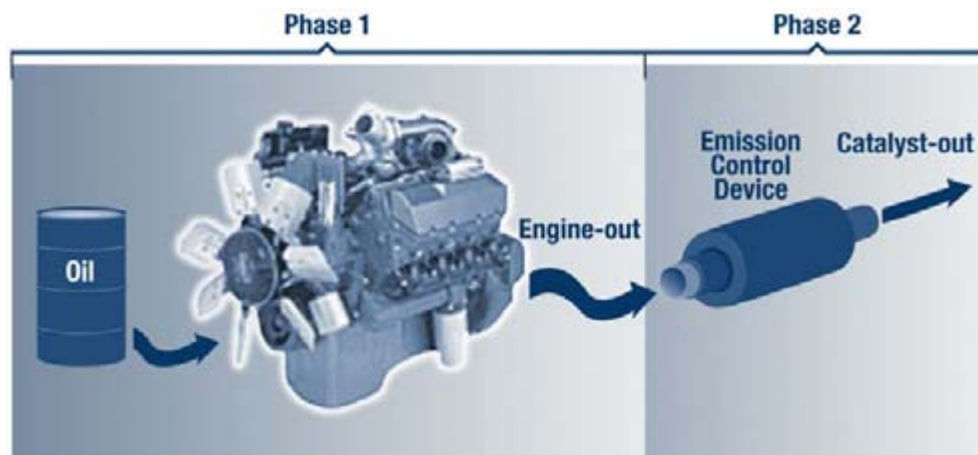
Figure ES-1.   Effects of lubricants on engine-out emissions.

Objectives

The objectives of Phase 1 were:

1. To characterize lubricant additive effects on engine-out emissions
2. To characterize lubricant basestock effects on engine-out emissions

3. To gather information that will provide the basis for follow-on work to study how these lubricant-derived emission species affect the performance of diesel emission control systems.

Phase 1 of the Lubricants project was conducted in two parts. Part 1 investigated the effects of lubricant formulation on engine-out emissions, and Part 2 was designed to show how the rate and mechanism (combustion versus blowby) of oil consumption might affect the relationship between oil formulation and oil-derived emissions.

Research Questions

The following research questions were developed to guide the Phase 1 experimental design, testing, and subsequent data analyses:

Phase 1, Part 1

Q1.1: Are there significant differences in engine-out emissions that can be attributed to oil properties?

Q1.2: If so, how much of an impact is attributed to the properties of the additive packages and how much is attributed to the base oil?

Q1.3: Can the emissions of selected species (specifically metals) be predicted from the properties of the test oils and fuel.

Q1.4: Can we identify other indirect (empirical) relationships between oil properties and engine-out emissions?

Phase 1, Part 2

Q2.1: How do emissions change as a function of oil consumption rate for each oil type and acceleration method?

Q2.2: How does oil type affect these changes?

Q2.3: How does the oil consumption method affect these changes?

Q2.4: Can the combined effects of these methods be predicted from estimated effects of each method, i.e., are there interactions between the acceleration methods?

The purpose of Part 2 was to determine whether an appropriate method for accelerating oil consumption rates could be developed for use in a rapid catalyst aging protocol. This information was needed for Phase 2 of the program, which is looking at lubricant impacts on diesel emission control systems. 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.

Methods

Controlled laboratory tests, chemical and physical measurements, and statistical modeling were used to achieve the objectives of this study. The experimental design for Phase 1 involved the selection of the engine and test hardware, test fluids (fuels and lubricants), emissions measurements and test matrices. An International 7.3L T444E engine was used. The base engine as provided meets the applicable EPA emission standards for 1999 on-highway certification (4.0 g/bhp hr NO_x and 0.1 g/bhp hr PM). Additional retrofit hardware was installed on the engine to allow cooled exhaust gas recirculation (EGR) and closed crankcase 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 blends. A statistical approach was employed to select 13 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 API. They span the commercially available offerings in terms of sulfur content, saturation, viscosity index, and volatility. All test oils used the same olefin copolymer viscosity index improver that was dissolved in a light fraction of the base oil. All tests were conducted with the ultra-low sulfur (0.6-ppm S) base fuel developed previously for the Diesel Emissions Control – Sulfur Effects (DECSE) project, a predecessor to APBF-DEC.

Particulate matter (PM), NO_x, sulfur dioxide (SO_2), hydrocarbons (HC), carbon monoxide (CO), and carbon dioxide (CO_2) emissions were measured during four steady-state test modes from the OICA (13-mode) procedure. Each mode was run for 30 minutes to allow enough time for adequate sampling of PM and SO_2 emissions. The engine was allowed to stabilize at each mode before sampling was initiated. Before the start of an evaluation, the engine was triple flushed with the test oil to be evaluated. A 2-hour "break-in" was conducted and evaluations commenced. Each evaluation consisted of duplicate four mode steady-state runs.

Test matrices for Parts 1 and 2 of Phase 1 were developed to ensure that there would be sufficient high-quality data to meet statistical requirements for addressing the study questions without exceeding resource constraints. Results from the engine-out emissions tests in Part 1 were used to develop the test plan for the accelerated oil consumption measurements in Part 2.

Among other methods, 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. Actual consumption rates were used to calculate the mass rate of engine-out emissions for any given element. These calculated emission levels were then compared with the total measured emissions for any element. Regression analysis was performed to investigate the relationship between the measured values and the calculated values and to determine the recovery rate for any given element.

Figure ES-2 illustrates the system that was used to accelerate oil consumption in Part 2 of Phase 1. Precisely measured amounts of lubricating oil were introduced to the exhaust stream in three ways: (a) blended directly into the diesel fuel supply (doping), so that the oil was then burned in the engine along with the fuel, (b) injected under pressure into the exhaust manifold, to

simulate the blowby of oil from the engine reservoir directly to the exhaust, or (c) both blended in the fuel and injected into the exhaust manifold at the same time.
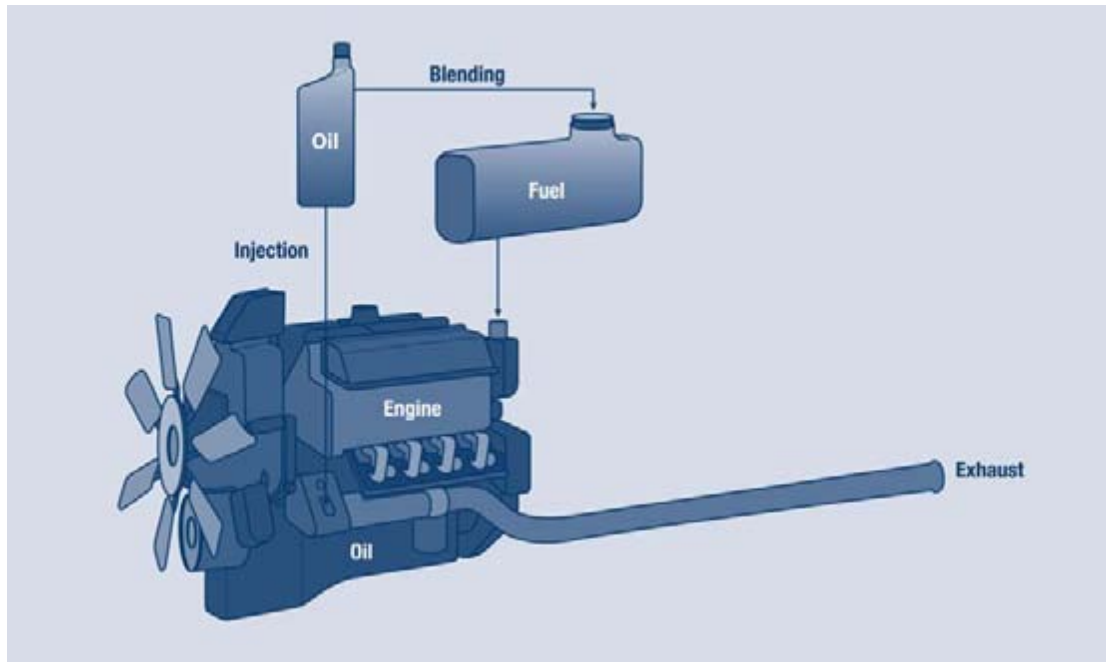
Figure ES-2.   Methods of simulating accelerated oil consumption.

ES.3 Phase 1 Findings and Conclusions

The following is a summary of the significant conclusions from this study. Further details are provided in sections 3 and 4 of the report.

Part 1 Findings and Conclusions

Impact of Oil Formulation on Gaseous and Total Particulate Emissions

- Oil formulation has statistically significant effects on nearly every emissions component. Figure ES-3 shows the range and selected percentiles of gaseous and total particulate matter (TPM) emissions that were observed across 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_x, CO, HC, and TPM emissions vary by 10% to 20% across the oils tested, while SO_2 emissions vary by an order of magnitude.
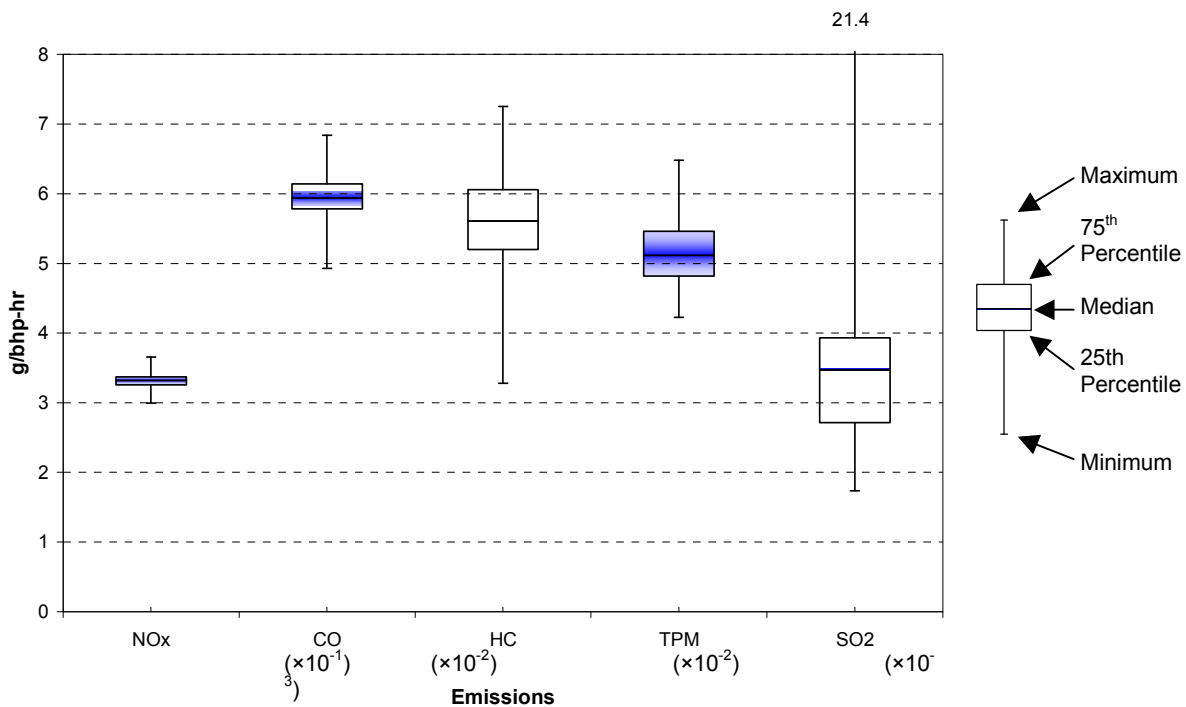
Figure ES-3.   Range and selected percentiles of mass emission rates of gaseous and particulate matter emissions across all oil types tested.

These findings support the motivating hypothesis for this study. That is, oil formulation for diesel engines using low sulfur fuel can have a significant impact on SO_2 emissions. It is known that SO_2 has a negative impact on the durability of certain advanced emission control technologies.

Impact of Oil Formulation on Emissions of Metals

- The emissions of lubricant-derived metals (S, Ca, Zn, P, and Mg) are highly correlated with emissions predicted from the composition of oil (and fuel sulfur); however, recovery rates vary considerably (ranging from 27% for Mg to 127% for S), and certain oils deviate significantly. Figure ES-4 is a comparison of predicted and actual emissions of total sulfur emissions. Oil i2 yielded sulfur emissions 8 to 10 times higher than predicted by the mass balance.
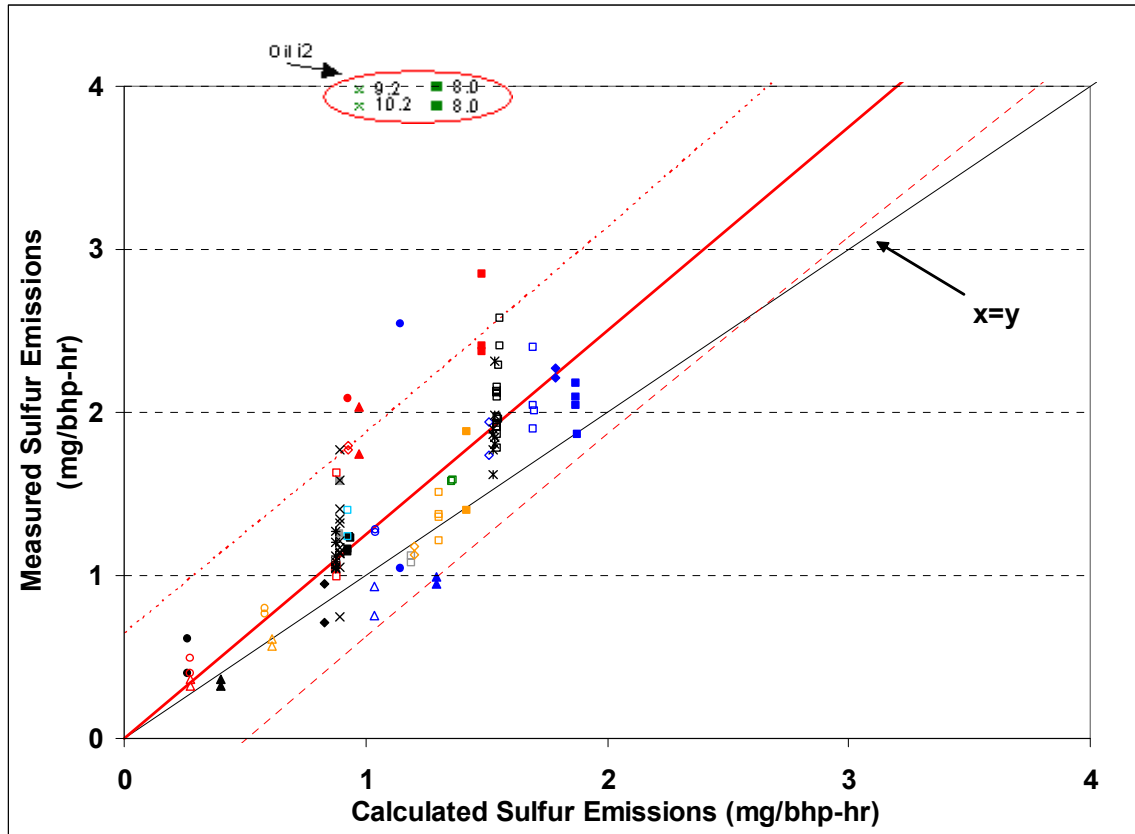
Figure ES-4.   *Mass balance comparison of measured and calculated sulfur emissions – with estimated recovery regression line and 95% confidence interval.*

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 likely not the case. The recovery rate is indicative of 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. However, it was beyond the scope of this study to investigate the specific fate of the various elements. The apparent recovery rate for sulfur is significant because it is perhaps the most scrutinized of the potential catalyst poisons, especially with respect to NO_x adsorber catalysts. It is important to note, though, that the nature of the sulfur compounds emitted is more important than the fact that they are emitted.

Impact of the Source of Sulfur in Oil on SO$_2$ Emissions

- Although the sulfur content of the oil is the primary predictor of SO$_2$ emissions (59% correlation), the results demonstrate that oils containing higher levels of Zn and Mo produce lower levels of SO$_2$ emissions. Adding Zn and Mo to the SO$_2$ prediction model increases the correlation from 59% to 74%.

This finding offers additional evidence that the *source* of sulfur in the lubricant has an impact on the resultant emissions of SO$_2$. 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_2 in the exhaust. This also suggests that chemical limits may need to include additional factors to maximize the ability to predict SO_2 emissions based on the oil analysis.

Part 2 Findings and Conclusions

Impact of Oil Consumption Method on Gaseous and Total Particulate Emissions

- 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 via injection of oil into the exhaust stream. However, if oil consumption is doubled by blending oil with the fuel, the impact on HC, CO, and PM emissions is negligible.
  - NO_x 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_x, and PM emissions when oil consumption is accelerated.
  - The relative increase in SO_2 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 ES-5).
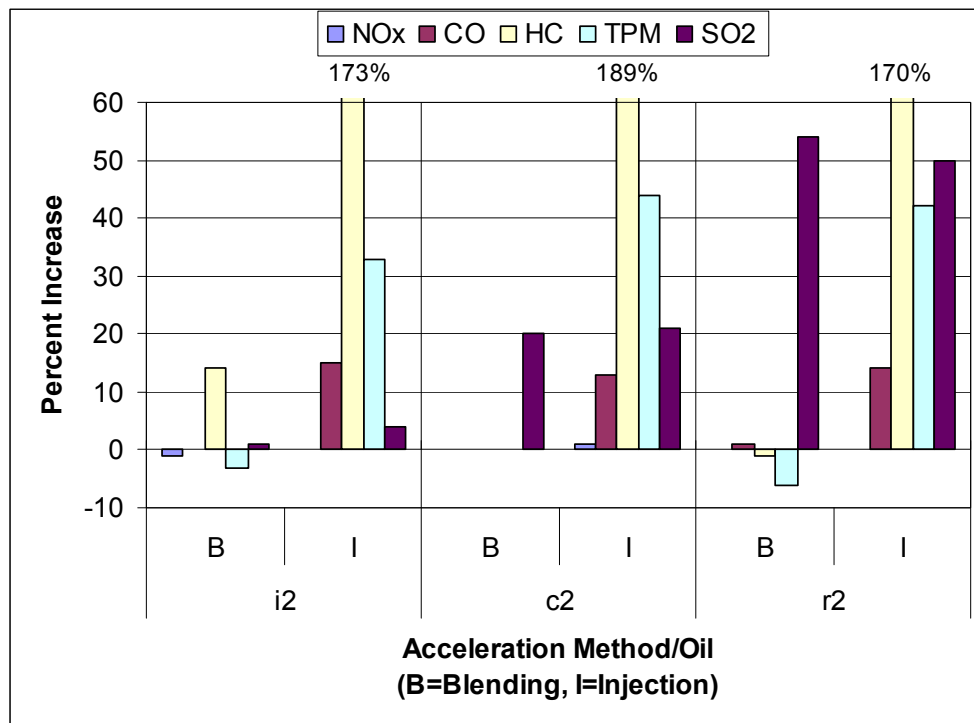
Figure ES-5. Estimated relative increase in gaseous and particulate emissions resulting from a 100% increase in oil consumption rate – by test oil and acceleration method.

Impact of Oil Consumption Method on Metal Recovery Rates

- 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%).
  - Relative recovery rates for S range from 25% to 85% depending on the particular combination of test oil and acceleration method (Figure ES-6).

Figure ES-6. Estimated sulfur recovery rate at baseline (normal) and 8x baseline oil consumption rates – by test oil and acceleration method.

As evidenced in Figures ES-5 and ES-6, neither the blending approach nor the injection approach was adequate to simulate the sulfur emissions expected from the baseline testing. This disparity was especially great with Oils i2 and c2, which exhibited non-standard behavior relative to the other products tested. This inability to accelerate oil consumption realistically—with respect to the measured emissions—left the project team little confidence in utilizing such a scheme for rapid catalyst aging. All Phase 2 tests (described below) were conducted without using artificial means to accelerate oil consumption.

ES.4 Future Work

A second phase of this study is currently underway. The primary objective of Phase 2 is to provide data to the industry that increases the collective knowledge base relative to the effects of lubricant on the performance and durability of diesel emission control systems. Specifically, the project will focus on NO_x adsorber catalyst systems. Studies specific to lubricant effects on PM control technologies are being addressed in other programs. Resource constraints require that this study focus on only one technology, even though other NO_x control technologies (e.g., urea selective catalytic reduction) are also being considered for use in future engines.

The fluid matrix examined in Phase 2 is not as robust as was tested in Phase 1, due largely to the time and expense involved in durability testing. Instead, a set of oils has been blended that varies in sulfur, phosphorus, and ash content by varying the levels of zinc dialkyl-dithiophosphate (ZDDP) and detergent (calcium sulfonate, calcium salicylate, and calcium phenate) additives.

A Cummins ISB engine with EGR is being used for the second phase of testing. A full-flow NO_x adsorber catalyst and hydrocarbon dosing system has been integrated with the test engine. For each test, a new NO_x adsorber catalyst is installed. Each test is 400 hours in duration and includes emission evaluations at 100-hour intervals. All Phase 2 tests are being conducted with the 0.6-ppm S base fuel. Certain test oils have been 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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Section 1: Introduction

1.1 APBF-DEC Overview

APBF-DEC is a government/industry project to identify and evaluate:

- 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 that could lead to even lower emissions beyond 2010.

The project is funded and directed by federal and state government agencies, trade associations, and private industry. The primary sponsors are 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).

The Environmental Protection Agency (EPA) is providing technical assistance. Additional technical support is being provided by 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 enable clean and efficient transportation systems. APBF-DEC consists of five individual projects that are evaluating how sulfur and other compounds impact the performance and durability of advanced diesel emission control systems. The projects are summarized in Table 1.1-1.

Table 1.1-1.   APBF-DEC Project Summary.

Technology	Platform	Test Vehicle/Engine	Subcontractor
NO_x Adsorber Catalysts and Diesel Particle Filters	Light-duty	1.9L TDI Audi A4	FEV
	SUV/Light Truck	Chevrolet Silverado Isuzu/GM Duramax	Southwest Research Institute
	Heavy-duty	Cummins ISX (engine only)	Ricardo, Inc.
Urea Selective Catalytic Reduction and Diesel Particle Filters	Heavy-duty	Caterpillar C12	Southwest Research Institute
Lubricants	Medium-duty	International T444E (Phase 1)	Automotive Testing Laboratories (Phase 1)
		Cummins ISB (Phase 2)	Analytical Engineering, Inc. (AEI)

This report summarizes the results from Phase 1 of the APBF-DEC Lubricants project.

1.2 Background

The previously completed Diesel Emission Control – Sulfur Effects (DECSE) project (NREL 2002) quantified the impact of diesel fuel sulfur on the performance and short-term durability of diesel emission control devices [diesel oxidation catalysts (DOCs), lean-NO_x catalysts, NO_x adsorber catalysts, and diesel particle filters (DPFs)]. Because some of these new technologies have demonstrated a sensitivity to fuel-borne sulfur, considerable research was conducted, and regulations limiting the permissible levels of sulfur in diesel fuel were promulgated. Beginning in June 2006, on-highway diesel fuel will be subject to a maximum sulfur content of 15 ppm. However, the sensitivity of the emission control devices is so demanding, and the durability requirements of heavy-duty commercial vehicles are so long, that 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.

Diesel lubricant is known to be consumed during the normal operation of the engine in small but not insignificant quantities. While the quantities may be small, the sulfur content of engine oil is typically higher than that of fuel by an order of magnitude or more, elevating the level of concern accordingly. Other constituents of the lubricating oil, such as wear control additives, have been found to be an issue for three-way catalysts used with gasoline engines (Ball et al. 1997; Ueda et al. 1994) and may cause similar problems in diesel emission control systems.

For these reasons, this project was planned within APBF-DEC to look specifically at lubricant effects on catalyst durability and emissions. Meanwhile, industry is actively developing a new category of diesel lubricants for use in catalyst-equipped engines (PC-10). The results of this study will provide critical information to the developers of this new performance standard.

1.3 Project Scope, Goals, and Objectives

The objective of this study is to evaluate the effects of lubricant formulation on emissions from a multi-cylinder engine (without a catalyst). This lubricants study serves as the first phase of a two-phase project to evaluate the impact of lubricant formulation on the performance and short-term durability of diesel emission control devices. Phase 2 will rely, to a great extent, on the results of this initial phase and will include extended- duration engine tests (with catalysts).

The objectives of Phase 1 are:

1. To characterize lubricant additive effects on engine-out emissions

2. To characterize lubricant basestock effects on engine-out emissions

3. To gather information that will provide the basis for follow-on work that will study how these lubricant-derived emission species impact diesel emission control system performance.

Phase 1 of the lubricants project was conducted in two parts. Part 1 investigated the effects of lubricant formulation on engine-out emissions and Part 2 was designed to show how the rate and mechanism (combustion versus blow-by) of oil consumption might affect the relationship between oil formulation and oil-derived emissions. A set of study questions were developed for Parts 1 and 2 to guide the experimental design, conduct of testing, and subsequent data analyses.

The purpose of Part 2 was to determine whether an appropriate method for accelerating oil consumption rates could be developed for use in a rapid catalyst aging protocol. This was desired for Phase 2 of the program, which will look at lubricant impacts on diesel emission control systems. It is believed that the effects, when present, are cumulative and may require long run times to reveal themselves. Therefore, accelerated aging protocols would allow for more tests in fewer hours and with less cost.

The study questions for Phase 1, Part 1 are as follows:

Q1.1:   Are there significant differences in engine-out emissions that can be attributed to oil properties?

Q1.2:   If so, how much of an impact is attributed to the properties of the additive packages and how much is attributed to the base oil?

Q1.3:   Can the emissions of selected species (specifically metals) be predicted from the properties of the test oils and fuel?

Q1.4:   Can we identify other indirect (empirical) relationships between oil properties and engine-out emissions?

The study questions for Phase 1, Part 2 are as follows:

Q2.1:   How do emissions change as a function of oil consumption rate for each oil type and acceleration method?

Q2.2:   How does oil type affect these changes?

Q2.3:   How does the oil consumption method affect these changes?

Q2.4:   Can the combined effects of these methods be predicted from estimated effects of each method? (i.e., Are there interactions between the acceleration methods?)

Phase 2 is planned to include a series of NO_x adsorber catalyst durability tests using a variety of test oils. The objective of the study is to determine which, if any, lubricant-derived emission species impact the performance or short-term durability of diesel emission control systems that are being developed to meet future regulations.

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Section 2: Technical Approach

This section presents the experimental design, including the laboratory and emissions measurement setup, as well as the data handling and statistical analysis methods.

2.1 Experimental Design

The experimental design for Part 1 began with the selection of the engine and emission test hardware, an ultra-low sulfur test fuel, and representative additive packages and base oils. These selections are discussed in Sections 2.1.1 through 2.1.5. Section 2.1.6 summarizes the measured properties of the test oils. The test matrices for Parts 1 and 2 are presented in Section 2.1.7.

2.1.1 Test Engine

A 1999 International T444E-HT engine was used in this study. All tests were preformed at Automotive Testing Laboratories (East Liberty, Ohio). The engine is direct-injected, electronically controlled, turbocharged and aftercooled, with a displacement of 7.3L in a V8 configuration with two valves per cylinder. It is equipped with a Siemens electronic control unit and hydraulically actuated electronic unit injectors. The engine produces 157 kW (210 hp) peak power at 2400 rpm and 680 Nm (500 ft lb$_f$) peak torque at 1500 rpm.

The base engine as provided meets the applicable EPA emission standards for 1999 on-highway certification (4.0 g/bhp hr NO$_x$ and 0.1 g/bhp hr PM). Additional retrofit hardware was installed to allow cooled exhaust gas recirculation (EGR) and closed crankcase ventilation (CCV). It is believed that such systems may be commonplace on engines meeting future regulations such as those proposed by the U.S. EPA for 2004 and 2007 (40 CFR Part 86, 2001).

2.1.2 Emissions Measurements

Particulate matter (PM), nitrogen-oxides (NO$_x$), sulfur dioxide (SO$_2$), hydrocarbons (HC), carbon monoxide (CO) and carbon dioxide (CO$_2$) emissions were measured during four steady-state test modes from the OICA (13-mode) steady-state test procedure (1999). Figure 2.1-1 is a performance curve measured on the International T444E illustrating the four steady-state test conditions utilized. Further details of the emission collection and measurement procedures are documented in Section 2.2.
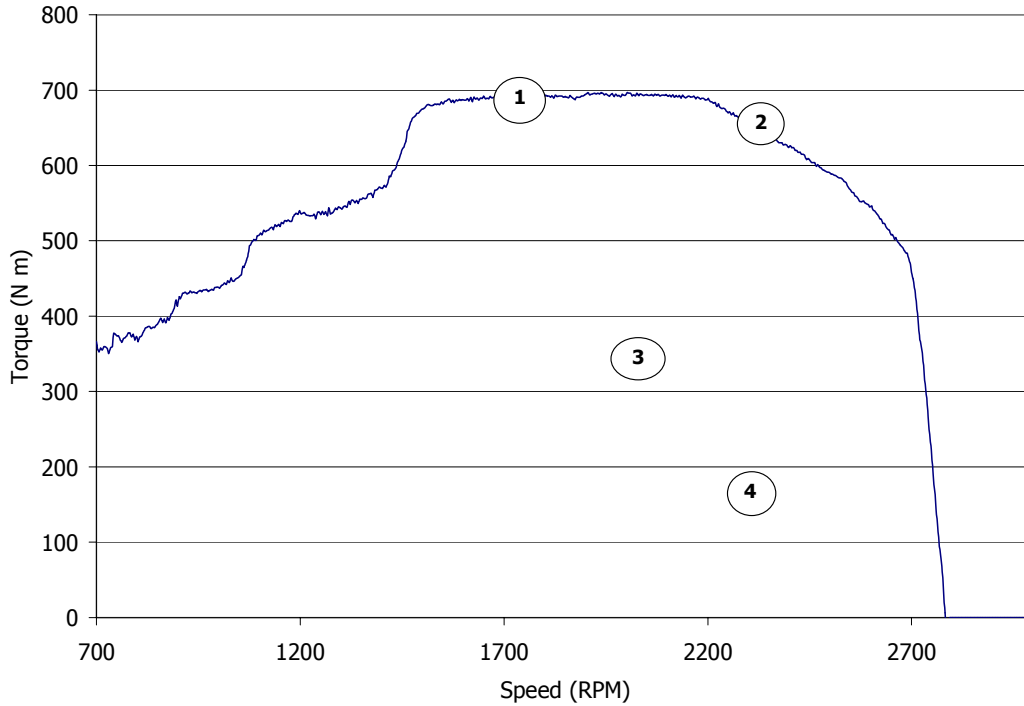
Figure 2.1-1.   *International T444E performance curve and steady-state emission test points.*

2.1.3 Test Fuel

Emissions tests were conducted with the ultra-low sulfur base fuel developed previously for the DECSE projects. Properties of the two batches of DECSE test fuel as determined at the production site are summarized in Table 2.1-1. For this project, an initial shipment of 6,000 gallons of fuel from the first batch of DECSE fuel was delivered via tanker truck to the steam-cleaned underground tank in May 2001. All tests conducted through March 10, 2002, were performed using fuel from this initial shipment, which had a measured sulfur level of 4.5 ppm. The slight difference in sulfur content (i.e., 3.1 ppm at the production site versus 4.5 ppm in the storage tank at the laboratory) is believed to be due to unintentional contamination during shipping. This volume proved to be inadequate to meet the needs of the full program, and a second delivery of 3,500 gallons was delivered in February 2002. However, this shipment came from the second batch of DECSE fuel, which possessed a slightly lower sulfur content: ~0.6 ppm. When combined with the small volume of residual 4.5 ppm sulfur fuel, the new blend contained 1.0 ppm sulfur. This fuel was used for all tests after March 10, 2002. All test fuel was stored in an underground 10,000-gallon tank.

Table 2.1-1.   Test fuel properties.

Fuel Property	ASTM Test	DECSE Goal	First Batch[a] Measured	Second Batch[b] Measured
Density, kg/m^3	D4052	820-850	826.1	826.2
Viscosity @ 40°C, mm^2/s	D445	>2.0	2	2
Distillation initial boiling point, °C	D86	171-182	185	180
5% recovery, °C	D86		198	197
10% recovery, °C	D86	210-226	207	203
20% recovery, °C	D86		222	219
30% recovery, °C	D86		238	233
40% recovery, °C	D86		251	244
50% recovery, °C	D86	254-271	259	251
60% recovery, °C	D86		266	257
70% recovery, °C	D86		274	265
80% recovery, °C	D86		287	279
90% recovery, °C	D86	310-321	314	312
95% recovery, °C	D86		338	339
Final boiling point, °C	D86	326-360	350	352
Carbon, mass %	D5291		86.3	86.5
Hydrogen, mass %	D5291		13.4	13.4
Sulfur, ppm	D5453	<10	3.1[c]	0.6
Saturates, vol. %	D1319	55-70	70.7	71.4
Olefins, vol %	D1319	1-3	2.3	4.6
Aromatics, vol. %	D1319	25-32	27.0	23.9
Aromatics, wt. %	D5186		28.5	26.9
Polyaromatics, wt. %	D5186	3-10	9.6	8.4
Non-aromatics, wt. %	D5186		71.2	73.1
Sediment, water, vol. %	D1796	<0.05	<0.01	0.0
Ash, wt %	D482		<0.001[d]	
Ramsbottom carbon, wt.%	D524		0.01[d]	0.06
Copper corrosion	D130		1A[d]	
Heat Comb, net, MJ/kg	D240		43.1[d]	43.0
Flash point, PMCC, °C	D93	>52	71	69
Cetane number	D613	42-48	44.8	42.5
Cetane index	D976		53.6[d]	51.5
Cloud point, °C	D2500		-21	-26
Pour point, °C	D97		-21	-23
HFRR[e] lubricity, um	D6079		635/355[d,f]	270[f]
Nitrogen, ppm	D4629			1.5
Particulate matter, mg/L	D2276			6.0

a.   The first batch was produced during the DECSE project.
b.   The second batch, designed to be the same as the first batch, was produced during the APBF-DEC project.
c.   Phillips used ASTM D4045 for sulfur determination.
d.   Value based on intermediate scale-up of fuel blend.
e.   HFRR = High-frequency reciprocating ring.
f.   First batch values shown without/with 55 ppm Octel 35a and 211 ppm OLI-9000 additives. Second batch value shown is with additives.

2.1.4 Lubricant Additive Packages

Five additive companies participating in this project (Ciba, ChevronOronite, Ethyl, Infineum, and Lubrizol) offered 26 candidate additive package formulations for consideration. These formulations were considered to be representative of current and future products. Due to resource constraints it was not possible to test all 26 packages in each of four base oils while also evaluating the repeatability of the test and measurement process. Therefore, a statistical design was employed to select 12 packages (plus one reference package) that would adequately span the range of properties of interest while balancing the contribution between additive suppliers. Table 2.1-2 shows the target ranges of ash and elemental compositions of the blended oils derived from these additives.

Table 2.1-2. Target ranges of additive constituent concentrations in finished oils – as proposed by additive suppliers.

Constituent	Range of Concentrations
Ash Level	0 – 1.85%
Sulfur	0 – 6590 ppm
Calcium	0 – 4770 ppm
Zinc	0 – 1900 ppm
N	0 – 2235 ppm
P	0 – 1700 ppm
B	0 – 1235 ppm
Cl	0 – 200 ppm
Mo	0 – 284 ppm
Mg	0 – 1700 ppm

Details concerning the statistical approach to selecting representative additive packages are documented by Orban et al. (2003) and are summarized in Appendix A. Specifications of the finished oils using the 12 test packages (randomly labeled "a" through "l") and the reference oil ("r") are provided in Table 2.1-3. These specifications account for the recommended blending rate but not the constituents of the base oils.

The reference oil that was selected used a commercial CH-4 additive package, typical of modern formulations. Its relatively high sulfur content (4454 ppm) was desired because it was originally planned to use for oil consumption measurements (via sulfur balance). Periodic evaluations using the reference oil complemented the data analysis by allowing for the tracking of data trends that might be the result of changes in measurement equipment or in the engine itself. Further details regarding the use of reference data are included in Appendix D.

Table 2.1-3.   Target properties of additive packages.

Property	Additive Package												
	a	b	c	d	e	f	g	h	i	j	k	l	r
Ash Level, %	1.20	0.00	1.20	1.50	1.85	0.75	1.44	1.40	0.60	1.40	0.30	0.23	1.35
S, ppm	0	5	4950	4500	6590	2785	3246	2921	4226	2224	20	725	4454
Ca, ppm	3484	0	3950	800	4770	1820	3130	3130	1748	4128	870	415	3412
Zn, ppm	0	0	0	1900	1560	860	1319	865	0	0	0	225	1269
N, ppm	0	950	2000	1200	970	1286	1182	1137	0	1560	2235	1457	855
P, ppm	0	670	600	1700	1420	760	1201	788	0	0	0	587	1156
B, ppm	1099	0	0	300	150	60	1235	143	0	0	985	176	0
Cl, ppm	100	0	<100	200	0	126	0	0	100	18	0	60	80
Mo, ppm	0	0	0	0	170	0	0	284	0	0	0	0	0
Mg, ppm	0	0	<50	1700	0	0	277	277	0	0	0	0	0

2.1.5 Lubricant Base Oils

Base oils were selected from each of the four major base oil categories as defined by API. They span the commercially available offerings in terms of sulfur content, saturation, viscosity index, and volatility. Table 2.1-4 contains specifications of the selected base oils.

Table 2.1-4.   Base oils selected for Phase 1 testing.

API Group	Supplier	Refinery	Sulfur Content (ppm)	Saturates (%)	Finished Oil Viscosity Grade
Group 1	Valero	Paulsboro, NJ	4800-5600	75	15W40
Group 2	Excel	Lake Charles, LA	<20	99+	15W40
Group 3	Motiva	Port Arthur, TX	<5	99+	10W40
Group 4	BP	*synthetic*	0	PAO	5W40

Analysis of Test Lubricants

Oil samples were taken from the oil gallery at the end of each emission test and sent to Southwest Research Institute for full analysis. The measured properties of all finished test oil samples as well as some unused oils are summarized in Appendix B. In general, the measured properties matched the target properties with reasonable agreement. As discussed below in Section 2.1.7, all 13 additive packages were blended in the Group 2 base oil; but only the first six ("a" through "f") were blended in base oils 1, 3, and 4. Figure 2.1-2 displays pairwise plots of selected average measured properties and the interdependence of certain key properties.
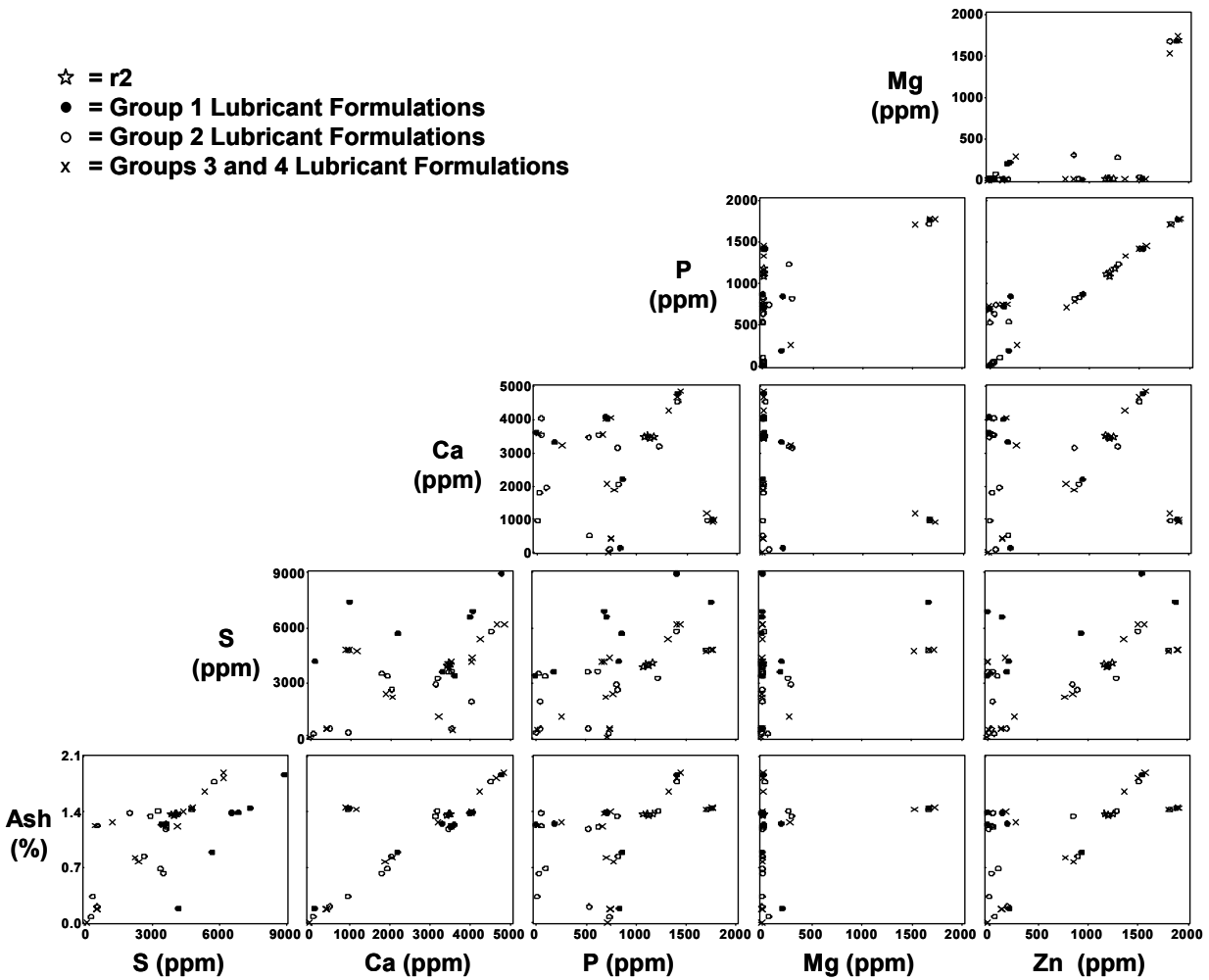
Figure 2.1-2.   Pairwise plot of measured properties.

2.1.7 Test Matrix

Part 1

The objective of Part 1 was to characterize effects of lubricant properties on engine-out emissions. The test matrix was developed to ensure that there would be sufficient high-quality data to meet statistical requirements for addressing the study questions without exceeding resource constraints. The resulting design matrix for Part 1 included the following features:

▪ One base oil was selected from each of the four API 1509 base oil groups.

▪ Twelve (12) additive packages were chosen to be statistically representative of current and future products offered by additive suppliers, while spanning the target ranges of elemental composition and ash levels as shown in Table 2.1-3.

- One additive package was blended in a Group 2 base oil to create a reference oil that is representative of current products. The reference oil was tested periodically to ensure consistency of the test and measurement procedures.

- Tests were performed in random order within oil groups.

- Each combination of additive and base oil was tested with two consecutive four-mode steady-state emissions tests.

- All 12 additive packages were tested in the Group 2 base oil and six were also tested with Groups 1, 3, and 4 base oils. These same six additive packages were tested in duplicate with the Group 2 base oil.

The final test matrix for Part 1 testing is shown in Table 2.1-5.

Each letter represents a single four-mode emissions test performed with one of the 12 additive packages (denoted by letters "a" through "l") or the reference oil (denoted by the letter "r"). Before starting the main test matrix, demonstration tests were performed with additive packages "b" and "e," which had very different sulfur and ash levels. Results from these tests provided assurances that measurement precision and testing consistency were adequate to statistically demonstrate that there are differences in certain emissions levels that can be attributed to variations in oil formulation.

During the main testing program, emissions tests were performed with the reference oil (consisting of additive package r in the Group 2 base oil) after each group of three evaluations. This provided useful data to diagnose variations in test procedures or problems with analyzers or instrumentation. Every other set of reference tests was conducted in conjunction with a 40-hour aging sequence (designated as "rr-age-rr"). This provided additional data to monitor any changes oil consumption rate (based on oil drain-and-weigh measurements before and after the 40-hour period) and to evaluate potential impacts of oil aging on the oil-derived emissions.

Table 2.1-5. Test matrix for Part 1 of Phase 1 testing.

Testing Order	Demo Runs*	Base Oil			
		Group 2	Group 1	Group 3	Group 4
1	bbb	rr-age-rr	rr-age-rr	rr-age-rr	rr-age-rr
2	eee	aa	aa	aa	aa
3	bbb	bb	bb	bb	bb
4	eee	cc	cc	cc	cc
5		rr	rr	rr	rr
6		dd	dd	dd	dd
7		ee	ee	ee	ee
8		ff	ff	ff	ff
9		rr-age-rr			rr-age-rr
10		gg			
11		hh			
12		ii			
13		rr			
1		jj			
15		kk			
16		ll			
17		rr-age-rr			
18		dd			
19		bb			
20		ee			
21		rr			
22		cc			
23		ff			
24		aa			
# of Tests	12	54	18	18	22

* Tested with Group 2 base oil.

Part 2

The objectives of Part 2 were (1) to develop methods to accelerate exposure of emission control systems (ECS) to lubricant-derived emissions, and (2) to compare emission rates among the acceleration methods to determine if any provide a suitable strategy for studying the impact of lubricant formulation on the performance and short-term durability of diesel emission control devices. Three oil consumption acceleration methods were developed: blending oil into fuel, injecting oil into the exhaust manifold, and combination (1/2 blending and 1/2 injection). The resulting design matrix for Part 2 included the following features:

- In addition the reference oil, two oils with unusual mass balance results in Part 1 were selected for testing in Part 2. The two oils are c2 (additive "c" in a Group 2 base oil) and i2. Oil i2 has unusually high sulfur emissions, and oil c2 has unusually high P emissions.

- Three oil consumption rates were studied: 2 times, 4 times and 8 times the base oil consumption rate. The base oil consumption rate was determined from the Part 1 data.

- Oil r2 was tested using all three techniques. Oils i2 and c2 were tested using the blending and the injection techniques but not the combination approach.

- Each combination of oil and acceleration technique was tested with two consecutive 4-mode steady-state emissions tests.

- Duplicate testing was conducted for oil r2 with the blending and the injection techniques.

The final test matrix for Part 2 testing is shown in Table 2.1-6.

Table 2.1-6.   Test matrix for Part 2 of Phase 1 testing.

Testing Order	Oil Consumption Acceleration Technique								
	Blending			Injection			Combination (1/2 injection, 1/2 blending)		
	Oil r2	Oil i2	Oil c2	Oil r2	Oil i2	Oil c2	Oil r2	Oil i2	Oil c2
1	2X, 2X	2X, 2X	2X, 2X	2X, 2X	2X, 2X	2X, 2X	2X, 2X		
2	4X, 4X	4X, 4X	4X, 4X	4X, 4X	4X, 4X	4X, 4X	4X, 4X		
3	8X, 8X	8X, 8X	8X, 8X	8X, 8X	8X, 8X	8X, 8X	8X, 8X		
4	2X, 2X			2X, 2X					
5	8X, 8X			8X, 8X					
6	4X, 4X			4X, 4X					
# of Tests	12	6	6	12	6	6	6	0	0

2.2 Test Procedures and Data Analysis

2.2.1 Test Engine and Associated Hardware

A 1999 International T444E-HT engine, previously used at West Virginia University in support of the DECSE project, was used in this phase of the study. The engine is direct-injected, electronically controlled, turbocharged and aftercooled, with a displacement of 7.3L in a V8 configuration with two valves per cylinder. It is equipped with a Siemens electronic control unit and hydraulically actuated electronic unit injectors. The engine produces 157 kW (210 hp) peak power at 2400 rpm and 680 Nm (500 ft lb$_f$) peak torque at 1500 rpm.

The base engine as provided meets the applicable EPA emission standards for 1999 on-highway certification (4.0 g/bhp hr NO$_x$ and 0.1 g/bhp hr PM). Additional retrofit hardware was procured to allow cooled exhaust gas recirculation (EGR) and closed crankcase ventilation (CCV).

The EGR system is a high-pressure loop configuration which routes exhaust gas from upstream of the turbocharger through a heat exchanger and into the intake, downstream of the compressor and intercooler. A valve installed on the outlet of the cooler allowed modest control of EGR rate.

In addition, exhaust back-pressure control was used to drive EGR flow. The EGR system as installed on the test engine is illustrated in Figure 2.2-1.
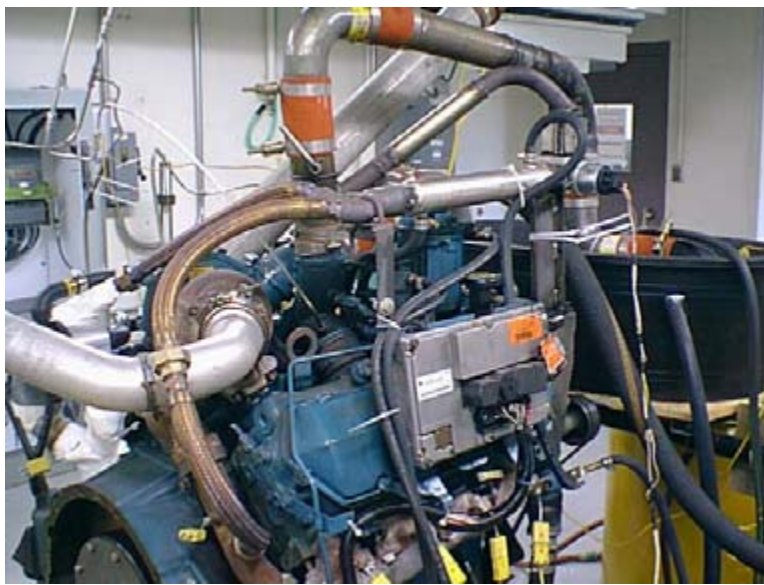
Figure 2.2-1. EGR system installed on the T444E.

The CCV system redirects pressurized crankcase vapors to the pre-compressor intake stream. Because these vapors have the potential to condense on the walls of the intercooler and within the compressor, an impactor type CCV filter, provided by Fleetguard-Nelson, was installed. This particular filter is designed to remove nearly 100% of the oil droplets and up to 70% of the aerosol in the engine blowby. Collected oil was drained back into the engine sump.

2.2.2 Test Cell and Gaseous Emission Measurements

Initially, power absorption was to be switched between an electric DC dynamometer for evaluations and a water-brake dynamometer for long-term oil aging and break-in. Preliminary testing revealed that the control hardware used with the DC dynamometer had a circuit heat limit that precluded its use for 30-minute modal testing. To work around this limitation, the water-brake dynamometer was mated to the DC electric dynamometer to absorb excess power and to allow the requirements of the program to be met.

Exhaust from the engine was ducted into a 15" diameter dilution tunnel. The dilution tunnel flow rate was controlled by a constant volume sampler (CVS) rated at 2700 standard cubic feet per minute (SCFM). Dilution air entering the tunnel was transported through four 8 sq. ft. HEPA filters to remove background particulate matter. These filters are manufactured with blower fans attached to lower the pressure drop across them and to reduce the load on the tunnel blower system. The filters form a box attached to the entrance of the dilution tunnel.

Gaseous emissions were sampled in accordance with the Federal Register (40 CFR 86.1310-90) guidelines for measuring emissions from heavy-duty engines. NO_x was measured via chemiluminescence using a Horiba Model CLA-220 heated NO_x analyzer. CO and CO_2 were measured with Non-Dispersive Infrared (NDIR) analyzers. HC was measured using a heated Flame Ionization Detector (FID).

SO_2 was measured via a wet chemistry technique modeled after EPA Methods 6, 8, and 16. In this method, dilute exhaust is sampled from the tunnel and is passed through a heated filter (to remove PM) and then through a set of impingers that are immersed in an ice bath. The impingers (Figure 2.2-2) are filled with a 3% aqueous hydrogen peroxide solution. SO_2 in the dilute exhaust reacts with the impinging solution and is converted into a sulfate, which can be detected post-analysis using an ion chromatograph. Because sampling during each test mode is integrated instead of measured in real time, part-per-billion (ppb) sensitivity is possible with this technique.
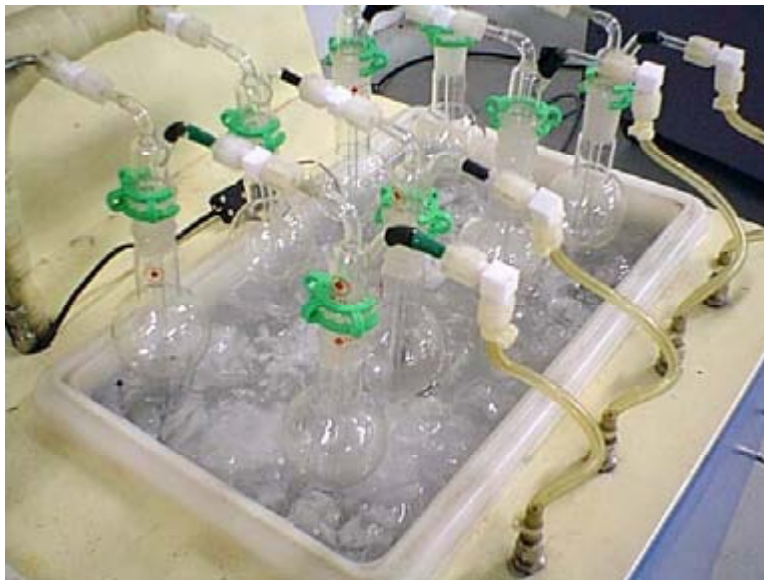
Figure 2.2-2. Impingement system for SO_2 measurement.

2.2.3 PM Collection and Measurement

Three PM sampling trains were installed: one with standard PM sampling filters (EMFAB TX40HI20WW – 70 mm), a second utilizing a separate sample filter (47 mm TEFLO - low metals background, high efficiency) for metals analysis, and a third, larger sampling train (using Pallflex T60A20 70 mm filters) for collecting sufficient quantities (>5 mg) of PM for polycyclic aromatic hydrocarbon (PAH) analysis.

The main PM sampling system used a stainless steel sample probe with 0.333" diameter that collected a sample stream from the tunnel. This sample stream was deposited into a secondary dilution tunnel where it was mixed with room air to reduce the temperature below 125°F as specified in the CFR. The secondary dilution tunnel consisted of an enclosed section of 4" stainless steel pipe approximately 18" in length. The exit of the secondary dilution tunnel led to

the 47 and 70 mm filters for metals and PM measurement respectively. The system was designed to keep filter face velocities below 100 cm/s while providing enough dilution air to keep the filter face temperature below 125°F. The 47 mm filter alone was insufficient to meet these criteria, so this system employed a parallel filter construction. A schematic of this arrangement is provided in Figure 2.2-3. A third sampling train (for PAH) operated on a separate secondary dilution tunnel, and is shown separately in Figure 2.2-4.

The PM filters from train #1 were weighed at ATL, then shipped to Oak Ridge National Laboratory (ORNL) for sulfate and soluble organic fraction analysis. PM filters from train #2 were sent to Desert Research Institute (DRI) for metals analysis (by x-ray fluorescence). PM filters from train #3 were sent to Southwest Research Institute (SwRI) for PAH analysis.
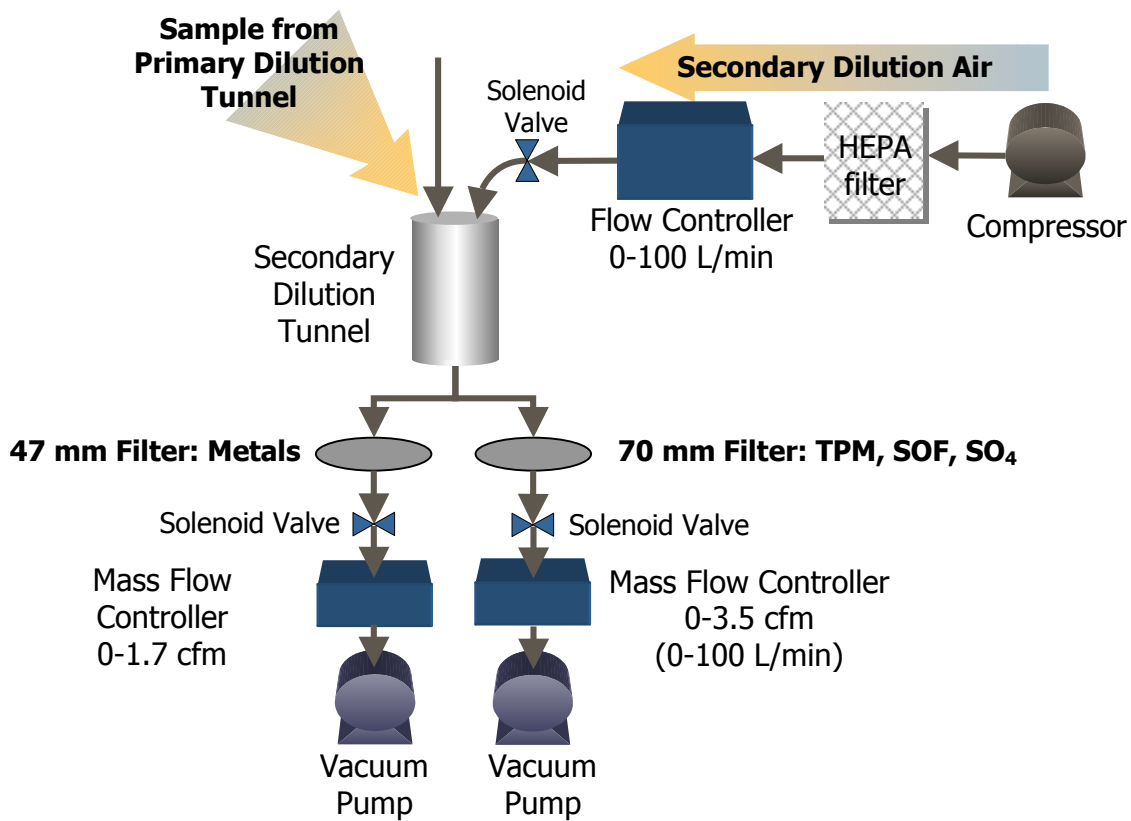
Figure 2.2-3.   Sampling system for PM and metals.
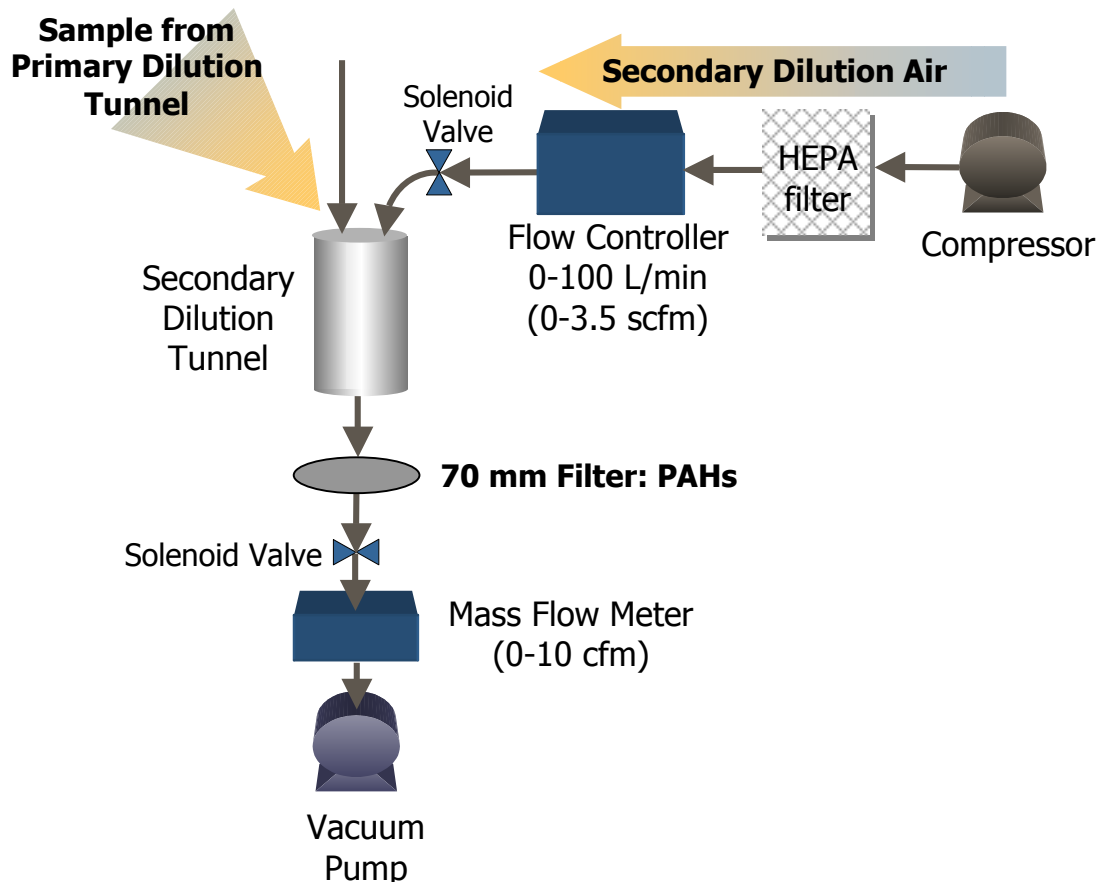
Figure 2.2-4.   Sampling system for PAH.

2.2.4 Testing Methodology

The following is a summary of the step-by-step procedure used on each test day:

1. Previous test oil drained through quick-disconnect fitting by an oil drain pump
2. Oil filter removed and replaced
3. Engine filled with next test oil
4. Engine started and brought to operating temperature
5. Engine stopped and allowed to cool down
6. Engine oil drained through quick-disconnect fitting by oil drain pump (1st Flush)
7. Oil filter removed and replaced
8. Refill engine with test oil
9. Engine started and brought to operating temperature
10. Engine stopped and allowed to cool down
11. Engine oil drained through quick-disconnect fitting by oil drain pump (2nd Flush)
12. Oil filter removed and replaced

13. Refill engine with test oil
14. Engine started and brought to operating temperature
15. Engine run at rated power for 2 hours
16. Engine stopped and water brake dyno mated with driveshaft to DC dynamometer
17. Engine returned to operating temperature
18. Engine set to rated power (2300 rpm, full torque)
19. Back-pressure valve set to produce exactly 4" Hg gauge pressure in exhaust
20. Engine brought to idle and prepared for four-mode test
21. Engine set to steady-state point and emissions analyzers allowed to stabilize
22. After stabilization, sampling systems initiatied and data recorded for 10 minutes
23. Repeat steps 21 and 22 for remaining test modes
24. Repeat steps 18 through 23 for second evaluation test
25. Stop engine and allow to cool slightly and then draw sample of aged oil for analysis.

The same general procedures were used during Part 2 of the experiment, except for the use of blended fuel (metered from drums) in some tests and/or the utilization of the oil injection system as prescribed by the test plan. Complete details of the oil injection system have been included in Appendix C.

2.2.5 Oil Consumption Measurement

Because of its potential to influence oil-derived emission rates, engine oil consumption was closely monitored during the project. Oil consumption was measured using a drain-and-weigh (gravimetric) approach. After an initial characterization of the engine's oil consumption rate, periodic measurements were made throughout the test program. Each "rr-age-rr" test listed in Table 2.1-5 indicates a 40-hour test during which the oil consumption characterization was conducted. Careful documentation of the mass of the initial lubricant charge and the mass of oil drained from the engine after the aging test allowed for the calculation of oil consumption rate. For the purposes of the mass balance analysis, a brake-specific oil consumption rate of 0.179 g/bhp hr was used. This was calculated from a regression analysis of the series of gravimetric measurements made during the test program and equates to approximately 30 g/hr oil consumption over the 40-hour aging test.

2.2.6 Data Handling

Data provided by ATL included second-by-second measurements of NO_x, SO_2, HC, CO, and CO_2, as well as measures of various engine performance parameters. The laboratory also prepared summary reports containing average brake-specific gaseous emissions (g/bhp-hr) for each mode of the 4-mode steady-state tests and weighted average emissions across the composite 4-mode steady-state tests. Total PM emissions for the composite steady-state tests were also reported.

PM breakdown analysis, performed at ORNL, produced estimates of SOF and SO_4 on a mg/filter basis. In addition to the primary and secondary main filters, travel blanks were analyzed. Metals emissions analysis produced estimates of 41 metals on a mg/filter basis with uncertainties of the

estimates. For PAH emissions analysis, the primary and secondary filters were analyzed together. Thus, the chemical analysis produced estimates for 16 compounds on a ng/test basis.

The conversion of SOF, SO$_4$, and metals filter concentrations to brake-specific emissions was performed according to the following formula (as shown for SOF):

$$SOF(g/bhp\text{-}hr) = [PM(g/bhp\text{-}hr) / PM(mg/filter)] \times [SOF(mg/filters) - 2 \times SOFblank(mg/filter)],$$

where SOF(mg/filters) is the sum of SOF weights on the primary and secondary filters, and SOFblank(mg/filter) is the weight of SOF on the associated travel blank. The PAH compound concentrations were converted to brake-specific emissions in the same fashion.

Because most emissions data reduction routines require calculations involving calibration equations or differences in gross filter weights, gaseous and PM emissions measurements were sometimes reported as negative values. These values were replaced with zero before any further calculations or statistical analyses were performed.

Prior to conducting the statistical analysis, an extensive data review was conducted to ensure data completeness and accuracy. After comparing the data received with the data collection plan, the data were stored in a controlled database. Changes, updates, and corrections were carefully monitored and controlled. To identify gross outliers (unusual and unexplained emissions results) and unexplained variations or trends associated with laboratory procedures, plots of emission data versus test order were prepared and shared with the laboratories and the technical committee. In addition, emission results for the reference oils were plotted and statistically analyzed to determine if there were systematic trends over time for data collected during Part 1. If the analysis results indicated significant trends in the reference oil for a particular emission species, appropriate adjustments were made to that emission species. Details for the analysis of trends with reference oil and resolution of measurement issues and outliers are provided in Appendix D. After resolving the data issues identified from the preliminary outlier analysis and the trends analysis on the reference oil, statistical models were fit to the updated data. A second outlier analysis was performed using the standardized residuals from the statistical model fitting. Standardized residuals are commonly used to identify individual data points that are statistically inconsistent with the underlying structure of the data set.

Two types of data issues were identified: individual outliers and testing trends. Lists of outliers identified from the preliminary outlier analysis were sent to the laboratories with instructions to check for clerical errors, equipment failures, or other external factors that could explain the deviation in results. Clerical errors were corrected, and outliers due to known problems were corrected whenever possible. If the data could not be corrected, but the outliers were found to be associated with documented testing or measurement issues, they were eliminated from the analysis. If no explanation was available, the data were retained for the statistical analysis. If the remaining outliers had a major impact on the fit of the statistical model, the analysis was performed twice: once with and once without the outliers.

2.2.7 Statistical Analysis and Modeling

Statistical Analysis Methods for Part 1 Study Questions

The statistical analysis approaches used to address the four study questions of Part 1 are described below, organized by study question.

Q1.1: Are there significant differences in engine-out emissions that can be attributed to oil properties?

Q1.2: If so, how much of an impact is attributed to the properties of the additive packages and how much is attributed to the base oil?

The methods for addressing study questions Q1.1 and Q1.2 are straightforward. A simple one-way analysis of variance (ANOVA) was performed to determine if there are significant differences in engine-out emissions among the oils. Appropriate ANOVA analysis methods were used to test for statistically significant differences in the estimated average emissions among tests performed with different oils. The average emissions for each combination of base oil and additive package and the 95% confidence intervals on these estimates were also calculated. To address the second study question, the following two-factor ANOVA model with interaction terms was fitted to the emissions data obtained from packages tested in all four groups of base oils:

$$\text{Emission}_{ijk} = \mu + \alpha_i + \beta_j + \alpha\beta_{ij} + e_{ijk},$$

where μ = overall mean, α_i = the effect of ith additive (a, b, c, d, e, f), β_j = the effect of jth base oil (Group 1, Group 2, Group 3, Group 4), $\alpha\beta_{ij}$ = the interaction effect, and e_{ijk} = random error.

This model explores the effects of additive packages, base oils, and their interactions on engine-out emissions. If the interaction term was not statistically significant, a reduced model without the interaction term was fitted. Multiple comparison analysis was performed on the main effects to determine which additives or base oils are significantly different from each other in terms of lubricant-derived emissions. When the interaction term was statistically significant, multiple comparisons were performed on all additive and base oil combinations to determine which ones differ significantly.

Q1.3: Can the emissions of selected species (specifically metals) be predicted from the properties of the test oils and fuel?

Mass balance analysis, illustrated in Figure 2.2-5, was used to predict system outputs (particulate and gaseous emissions) from system inputs (e.g., sulfur from fuel and oil consumption). The properties of the fuel and lubricants and their consumption rates, estimated in separate analyses, were used to calculate the mass rate of emissions for any given element. Fuel properties (sulfur content) were measured from the fuel supply line and were routinely checked throughout the project. The metallic content of the fuel was confirmed to be negligible. Properties of lubricants

were derived from lube oil samples taken from the oil gallery at the time of the actual emission test and later analyzed. The total measured emissions for any element, except sulfur, were determined from the results of metal analysis for that element. The total measured emissions for sulfur were determined from sulfur emissions from metal analysis plus SO_4 and SO_2 emission rates. Regression analysis was performed to investigate the relationship between the measured values and the calculated values and to determine the recovery rate for any given element.
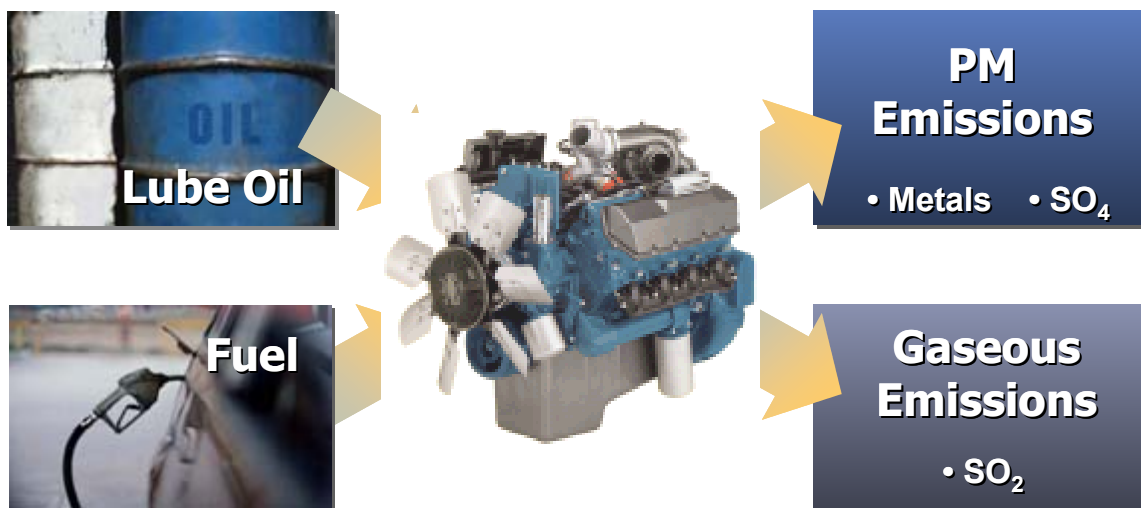
Figure 2.2-5.   Mass balance diagram.

To illustrate this approach, consider the mass balance analysis for sulfur. Concentrations of sulfur in the fuel and most of the test oils were measured. Fuel consumption was measured with a mass flow meter during each of the emissions tests, and oil consumption was determined by drain-and-weigh measurements made during each of the 40-hour aging tests, as described in Section 2.2.5. Measurements of SO_2, sulfate SO_4, and elemental sulfur emissions were used to estimate <u>total</u> sulfur emissions. The wet chemistry method, combined with the extended sampling times, yielded accurate measurements of SO_2, which accounted for more than 95% of the sulfur emissions. The balance of the sulfur came from PM emissions of SO_4 and elemental sulfur. Hydrogen sulfide (H_2S) was not expected to be a significant source of sulfur emissions.

The following regression model was fit to the data:

$$\text{Measured Emissions}_i = \beta_0 + \beta_1 \times E_i + e_i,$$

where β_0 = the intercept, β_1 = the slope (recovery rate), E_i = calculated emissions for i^{th} test, and e_i = random error component.

Hypothesis testing was conducted to test if $\beta_0 = 0$. When there was significant evidence that $\beta_0 = 0$, the reduced model without the intercept was fitted. A 95% confidence interval on β_1 of the final model was constructed to estimate the recovery rate.

Q1.4: Can we identify other indirect (empirical) relationships between oil properties and engine-out emissions?

Multiple regression analysis was used to address study question Q1.4. First, oil properties were determined from the analyses of test oils collected during the testing phase. Stepwise regression analysis was used to select the best models (using the maximum r-squared criterion) for predicting engine-out emissions based on one, two, or three oil properties. The backward selection approach was used to generate a model containing all significant variables. This same approach was applied to the principal component scores for each oil. The principal component scores were obtained by applying Principal Components Analysis to the average values of measurable properties.

Statistical Analysis Methods for Part 2 Study Questions

The statistical analyses approaches used to address the four study questions of Part 2 are described below, organized by study question.

Q2.1: How do emissions change as a function of oil consumption rate for each oil type and acceleration method?

Q2.2: How does oil type affect these changes?

Q2.3: How does the oil consumption method affect these changes?

Q2.4: Can the combined effects of these methods be predicted from estimated effects of each method, i.e., are there interactions between the acceleration methods?

The following regression model was developed to address all four study questions:

$$\text{Emission}_{ijk} = \tau_i + \beta_{ij} \times (R - 1) + e_{ijk},$$

where τ_i = the effect of ith oil (r2, i2, c2) at baseline, R = oil consumption ratio relative baseline, β_{ij} = the linear regression coefficient indicating the dependence of emissions effect on oil consumption ratio for i^{th} oil and j^{th} acceleration method (Blending, Injection, Combination), and e_{ijk} = random error.

This model fits separate intercepts to each oil type and separate slopes to each combination of oil and acceleration method such that the regression lines associated with the same oil have the same predicted emissions at baseline. Data collected during Part 1 testing were not used in this analysis. However, to check for consistency, the predicted emissions at baseline (extrapolated from the data in Part 2) were compared with the average emissions measured in Part 1.

The following hypotheses tests were conducted to address study questions.

Study Question	Hypotheses Tested
2.1	Emissions change as a function of oil consumption rate for each oil type and acceleration method. $H_0: \beta_{ij} = 0$ vs. $H_1: \beta_{ij} \neq 0$.
2.2	(a) Oil type affects the predicted emissions at baseline. $H_0: \tau_i = \tau_i$ vs. $H_1: \tau_i \neq \tau_i$ (b) Oil type affects the slopes (rate of change of emissions) for each method. Blending: (r2 vs. i2, r2 vs. c2, and i2 vs. c2) $H_0: \beta_{i1} = \beta_{i1}$ vs. $H_1: \beta_{i1} \neq \beta_{i1}$ Injection: (r2 vs. i2, r2 vs. c2, and i2 vs. c2) $H_0: \beta_{i2} = \beta_{i2}$ vs. $H_1: \beta_{i2} \neq \beta_{i2}$
2.3	Oil consumption method affects the slopes for each oil. Oil r2: (Blending vs. Injection, Blending vs. Combination, and Injection vs. Combination) $H_0: \beta_{11} = \beta_{12}$ vs. $H_0: \beta_{11} \neq \beta_{12}$ $H_0: \beta_{11} = \beta_{13}$ vs. $H_0: \beta_{11} \neq \beta_{13}$ $H_0: \beta_{12} = \beta_{13}$ vs. $H_0: \beta_{12} \neq \beta_{13}$ Oil i2: (Blending vs. Injection) $H_0: \beta_{21} = \beta_{22}$ vs. $H_0: \beta_{21} \neq \beta_{22}$ Oil c2: (Blending vs. Injection) $H_0: \beta_{31} = \beta_{32}$ vs. $H_0: \beta_{31} \neq \beta_{32}$
2.4	The effects of the combination method can be predicted from estimated effects of each method. $H_0: \beta_{13} - 0.5 \times (\beta_{11} + \beta_{12}) = 0$ vs. $H_1: \beta_{13} - 0.5 \times (\beta_{11} + \beta_{12}) \neq 0$.

Confidence intervals (95%) were used to characterize the statistical uncertainty of emissions estimates and to determine if there were significant effects of oil type and oil consumption method on engine-out emissions.

Finally, mass balance analysis was performed to quantify the rate of recovery of metals in the emission as a function of oil type, oil consumption rate, and acceleration method. The following regression model was developed for investigating the relationships between measured emissions and calculated emissions.

$$\text{Measured Emission}_{ijk} = \tau_i + \beta_{ij} \times (E_{ij} - E_{i@\text{baseline}}) + e_{ijk},$$

where τ_i = the effect of ith oil (r2, i2, c2) at baseline, β_{ij} = the linear regression coefficient indicating the dependence of measured emissions and calculated emissions for i^{th} oil and j^{th} acceleration method (Blending, Injection, Combination), E_{ij} = calculated emissions for i^{th} oil and j^{th} acceleration method, $E_{i@\text{baseline}}$ = calculated baseline emissions for i^{th} oil, and e_{ijk} = random error.

The model fits separate intercepts to each oil type at the baseline (i.e., no blending and/or injection) and separate slopes to each combination of oil type and acceleration method. Thus, this model gives the same estimated mean emissions for different acceleration methods associated

with the same oil when there is no blending and/or injection. Part 2 mass balance analysis results are compared with those from Part 1 to find out how comparable/different they are.

Model Validation

The statistical models used in these analyses are based upon assumptions that the model errors are distributed independently according to normal distributions. Preliminary analysis demonstrated that the emissions data tend to follow a normal distribution; therefore, no transformation was applied to the data. In general, moderate departures from normality are of little concern in the fixed effects ANOVA, since ANOVA is fairly robust to the normal assumptions. Although Part 1 tests were performed in random order within oil groups, events, such as fuel change and equipment failures, could create correlation among data collected in the same time period. Fortunately, the extensive testing performed with the reference oil throughout the test program allows us to identify and make appropriate adjustments for significant trends that may have occurred in the testing and measurement processes.

To demonstrate model fit, the following plots were prepared:

- Normal probability plot of residuals
- Plot of residuals against test date
- Plot of residuals against predicted emissions.

In addition, individual data points that are statistically inconsistent with the underlying structure of the data set were identified based upon the standardized residuals from the statistical model fitting.

Section 3: Results

Results of the study are organized according to the study questions. First, Section 3.1 demonstrates that there are statistically significant differences in engine-out emissions that can be attributed to lubricant formulation and explores the interactions between the additive packages and the base oils in terms of their effects on engine-out emissions. Next, Section 3.2 presents the results of the mass balance analysis for metals contained in the lubricant additives, and Section 3.3 explores empirical relationships that may exist between engine-out emissions and lubricant properties. Finally, Section 3.4 presents findings from a special study (Phase 1, Part 2) to compare engine-out emissions resulting from different methods (fuel and oil doping or blending vs. exhaust injection) for accelerating oil consumption. These results were used to evaluate potential test procedures for the Phase 2 study of lubricant effects on the performance and durability of NO_x adsorber catalysts.

3.1 Effects of Additives and Base Oils on Engine-Out Emissions

Following the approach described in Section 2.2.7, a series of statistical analyses were performed to determine if there are statistically significant differences in engine-out emissions that can be attributed to (a) differences in overall oil properties or (b) differences in the formulations of additive packages or base oils. We began with a simple one-way analysis of variance model to compare emissions among all oils tested. For this analysis, every possible combination of additive and base oil was treated as a separate oil. Table 3.1-1 shows that there were statistically significant differences in the emissions of nearly every emission component when comparing across all oils tested. Next, two-way analysis of variance models were used to determine whether these differences can be attributed to constant effects of differences in the additives or base oils, or some unspecified interaction between them. This analysis was restricted to the additive packages a through f, which were tested in all four base oils.

Table 3.1-1 indicates that there are significant interactions between additive packages and base oils for six emission components and constant additive effects of both the additives and base oils for three components. This means that both the additive and base oils were found to have an impact on engine-out emissions for all of the target emission components.

Table 3.1-1. Summary of statistically significant effects of lubricant additives and base oils on diesel emissions.

Emissions Component	Differences Among Oils	Significant Interactions	Significant Additive Effect	Significant Base Oil Effect
HC	X	X		
CO	X		X	X
NO_x	X[a]	N/A	N/A	N/A
SO_2	X	X		
TPM	X		X	X
S	X		X	X
Ca	X	X		
Zn	X	X		
P	X	X		
Mo	X	X		

X   Indicates that the differences or effects are significantly different from zero at the 0.05 level of statistical significance.

(a) NO_x results available only for Group 2 base oil. Therefore, tests for interactions and main effects were not available (N/A).

Appendix E contains estimated average emissions of gases and particulate matter components (TPM, SOF, SO_4, and metals), along with 95% confidence limits, for each combination of additive package and base oil. The estimates and confidence bounds were based on the best fit model as described in Section 2.2.7. Data obtained on NO_x emissions during testing with Group 1, 3, and 4 base oils were not statistically analyzed due to data quality issues. Also, PAH data, which will serve as a baseline for a future assessment of toxic emissions from emission control systems, were not statistically analyzed. However, the PAH data are presented graphically in Appendix F. Results for gases, total particulate matter and selected components, and metals are discussed separately in the sections that follow.

3.1.1 Gaseous Emissions

Figure 3.1-1 shows the estimated emissions of HC, SO_2, and CO, along with 95% confidence limits for each combination of base oil and additive package tested. Also shown are the "interaction plots" which illustrate the degree to which emissions from particular combinations of base oils and additive packages deviate from the constant effects of each factor. Each line represents the results for a different base oil. If there were no interactions between base oils and additives, the lines would be parallel. However, as indicated in Table 3.1-1, there are statistically significant interactions between the additive packages and base oils in terms of emissions of HC and SO_2. HC emissions for oil d1 (additive "d" in group 1 base oil) and f4 appear to be lower than one would expect if no interaction were present. On the other hand, the interactions of additives and base oils on the emissions of SO_2 are more subtle. We see the significant effect of the high sulfur content of the Group 1 base oil; however, the magnitude of the effect is relatively small for additives "a," "c," and "f" and relatively high for oils "b" and "e." We also observe an unusually high level of SO_2 from oil i2; however, we cannot determine whether this effect is a constant effect of additive "i" or an interaction of additive "i" with the Group 2 base oil. Additive

"i" was only tested with Group 2 base oil. Because the initial result for oil i2 was so unusual, the test was repeated and the new result was found to be consistent with the original.

The interaction of additives and base oils on the emissions of CO is not statistically significant. However, there were differences in CO emissions associated with both additives and base oils. Multiple comparison analysis was performed to identify particular additives or base oils that behave differently from others. The analysis determined that additive "a" produces slightly higher CO emissions than additives "b" or "d." Also, Group 1 CO emissions are higher than those involving Groups 2 or 4.
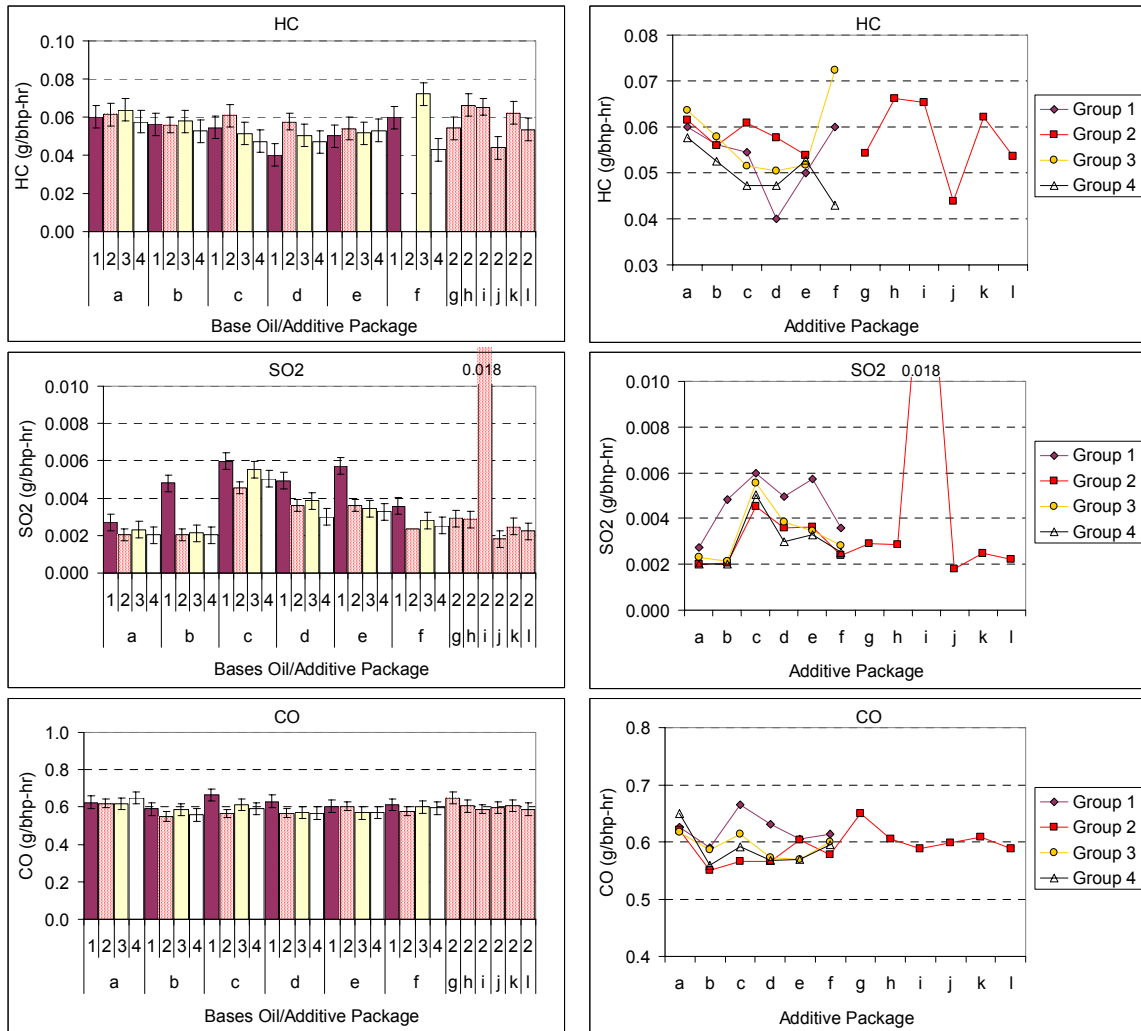
Figure 3.1-1.   Estimates of HC, SO$_2$, and CO emissions with 95% confidence intervals (left) and interaction plots (right).

Figure 3.1-2 shows the estimated emissions of NO$_x$ for the Group 2 oils. Confidence intervals are provided for each estimate. The NO$_x$ emissions for oils i2, j2, and l2 are slightly lower than those from a2, c2, e2, and f2. The nature of this apparent NO$_x$ reduction with certain lubricants is not well understood.
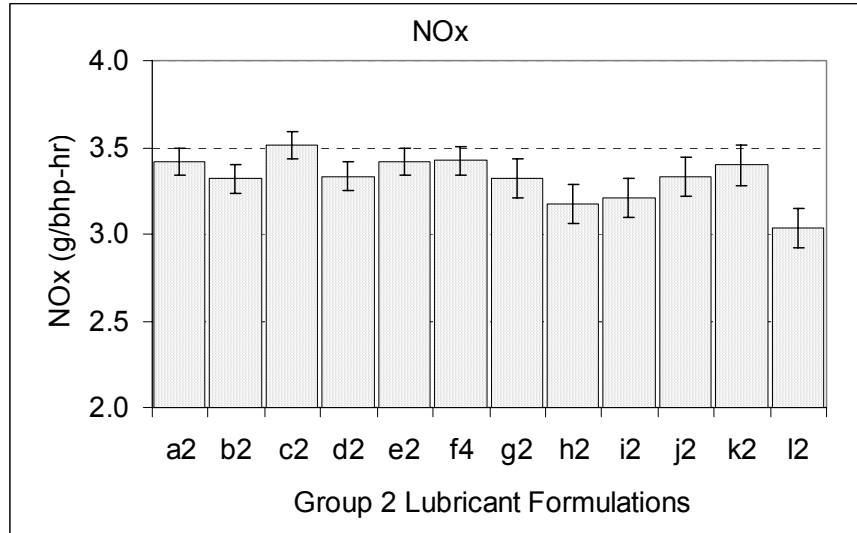
Figure 3.1-2. Estimates of additive and base oil effects on NO$_x$ emissions with 95% confidence intervals.

3.1.2 Particulate Matter Emissions

Figure 3.1-3 shows the estimated PM emissions with 95% confidence intervals for each additive package in the Group 2 base oil and average emissions for packages "a" through "f" in each of the four base oil groups. The analysis of variance determined that there were statistically significant main effects of the additives and base oils, but no interactions. Multiple comparison analysis revealed that Group 1 oils had higher PM emissions than Group 2. No other pairwise comparisons of additives or base oils were found to be statistically significant.

SOF and SO$_4$ components of the PM emissions are shown in Figure 3.1-4. Depending on the base oil additive package used, the contribution of SOF to overall PM emissions ranges from 0 to 57 percent, and sulfates represent less than 10 percent of the overall PM emissions.
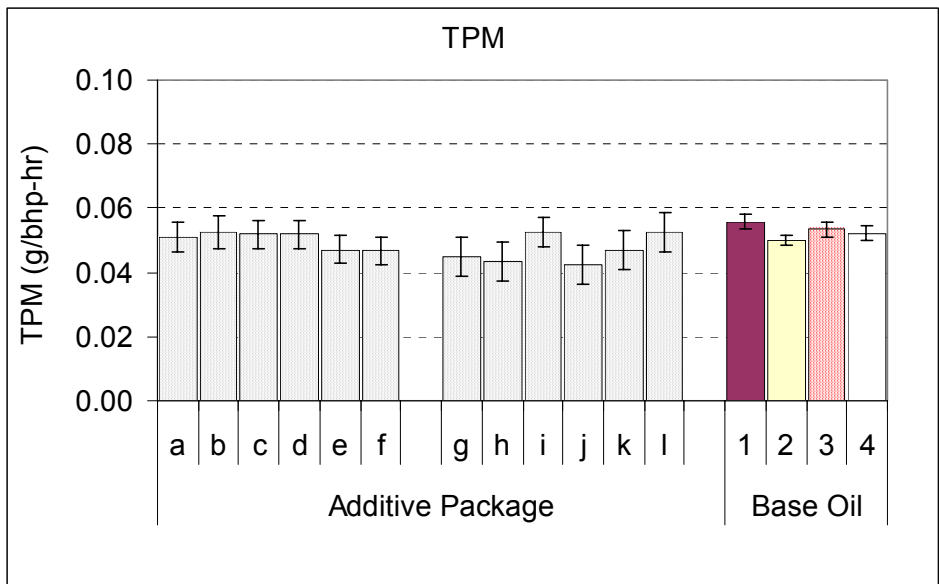
Figure 3.1-3. Estimates of additive and base oil effects on TPM emissions with 95% confidence intervals. (Additive effects are presented as estimated emissions with Group 2 base oil.)
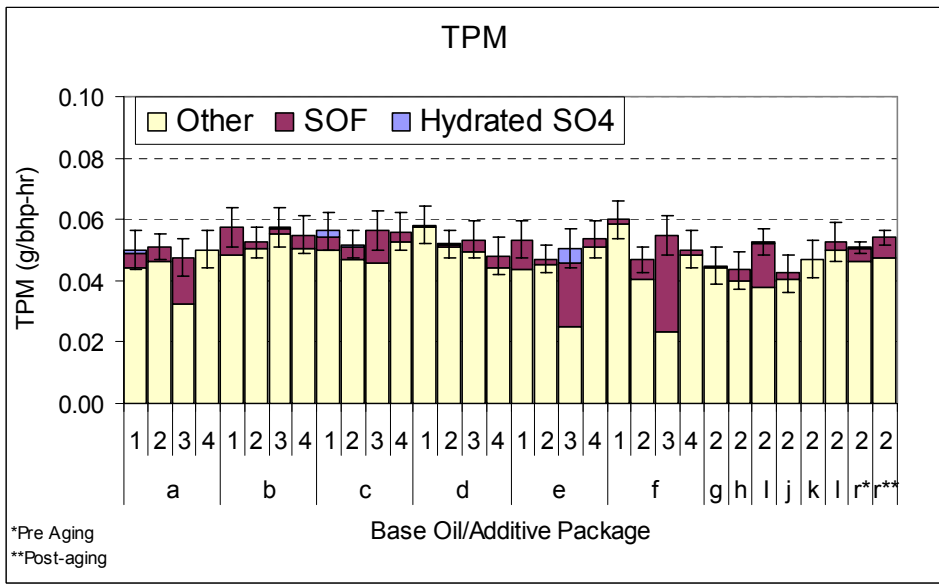
Figure 3.1-4. Average TPM emissions with 95% confidence intervals – showing breakdown of SOF and SO_4 components.

3.1.3 PM Metal Emissions

Figure 3.1-5 shows estimated emissions and interaction plots for four PM-bound metals of interest. The statistical analysis found significant effects of base oils and interactions between base oils and additives for Ca, Zn, and P. However, the interaction plots illustrate how the additive package, as expected, plays a dominant role in determining emissions of these metals. The interaction effect of additives and base oils was not statistically significant for sulfur. Multiple comparison analysis, performed on sulfur emissions from packages "a" though "f" tested in all four base oils, found that additive "c" produces significantly higher S emissions compared to packages "a" and "b." Also, the Group 4 base oil produced lower S emissions than Groups 1 or 2. Among all additive packages tested, oil i2 (additive i in base oil 2) produced significantly higher S emissions than any other oil. Recall that oil i2 also produced unusually high levels of SO_2 (See Figure 3.1-1.).

3.2 Mass Balance Analysis

The mass balance analysis for metals was performed to determine the degree to which the emissions of metals can be predicted by known concentrations of the metals in the test oils and fuel as well as oil and fuel consumption rates. Key statistics used in the mass balance analysis include the estimated oil consumption rate (179.3 mg/bhp-hr - gravimetric), the measured fuel consumption rate (0.40 to 0.41 lbs/bhp-hr – measured fuel flow), and the estimated sulfur level in the fuel (first batch: 4.5 ppm, fuel blend after second batch had been added: 1.0 ppm). Details of the statistical analysis approach were presented in Section 2.2.7.

Figure 3.2-1 compares the measured and calculated emissions of six target metals contained in the additives, base oils, and fuel. Each symbol represents a different finished oil, according to the accompanying key. A regression line was fit to the data in order to determine the degree of correlation and to estimate the recovery rate. If the oil and fuel represent the only sources of a metal and the relationship between measured and calculated emissions is linear, then the intercept of the regression line (the estimated measured value at a calculated value of zero) should be zero and the slope of the regression line can be used to estimate the constant recovery rate. However, if there are other sources of the metal, or the relationship between measured and calculated emissions is nonlinear, the recovery rate will vary with emissions levels. Using regression analysis, we first determined whether or not the estimated intercept was statistically different from zero. If not, a new regression model was fit without an intercept. The intercept was found to be different from zero for three metals: Zn, Mo, and Mg. In those cases, the recovery is estimated by taking the ratio of the predicted (regression line) and calculated emissions at the maximum calculated emissions level.
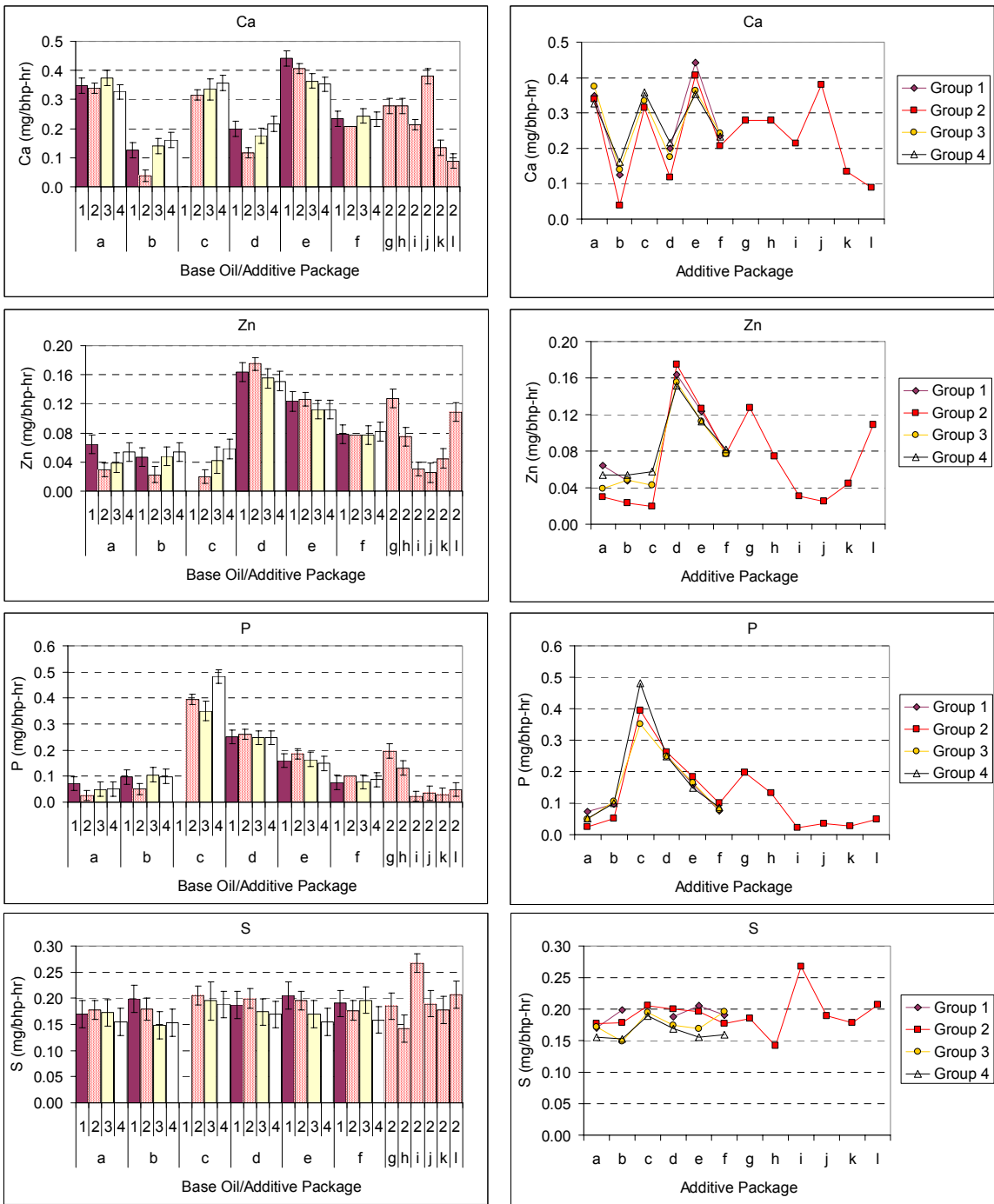
Figure 3.1-5. Estimates of Ca, Zn, P, and S emissions with 95% confidence intervals (left) and interaction plots (right).

Figure 3.2-1. *Mass balance comparisons of S, Ca, Zn, P, Mo and Mg with estimated recovery regression line and 95% confidence intervals.*
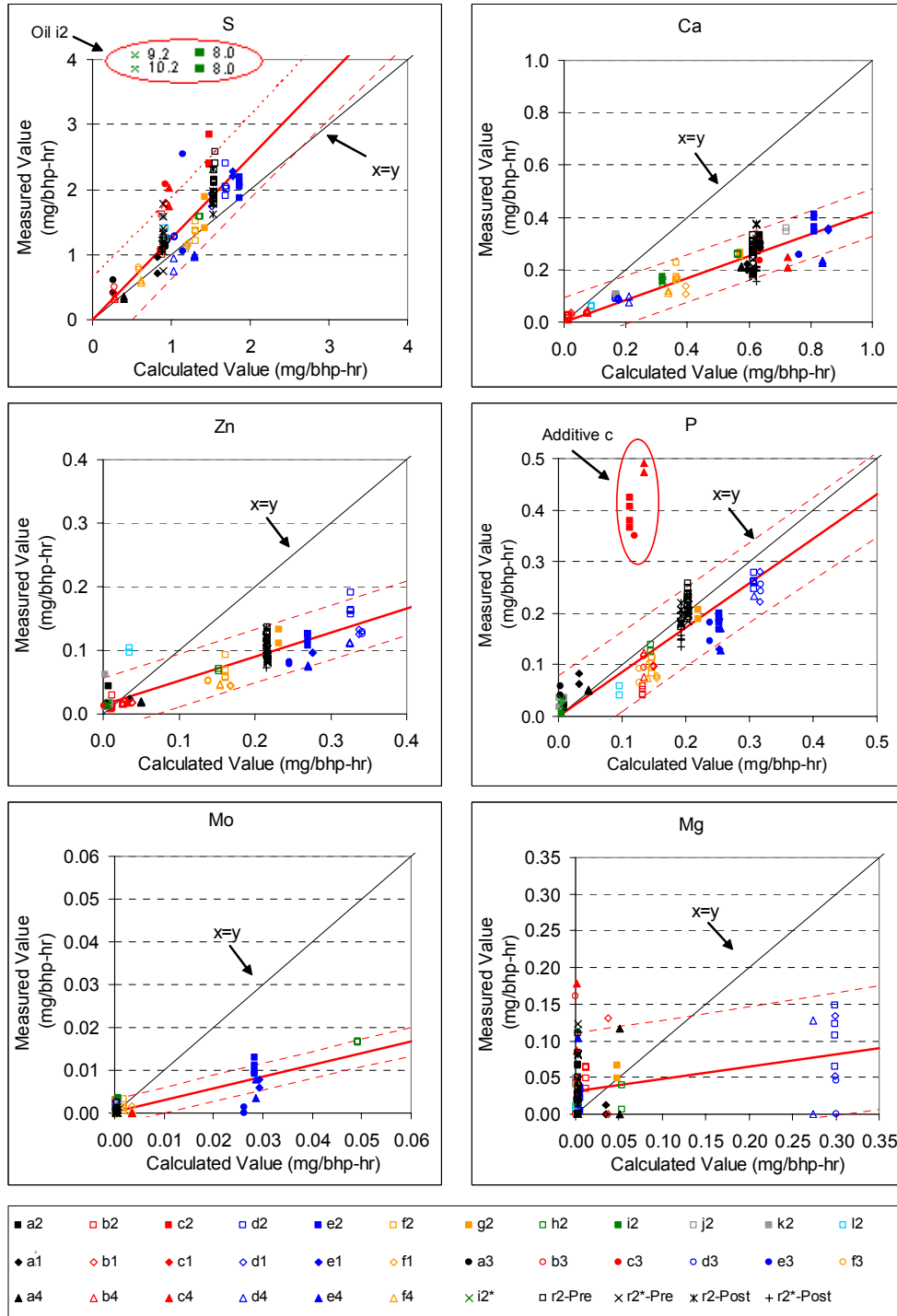
Notes:
1. a2 (for example) refers to additive "a" in the Group 2 base oil.
2. r2-Pre and r2-Post refer to reference oil before and after 40-hour aging cycle.
3. Group 2 oil tests were performed with 4.5 ppm or 1.0 ppm sulfur fuel. Those performed with 1.0 ppm sulfur fuel were indicated by *. All groups 1, 3 and 4 oil tests were performed with 1.0 ppm sulfur fuel.

As shown in Table 3.2-1, the recovery rates estimated by the slope of the regression lines vary considerably among the target metals – ranging from 27% for Mg to 127% for sulfur. 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 likely not the case and the recovery rate therefore is indicative of the actual composition of the consumed lube oil. Several factors ultimately determine the fate of a given species: volatility, surface activity, and tendency to break down at elevated temperatures. However, it was beyond the scope of this study to investigate the specific fate of the various elements. The apparent recovery rate for sulfur is significant because it is perhaps the most scrutinized of the potential catalyst poisons, especially with respect to NO_x adsorber catalysts. It is important to note, though, that the nature of the sulfur compounds emitted is more important than the fact that they are emitted.

The mass balance calculations for most metals were fairly straightforward because it was only necessary to consider the quantity of the metals contained in the test oil. However sulfur is a constituent of the fuel as well as the oil and was present in three different emissions components: SO_2, SO_4, and elemental sulfur in the PM. Figure 3.2-2 shows the sources of calculated and measured sulfur emissions for each test oil that was used in the mass balance analysis. Concentrations of sulfur in the fuel and oil, as well as estimated fuel and oil consumption rates, were used to determine the "calculated" sulfur emissions. The "measured" sulfur emissions were determined from measurements of the three constituents containing sulfur.

Table 3.2-1.   Mass balance recovery rates for target elements – with 95% confidence intervals.

Element	Recovery Rate
S	127% (122%, 132%)
Ca	42% (40%, 43%)
Zn	42% (30%, 55%) @ 0.34 mg/bhp-hr
P	86% (82%, 90%)
Mo	28% (21%, 35%) @ 0.05 mg/bhp-hr
Mg	27% (0%, 55%) @ 0.3 mg/bhp-hr

Note: Group 2 oil tests were performed with 4.5 ppm or 1.0 ppm sulfur fuel. Those performed with 1.0 ppm sulfur fuel were indicated by *. All groups 1, 3 and 4 oil tests were performed with 1.0 ppm sulfur fuel.

Figure 3.2-2.   Sources of S for mass balance comparison of calculated and measured emissions of sulfur.

3.3 Relationships Between Engine-Out Emissions and Oil Properties

The mass balance analysis in the previous section established statistical relationships between the emissions of select metals and the known concent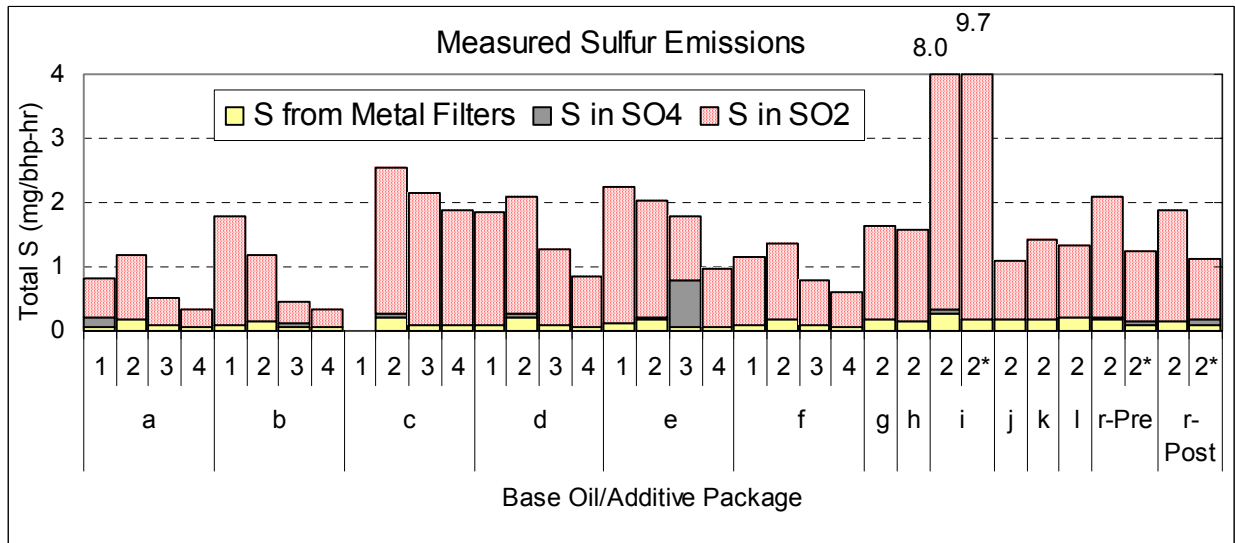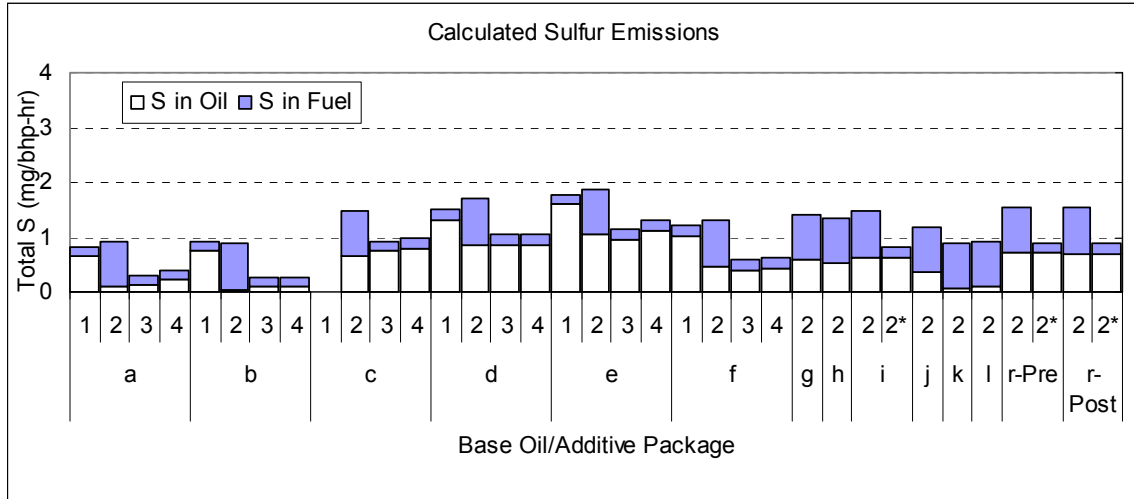rations of those metals in the lubricants and fuel. To further investigate other relationships between emissions and oil properties, more exploratory methods were employed.

We began by identifying the compositional elements and properties of the test oils that were measured in the used test oils and that spanned a meaningful range of values. Elements found at trace levels (< 10 ppm) in the test oils were excluded from this analysis. Also, measured properties such as viscosity, which (by design) did not vary much among the test oils, were excluded. The elements and properties of the test oils included in the analysis were ash (%); S, Ca, Zn, P, B, Mo, Mg (ppm); and total base number (TBN). We also used principal component analysis to help characterize the key features (i.e., principal components) of the test oils. We then modeled PM and gaseous emissions as a function of the principal components. Recall that principal component analysis was used in the experimental design to select the representative sample of oils (See Section 2.1.3 and Appendix A). At the design stage, principal component analysis was performed on *target* properties for additives in a Group 2 base oil, whereas, in this case, the analysis is based on the average *measured* properties of all 32 test oils (12 from Group 2; six each from Groups 1, 3, and 4; and the reference oil – before and after the change in fuels).

Table 3.3-1 displays the principal component coefficients applied to the elements and properties of the test oils. Ash was not included in the principal component analysis because it is already correlated with the levels of Ca, Zn, Mo, and Mg. Coefficients greater than 0.3 or less than -0.3 are highlighted to help "define" the principal components. For example, PC1, which explains 43% of the variability in the elements and properties, is highly correlated with the amount of ZDDP contained in the oil. PC2 appears to be related to the detergent level of the lubricant.

Stepwise regression analysis was used to identify which combinations of the compositional elements and properties (or principal components) could be used to predict emissions of PM and selected gaseous emissions (HC, CO, NO_x, and SO_2). There are many ways to define the "best" fit model using stepwise regression. A "maximum R^2" method was employed here to select the best 1-, 2-, and 3-variable models. In addition, we applied the "backward selection" method to the best fitting model that contains only statistically significant variables. The rationale for using these methods was discussed in Section 2.2.7.

Table 3.3-1. *Principal component coefficients for measured oil properties - and contributions to total variance.*

Element/ Property	PC1	PC2	PC3	PC4	PC5	PC6	PC7	PC8
S	0.462	0.056	-0.166	-0.419	-0.084	0.753	-0.081	-0.003
Ca	0.188	0.623	-0.055	-0.254	0.166	-0.277	0.156	0.617
Mg	0.296	-0.404	0.449	0.010	-0.572	-0.102	0.065	0.453
Zn	0.484	-0.180	0.093	0.186	0.472	-0.178	-0.655	0.077
P	0.464	-0.292	-0.067	0.072	0.372	-0.120	0.716	-0.158
Mo	0.277	0.303	-0.344	0.769	-0.321	0.127	0.029	0.031
B	-0.117	0.256	0.720	0.312	0.305	0.434	0.141	0.048
TBN	0.351	0.414	0.343	-0.177	-0.283	-0.308	-0.049	-0.616
Cumulative % of Total Variance	43%	69%	86%	93%	97%	99%	100%	100%
Description	S+Zn+P+TBN	Ca-Mg+Mo+TBN	Mg-Mo+B+TBN	-S+Mo+B	-Mg+Zn+P-Mo+B	S+B-TBN	-Zn+P	Ca+Mg-TBN

Table 3.3-2 defines the best fitting models according to the criteria described above. The first four models for each emission parameter are based on the nine elements and properties that were measured in the used test oil. The next four models describe the emissions as a function of the principal components. The p-values represent the level of statistical significance of the model for explaining the variability of the emissions parameter.

With the exception of models involving SO_2, we do not see many strong relationships between the emissions of PM, HC, CO, and NO_x and the properties of the test oils. (Note, the maximum R^2 for these models is less than 30%.) This is consistent with the findings in Section 3.1 which showed that these emissions do not vary significantly among the oils tested.

It is not surprising that the level of sulfur in the oil is the best predictor of SO_2 emissions ($R^2 = $ 59%). The prediction equation is:

$$SO_2(\text{g/bhp-hr}) = 0.002 + 4.15 \times 10^{-7} \times S(\text{ppm}).$$

The analysis also suggests that the levels of Zn and Mo can help improve the prediction of sulfur emissions. Notice that the correlation between the observed and predicted SO_2 emissions increases from 59% to 71% when Zn is included in the prediction model. Adding Mo to the model, in addition to Zn, increases the correlation to 75%. Initially, this analysis was performed using data from all test oils. However, the unusually high SO_2 emissions associated with test oil "i2" resulted in a very low correlation between SO_2 emissions and oil properties. Therefore, the data from tests performed with oil "i2" were removed from the analysis in order to characterize

the relationship for other, more traditional, oils. Figure 3.3-1 illustrates how the prediction of SO_2 emissions is improved by including Zn or Zn and Mo in the prediction model.

In particular, the predicted emissions from oil "c," which contains no Zn, is more consistent with the observed emissions when Zn is included in the model. The prediction equation for the two-variable model containing S and Zn is:

$$SO_2(\text{g/bhp-hr}) = 0.002 + 5.85 \times 10^{-7} \times S(\text{ppm}) - 7.77 \times 10^{-7} \times Zn(\text{ppm}).$$

The equation demonstrates that, at a fixed oil sulfur level, higher levels of Zn are associated with lower SO_2 emissions. The analysis also suggests that the Mo will further improve the prediction model. The prediction equation for a three-variable model containing S, Zn, and Mo is:

$$SO_2(\text{g/bhp-hr}) = 0.002 + 6.29 \times 10^{-7} \times S(\text{ppm}) - 7.81 \times 10^{-7} \times Zn(\text{ppm}) - 38.3 \times 10^{-7} \times Mo(\text{ppm}).$$

All three factors in this model are statistically significant. The model indicates that, at a fixed level of sulfur and Zn in the oil, higher levels of Mo are associated with lower SO_2 emissions. As shown in the "Best 3 Variables" plot in Figure 3.3-1, the predicted emissions from oil "e," which contains medium level of Mo, are more consistent with the observed emissions when Mo is included in the model.

These expressions offer additional evidence that the *source* of sulfur in the lubricant has an impact on the resultant emissions of SO_2. It appears that, when oils have about the same level of total sulfur, but have varied levels of Zn, the emissions of SO_2 are lower for oils with higher Zn 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_2 in the exhaust. This also suggests that chemical limits may need to include additional factors to maximize the ability to predict SO_2 emissions based on the oil analysis.

Table 3.3-2.   Best fitting models for predicting TPM and gaseous emissions from oil properties.

Emissions (g/bhp-hr)	Model	R²	Elements/Properties Selected						value[1]
			1	2	3	4	5	6	
Total PM	Best 1 Variable[2]	0.0543	TBN						0.0139
	Best 2 Variables		S	TBN					0.0000
		0.2154	Mo	S	TBN				0.0000
	All Significant Variables	0.2154	Mo		TBN				0.0000
	Best 1 PC[3]	0.0597	PC4						
	Best 2 PCs	0.1429	PC4	PC6					0.0002
	Best 3 PCs	0.2010		PC4	PC6				0.0000
	All Significant PCs	0.2010	PC2	PC4	PC6				0.0000
w/o oil "i"	Best 1 Variable	0.5923	S						0.0000
	Best 2 Variables	0.7083	S	Zn					0.0000
	Best 3 Variables	0.7482	Mo	S	Zn				0.0000
	All Significant Variables	0.7890		Mo	P	S	Zn		0.0000
	Best 1 PC	0.2796	PC4						0.0000
	Best 2 PCs	0.5062	PC1	PC4					0.0000
	Best 3 PCs	0.6529	PC1	PC4	PC6				0.0000
		0.7893	PC1	PC3	PC4	PC5		PC7	0.0000
NO$_x$	Best 1 Variable	0.0536	TBN						0.0832
	Best 2 Variables	0.1223	ASH	Zn					
	Best 3 Variables	0.1808	P	TBN	Zn				0.0137
		0.1352	ASH	Ca	Mg				0.0511
	Best 1 PC	0.1196	PC7						0.0084
	Best 2 PCs	0.1604	PC2	PC7					0.0089
	Best 3 PCs		PC2	PC4	PC7				0.0105
	All Significant PCs	0.1196	PC7						0.0084
	Best 1 Variable	0.1187	P						0.0005
	Best 2 Variables	0.1537	P	Zn					0.0004
	Best 3 Variables	0.1803	P	TBN	Zn				0.0003
	All Significant Variables	0.1991	ASH	Ca	Mg	Zn			0.0003
	Best 1 PC	0.0900	PC1						0.0027
	Best 2 PCs	0.1436	PC1	PC7					0.0006
	Best 3 PCs	0.1750	PC1	PC4	PC7				0.0004
	All Significant PCs	0.1436	PC1	PC7					0.0006
CO	Best 1 Variable	0.1413	B						0.0000
	Best 2 Variables	0.2097	B	Ca					0.0000
	Best 3 Variables	0.2601	B	Mg	S				0.0000
	All Significant Variables	0.2652	ASH	B	Ca	S			0.0000
	Best 1 PC	0.1333	PC2						0.0001
	Best 2 PCs	0.2053	PC2	PC3					0.0000
	Best 3 PCs	0.2404	PC2	PC3	PC6				0.0000
	All Significant PCs	0.2703	PC2	PC3	PC4	PC6			0.0000

(1) p-values represent the level of statistical significance of the model for explaining the variability of the emissions parameter.
(2) Candidate variables are ash (%); S, Ca, Mg, Zn, P, Mo, B, and TBN.
(3) See Table 3.3-1 for definitions of principal components.
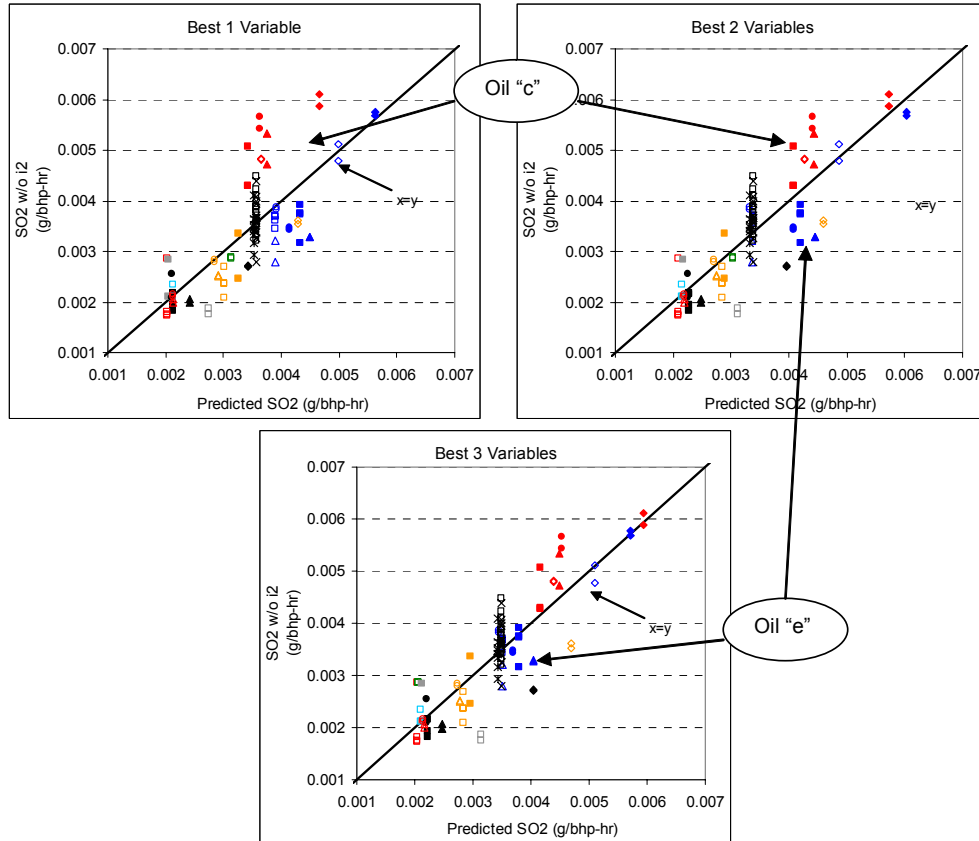
Figure 3.3-1. *Comparison of best one-variable (SO_2 vs. S), two-variable (SO_2 vs. S and Zn) and three-variable (SO_2 vs. S, Zn and Mo) predictions of SO_2 emissions.*

To further investigate the effects of Zn and Mo on SO_2 emissions, we divided the test oils into clusters according to their concentrations of Zn and Mo, then applied multiple regression analysis to compare the rates of SO_2 emissions per unit increase in oil sulfur levels among the clusters.

A scatter plot of the Zn and Mo levels for the test oils is shown in Figure 3.3-2. The plot demonstrates that the test oils fall into five distinct clusters:

Cluster #1: Low Zn/Low Mo – containing oils "a", "b", "c", "j", "k", and "l"
Cluster #2: Med Zn/Low Mo – containing oils "f", "g" and "r"
Cluster #3: High Zn/Low Mo – containing oil "d"
Cluster #4: High Zn/Med Mo – containing oil "e"
Cluster #5: Med Zn/High Mo – containing oil "h."

The regression model contains a common intercept and separate slopes for each cluster. The model for the ith cluster is

$$SO_2(g/bhp\text{-}hr) = \beta_o + \beta_i \times S(ppm) + e_i,$$

where β_o = the common intercept, β_i = the slope for i^{th} cluster, and e_i = random error.

The slope for each cluster represents the relative increase in SO_2 emissions per unit increase in oil sulfur level (i.e., grams of SO_2/bhp-hr per ppm of oil sulfur). The estimated intercept for the model is 0.00165 and the R^2 correlation is 76%. The estimated slopes for the individual clusters, shown in Figure 3.3-2, range from 3.693×10^{-7} to 6.985×10^{-7}. Multiple comparisons were performed to determine whether there are statistically significant differences in the slopes among the five clusters. The results can be summarized as follows:

1. The slope for the "Low Zn/Low Mo" cluster is significantly higher than that for any of the other four clusters,
2. The slope for cluster 2 ("Med Zn/Low Mo") is significantly higher than that for cluster 4 ("Med Zn/Med Mo"), and
3. Slopes for clusters 3, 4, and 5 are not significantly different.

Thus, at a fixed oil sulfur level, oils with Med Zn/Med Mo," "High Zn/Low Mo," or "Med Zn/High Mo" content are associated with lower SO_2 emissions. This implies that the source of sulfur has a significant effect on the SO_2 emissions. Although the general conclusion that higher levels of both Zn and Mo are associated with lower SO_2 emissions (for a fixed oil sulfur level) is supported by the data, additional testing with oils containing a broader range of Zn and Mo concentrations is needed to substantiate this finding.
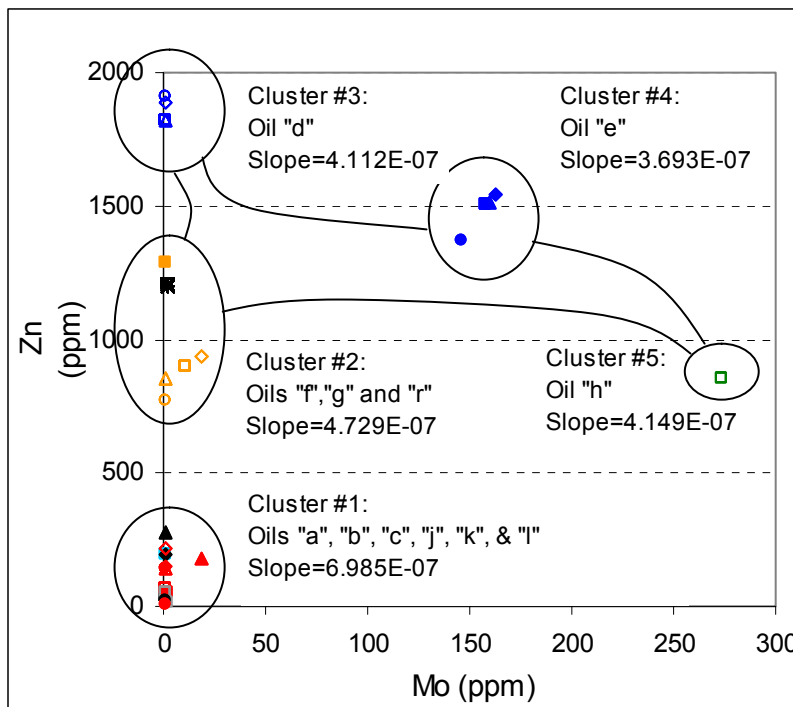

Figure 3.3-2.   *Scatter plot of Zn and Mo levels in test oils. The slope represents the rate of increase in SO_2 emissions per unit increase in oil sulfur levels (g SO_2/bhp-hr per ppm oil sulfur). Rates for connected clusters are not significantly different.*

3.4 Comparison of Oil Consumption Acceleration Methods

Following the Part 2 experimental design presented in Table 2.1-6 (Section 2.1.7), three test oils formulated with additives i, c, and r in the Group 2 base oil (thus, designated i2, c2, and r2) were tested at two, four, and eight times the engine's inherent oil consumption rate. Two different oil consumption acceleration methods were used: blending oil directly in the fuel and injecting oil under pressure into the exhaust manifold (Figure 3.4-1). In addition, tests were performed using a combination of the two methods with the reference oil, "r2," to study the interaction effects of the methods. The purpose of the study was to understand whether a validated approach to accelerate oil consumption rate could be developed. If demonstrated, it would provide a means for conducting rapid aging tests intended to investigate how consumed lube oil impacts the performance of emission control catalysts (Phase 2).

Two statistical analysis approaches were designed to address four study questions related to how emissions are affected by oil type, oil consumption acceleration rates, and acceleration methods. One approach models total particulate and gaseous emissions as a function of the oil consumption acceleration rate for each type of oil and acceleration method. The results of this analysis are presented in Section 3.4.1. We used a similar model to analyze metals emissions, except the measured emissions were compared to calculated emissions using mass balance methods. The latter results are presented in Section 3.4.2.

3.4.1 Total PM and Gaseous Emissions

Figure 3.4-2 illustrates the regression model that was fit to the PM and gaseous emissions data for each oil type (See Section 2.2.7). The model fits a different slope for each acceleration method, but assumes a common intercept at the baseline oil consumption rate (ratio = 1). For SO_2, the estimated intercept compares favorably to the Part 1 emissions data, which were collected under the baseline oil consumption conditions. The Part 1 data were not used in this regression analysis.
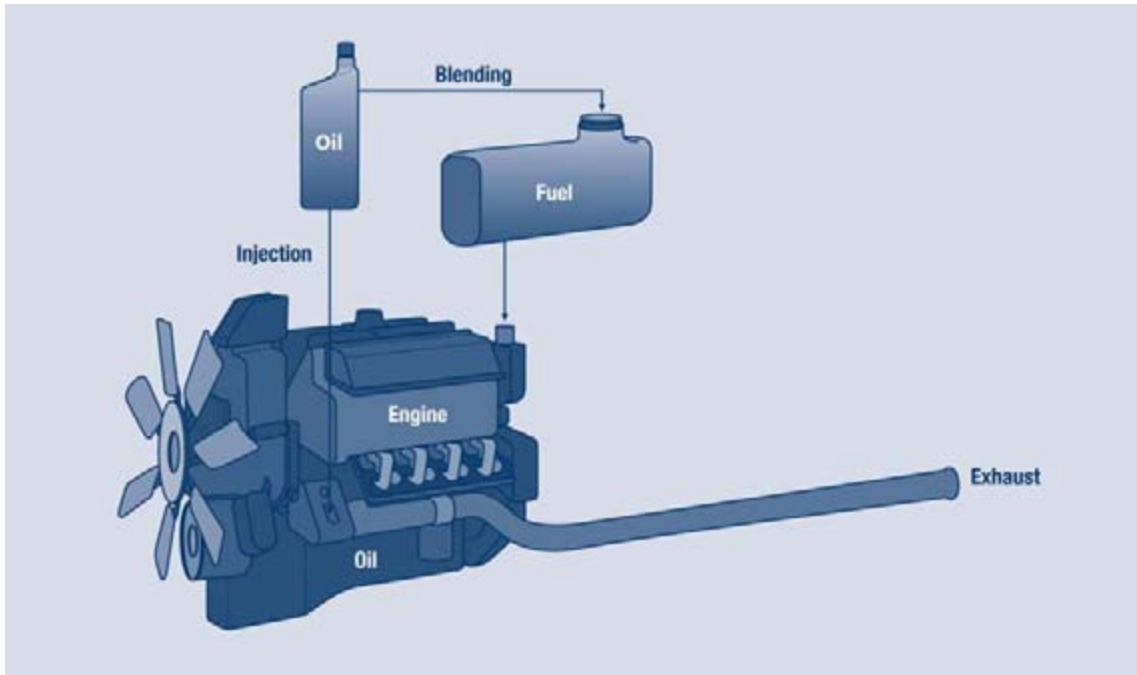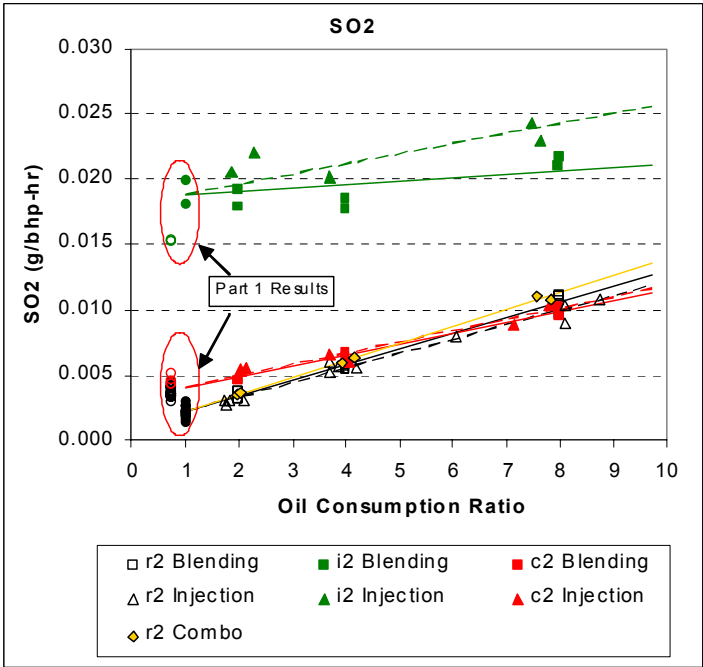
Figure 3.4-1.   Oil consumption acceleration methods (blend oil with fuel and/or inject oil into exhaust).

NOTE: Part 1 results are shown for reference – but not included in the regression analysis.

Figure 3.4-2.   Measured SO_2 emissions versus oil consumption ratio using fuel/oil blending and oil injection methods to accelerate oil consumption.

Similar analyses were performed for all target emissions. Figures 3.4-3 through 3.4-7 display the predicted emissions of PM and gaseous emissions, along with 95% confidence limits, at various oil consumption rates using each oil consumption acceleration method. Clearly, the oil type, acceleration rate, and acceleration method have significant impacts on emissions of SO_2, TPM, HC, and CO. The impacts on NO_x are minimal.

The increase in SO_2 emission rate follows the acceleration rate for all three approaches, but only for oils r2 and c2. All three approaches underpredict oil i2's tendency to increase SO_2 emissions. For PM, only the injection method (and to some extent the combination method) yields an increase in PM emissions that correlates with the rate of acceleration for all three oils. The same is true for HC and CO, as most of the lubricant is emitted in an unburned or partially burned state. No impact on NO_x was seen or expected with any of the approaches.

Tables 3.4-1 through 3.4-3 summarize the statistical findings associated with the study questions. For example, Table 3.4-1 shows how emissions change as a function of oil consumption rate for each oil type and acceleration method (Study question 2.1). It displays the estimated relative increases in total PM and gaseous emissions resulting from a 100% increase in oil consumption for each combination of test oil and acceleration method. For example, doubling the total oil consumption by injecting oil in the exhaust stream will increase HC emissions by 170% to 189%, depending on the type of oil. On the other hand, if the same amount of additional oil were consumed by blending oil with the fuel, the increase in HC would be no more than 14%. Except where indicated, all estimates were determined to be statistically different from zero. Most emissions are affected by increased oil consumption, but clearly, the effect is dependent on oil type and acceleration method.

Table 3.4-2 compares oil types in terms of baseline emissions levels and the rates of increase in emissions using each acceleration method. Consistent with the findings from Part 1 tests, there are significant differences in baseline SO_2 emissions among the three oils. Oil i2 produces the highest SO_2 emissions, followed by oil c2. However, oil i2 produces the smallest rate of increase in SO_2 emissions (< 5% versus 20 to 54%). Also, when oil i2 is injected into the exhaust, the rate of increase in TPM emissions is smaller than the rates of increase with oils c2 or r2 (33% increase versus 42 to 44% increase). Clearly, the mechanism by which oil i2 yielded an increase in SO_2 could not be mimicked or accelerated by either of the approaches utilized. This is viewed as a significant finding because it would not adequately predict the effects of an oil that behaved like i2.

Finally, Table 3.4-3 compares the two acceleration methods in terms of the rates of increase in emissions with higher levels of oil consumption using each type of oil. Compared to blending, the injection method produces a greater increase in all species of emissions for at least one of the oils. Furthermore, when the methods are combined, the rate of increase in CO emissions is more consistent with the rate of increase observed with injection. With the combination method, the NO_x and SO_2 emissions rate increases were also statistically higher that the average rate of increases; however, the magnitudes of these differences were not meaningful.
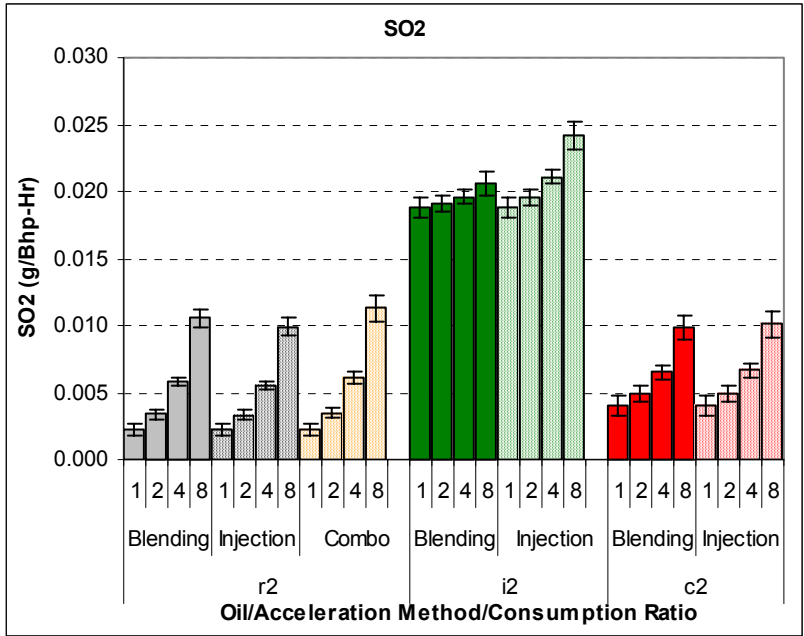
Figure 3.4-3. Predicted emissions of SO_2 at 1, 2, 4, and 8 times the baseline oil consumption rates using blending, injection, and a combination acceleration method.
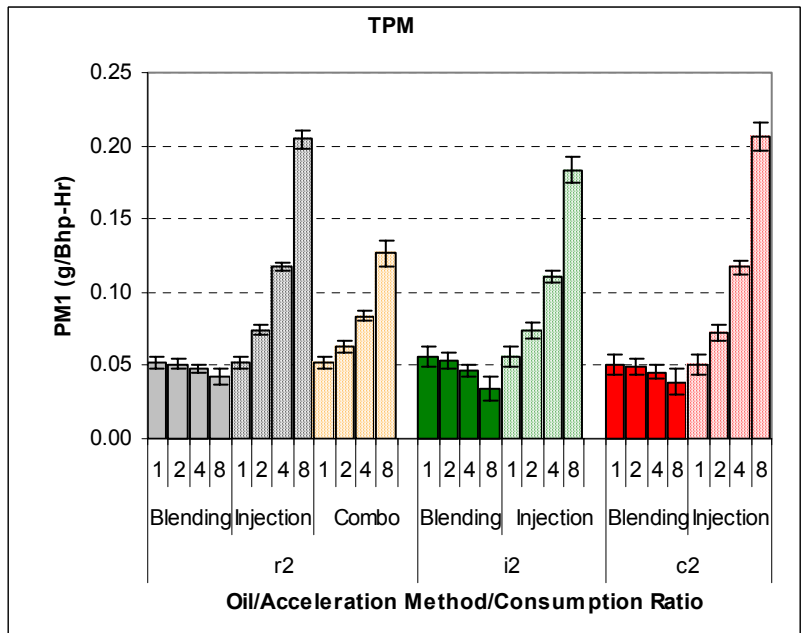
Figure 3.4-4. Predicted emissions of TPM at 1, 2, 4, and 8 times the baseline oil consumption rates using blending, injection, and a combination acceleration method.
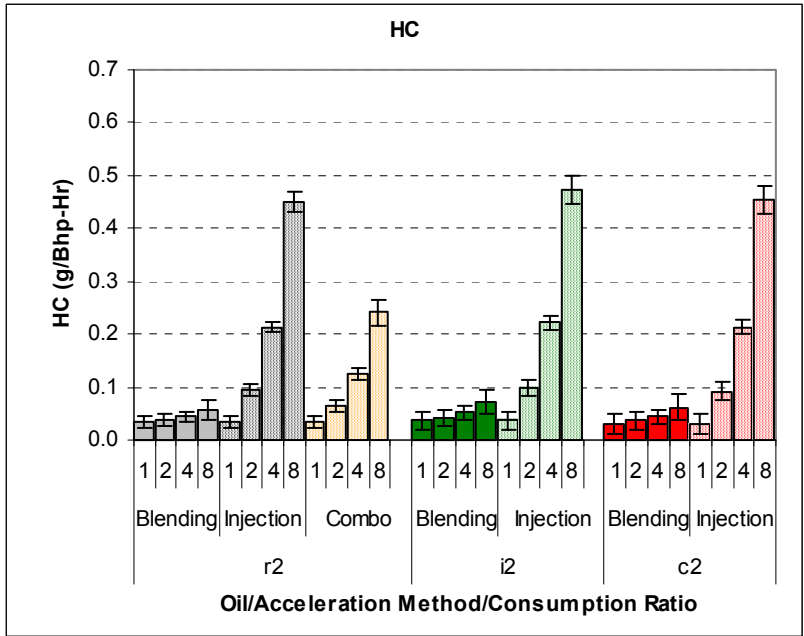
Figure 3.4-5. Predicted emissions of HC at 1, 2, 4, and 8 times the baseline oil consumption rates using blending, injection, and a combination acceleration method.
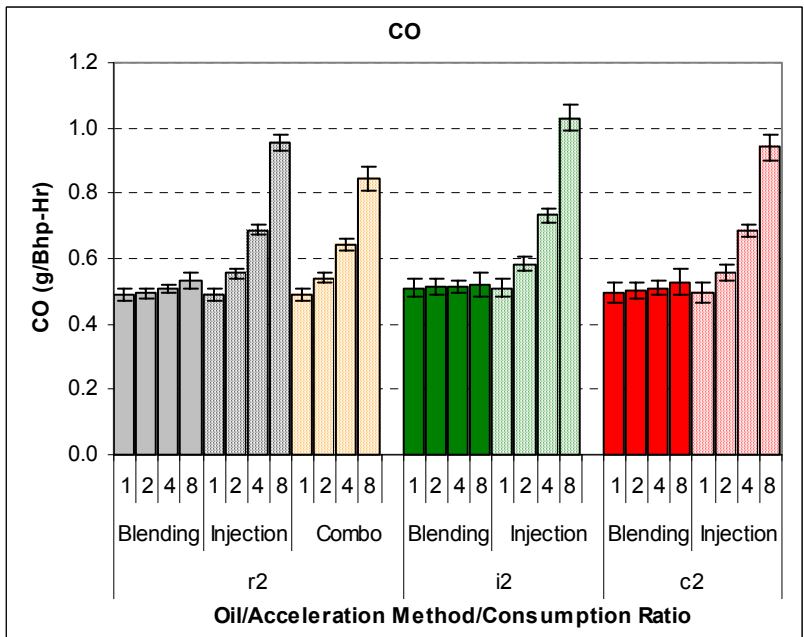
Figure 3.4-6. Predicted emissions of CO at 1, 2, 4, and 8 times the baseline oil consumption rates using blending, injection, and a combination acceleration method.
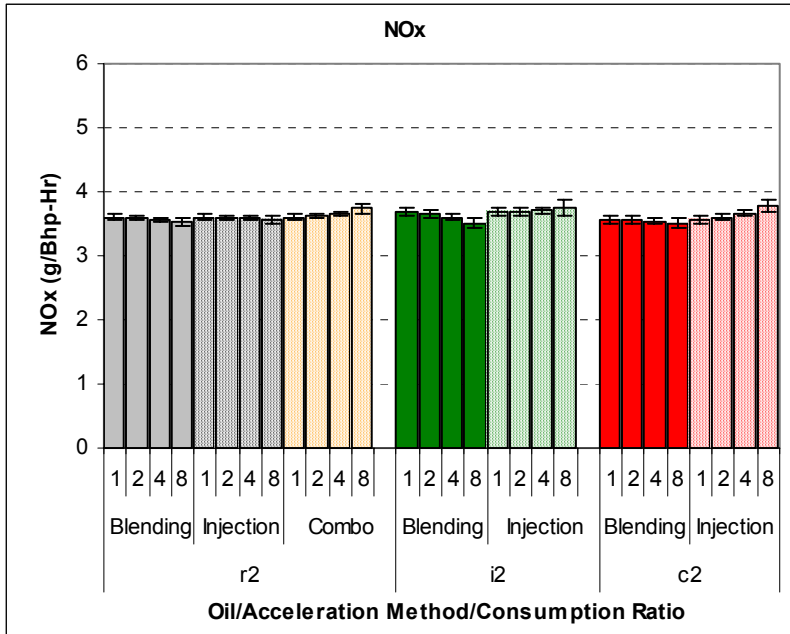
Figure 3.4-7. Predicted emissions of NO_x at 1, 2, 4, and 8 times the baseline oil consumption rates using blending, injection, and a combination acceleration method.

Table 3.4-1. Estimated relative increase in emissions resulting from a 100% increase in oil consumption rate – by acceleration method and oil type.

Acceleration Method	Oil	TPM	HC	CO	NO_x	SO_2
Blending	r2	-3%	-[1]	1%	-	54%
	i2	-6%	14%	-	-1%	1%
	c2	-	-	-	-	20%
Injection	r2	42%	170%	14%	-	50%
	i2	33%	173%	15%	-	4%
	c2	44%	189%	13%	1%	21%
Combination	r2	20%	84%	10%	1%	59%

(1) Estimated increase in emissions was not statistically significant

Table 3.4-2. *Statistically significant differences in baseline emissions (normal oil consumption)*
and rates of increase in emissions with two oil consumption acceleration methods.

Comparison	Oils Compared	TPM	HC	CO	NO$_x$	SO$_2$
Baseline Emissions (at 1X)	i2 vs. r2	-[1]	-	-	-	i2 > r2
	c2 vs. r2	-	-	-	-	c2 > r2
	c2 vs. i2	-	-	-	i2 > c2	i2 > c2
Rates of Increase in Emissions with Blending	i2 vs. r2	-	-	-	-	r2 > i2
	c2 vs. r2	-	-	-	-	r2 > c2
	c2 vs. i2	-	-	-	-	c2 > i2
Rates of Increase in Emissions with Injection	i2 vs. r2	r2 > i2	-	-	-	r2 > i2
	c2 vs. r2	-	-	-	c2 > r2	-
	c2 vs. i2	c2 > i2	-	-	-	-

(1)   Difference in estimated baseline emissions or rate of increase among oils compared is not statistically significant.

Table 3.4-3. *Statistically significant differences in the rates of increase in emissions between*
blending and injection oil consumption acceleration methods.

Comparison	Oil	TPM	HC	CO	NO$_x$	SO$_2$
Rates of Increase in Emissions using Blending vs. Injection	r2	I>B	I>B	I>B	-[1]	-
	i2	I>B	I>B	I>B	I>B	I>B
	c2	I>B	I>B	I>B	I>B	-
Rates of Increase in Emissions using Combination Method vs. Average Rate of Increase	r2	-	-	C > (B+I)/2	C > (B+I)/2	C > (B+I)/2

(1)   Difference in estimated rates of increase is not statistically significant.

3.4.2 Metals

A mass balance approach was used to analyze the effects of oil type, oil consumption rates, and oil consumption acceleration methods on metals emissions. We combined measured concentrations of Ca, S, P, and Zn in used test oils, as well as measured levels of S in the test fuel, with estimated oil and fuel consumption rates to calculate the expected emissions of each element. Measured emissions of these metals were compared with the calculated emissions using a regression model, as described in Section 2.2.7. The calculations of metals from input sources account for metals in the crankcase oil, oil blended with the fuel, and oil injected in the exhaust. Sulfur levels in the base fuel are also included in the calculation. Output sources of metals include elemental components of PM emissions as well as sulfur components of SO_2 and SO_4. Figure 3.4-8 identifies the input and output sources of sulfur used in the mass balance analysis, and Figure 3.4-9 illustrates the fit of the regression model that was used to estimate sulfur emission recovery rates for different combinations of test oils and oil consumption acceleration rates.
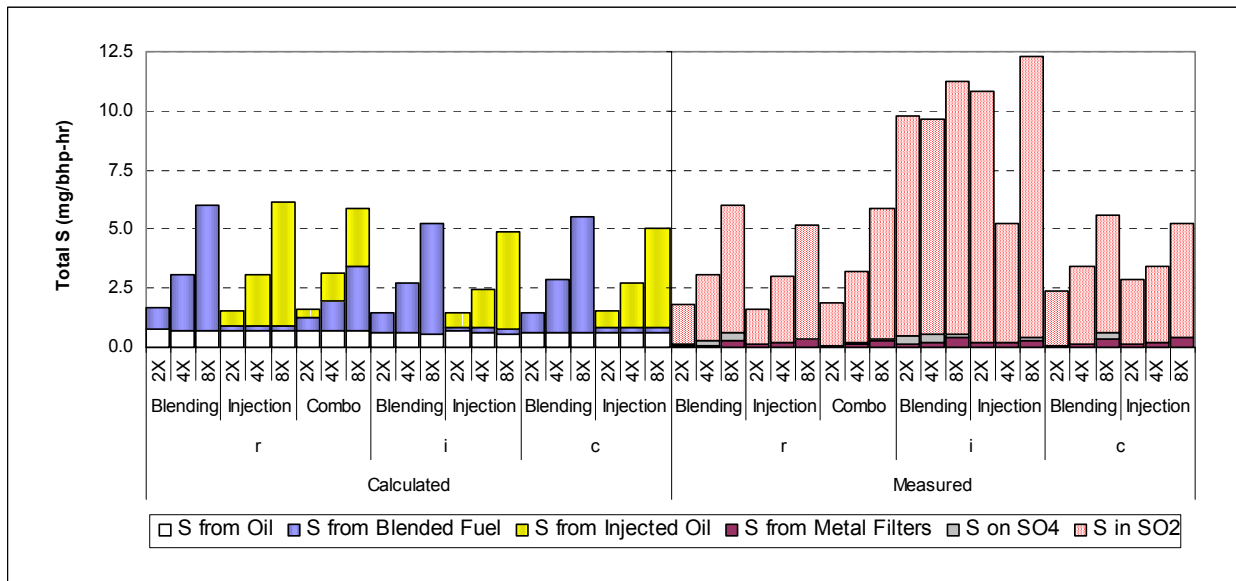
Figure 3.4-8.   Sources of S for mass balance comparison of calculated and measured emissions of sulfur for Part 2 oil consumption acceleration tests.
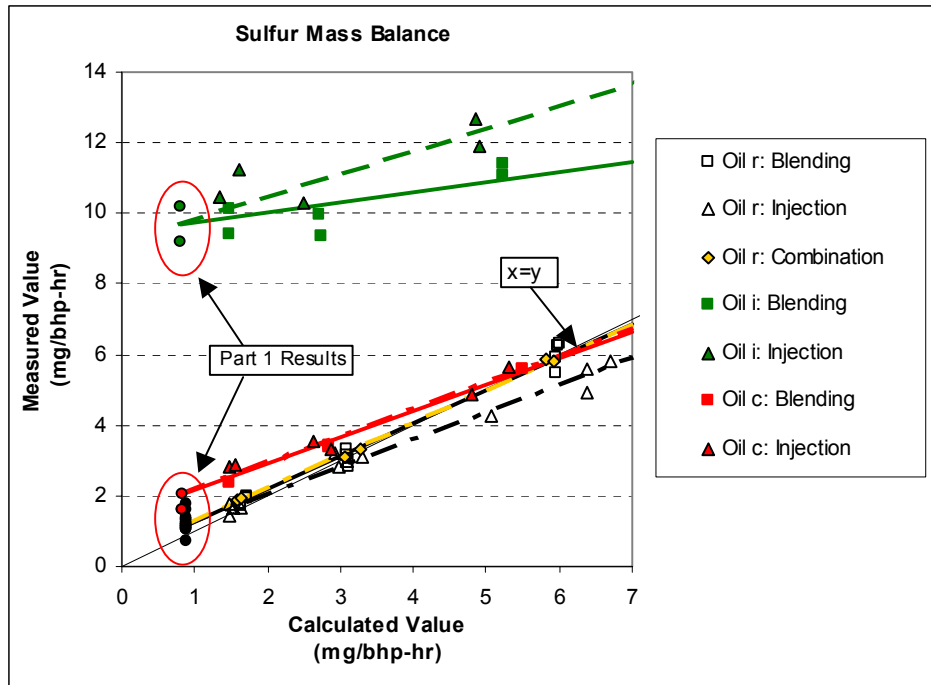
NOTE: Part 1 results are shown for reference – but not included in the regression analysis.

Figure 3.4-9. Mass balance comparisons of S emissions with estimated recovery regression lines – by oil type and oil consumption acceleration method.

Table 3.4-4 summarizes the results of our analysis. The estimated baseline recovery rates, representing the percent of the calculated baseline emissions (i.e., under normal oil consumption rate) recovered in the actual emissions, were derived from the fitted regression model and the calculated emissions. These estimates are consistent with the baseline recovery rates estimated in Part 1 (See Section 3.2.). For example, the average recovery rates for Ca, P, S, and Zn across all oils tested in Part 1 were 42%, 86% (excluding oil c), 125% (excluding oil i), and 38%, respectively. Although the results for oil c were included in the average sulfur recovery rate estimated in Part 1, it was recognized that the sulfur recovery rate for oil c was a bit higher than the average.

The relative recovery rates, representing the percent increase in emissions caused by a 100% increase in oil consumption, vary according to emissions species, oil type, and acceleration method. Generally, the relative recovery rates using oil/fuel blending to accelerate oil consumption are greater than those obtained using the injection method. Furthermore, the relative recovery rates for P and S are much smaller than the baseline rates.

As the level of oil consumption increases from 100% (at 2X) to 700% (at 8X), the relative recovery rates remained constant. Table 3.4-4 shows the overall emissions recovery rates at eight times the baseline oil consumption level.

Graphical summaries of the results are presented in Figures 3.4-10 through 3.4-13. Emissions were estimated using both Part 1 and Part 2 data. The Part 1 results at baseline (1X) are the

average emissions without accelerating oil consumption. At higher oil consumption levels the "projected" results were obtained by simply multiplying the average by the oil consumption acceleration rate. The estimates are based on Part 2 data calculated from the regression model, which was fit to the data collected at 2X, 4X, and 8X oil consumption levels. The predicted emissions at 100% recovery were calculated from the mass balance model.

To address study question 2.4, regarding interactions between the two oil consumption acceleration methods, Figure 3.4-14 compares the sulfur emissions using the combination (blending + injection) method to accelerate oil consumption with the average emissions of each method at the three levels of oil consumption tested. A statistical comparison of these estimates indicates that their difference is marginally significant at the 0.047 level of statistical significance. Similar comparisons for Ca, P, and Zn emissions did not produce statistically significant differences, indicating that the effects of the two acceleration methods on emissions are additive.

Table 3.4-4.   Recovery rates for selected metals – by oil type and oil consumption acceleration method.

Element	Test Oil	Baseline Recovery Rate[1]	Acceleration Method	Relative Recovery Rate[2]	Recovery Rate at 8X Baseline[3]
Ca	c2	26%*	Blending	67%*	62% *
			Injection	20%*	21% *
	i2	34%*	Blending	52%*	50% *
			Injection	16%*	19% *
	r2	28%*	Blending	68%*	63% *
			Injection	15%*	17% *
P	c2	267%*	Blending	65%*	90%*
			Injection	16%*	47% *
	i2	N/A	Blending	-	-
			Injection	-	-
	r2	92%	Blending	69%*	72% *
			Injection	35%*	42% *
S	c2	247%*	Blending	74%*	96%*
			Injection	76%*	97%
	i2	1196%*	Blending	28%*	174% *
			Injection	64%*	205% *
	r2	129%*	Blending	94%*	98% *
			Injection	78%*	84% *
Zn	c2	N/A	Blending	-	-
			Injection	-	-
	i2	N/A	Blending	-	-
			Injection	-	-
	r2	37%*	Blending	44%*	43% *
			Injection	37%*	37% *

(1)   Percent of calculated baseline (no acceleration) emissions.
(2)   Estimated emissions recovery of the element during a 100% increase in oil consumption rate.
(3)   Estimated emissions recovery of the element at eight times the baseline oil consumption rate.
*   Indicates that the estimated recovery rate is different from 100% at the 0.05 level of statistical significance.
N/A = Not applicable. Test oils do not contain significant amounts of the element.

Figure 3.4-10. Emissions of S at 1X (baseline), 2X, 4X, and 8X oil consumption rates, by oil consumption acceleration method and oil type – with 95% confidence limits.

Figure 3.4-11. Emissions of Ca at 1X (baseline), 2X, 4X, and 8X oil consumption rates, by oil consumption acceleration method and oil type – with 95% confidence limits.
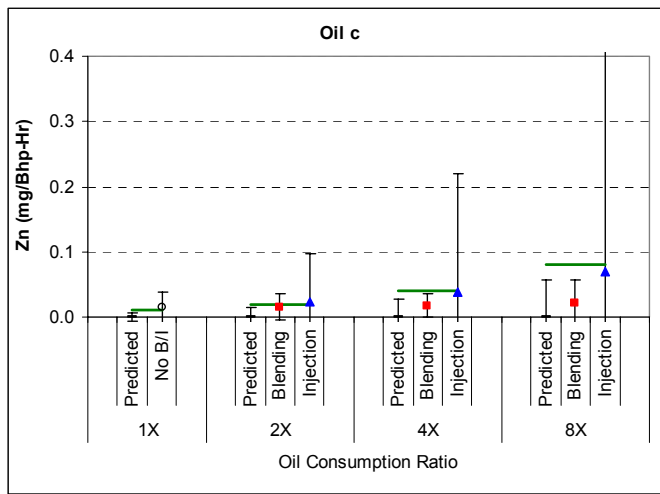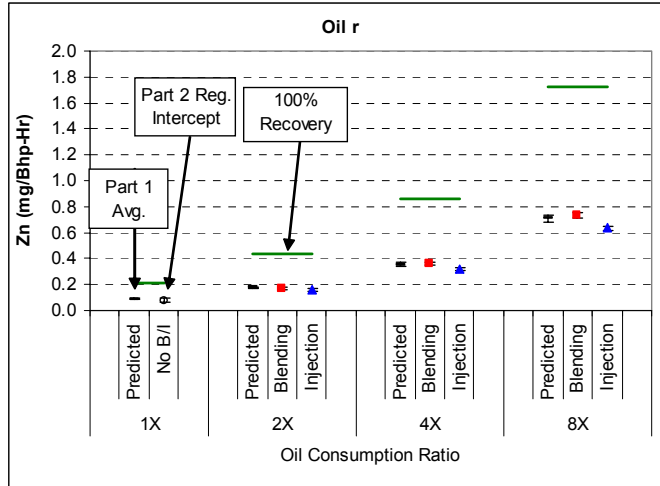
Figure 3.4-12. *Emissions of Zn at 1X (baseline), 2X, 4X, and 8X oil consumption rates, by oil consumption acceleration method and oil type – with 95% confidence limits.*
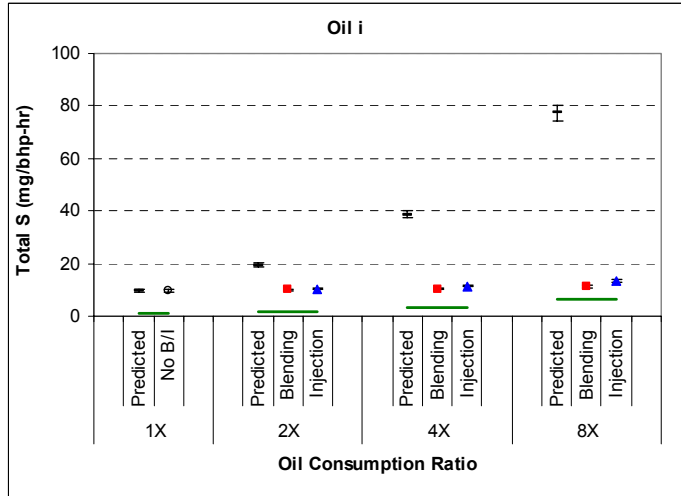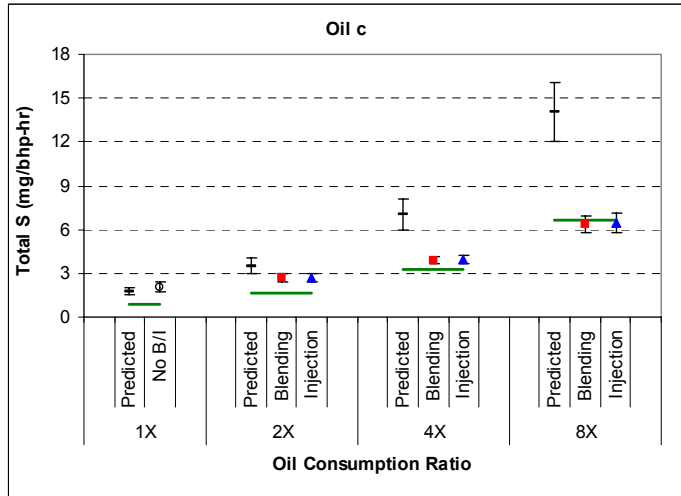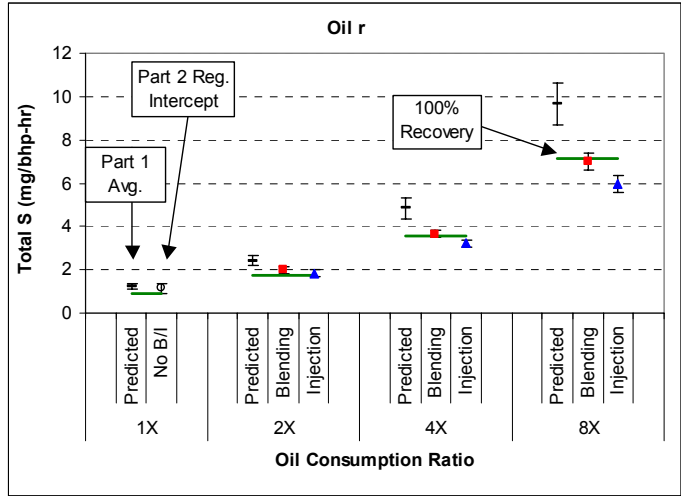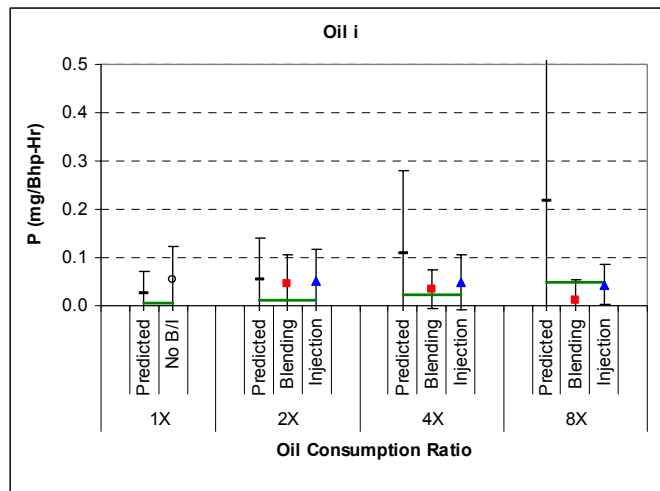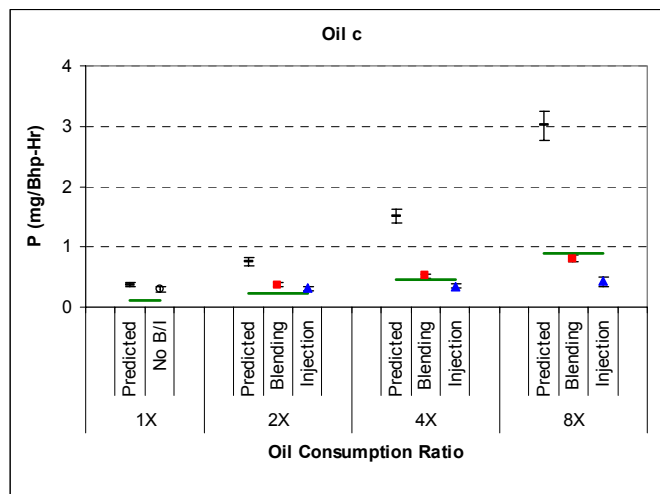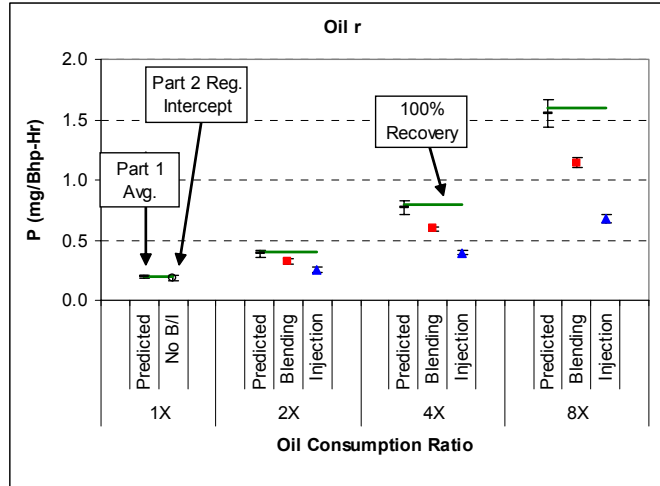
Figure 3.4-13. Emissions of P at 1X (baseline), 2X, 4X, and 8X oil consumption rates, by oil consumption acceleration method and oil type – with 95% confidence limits.
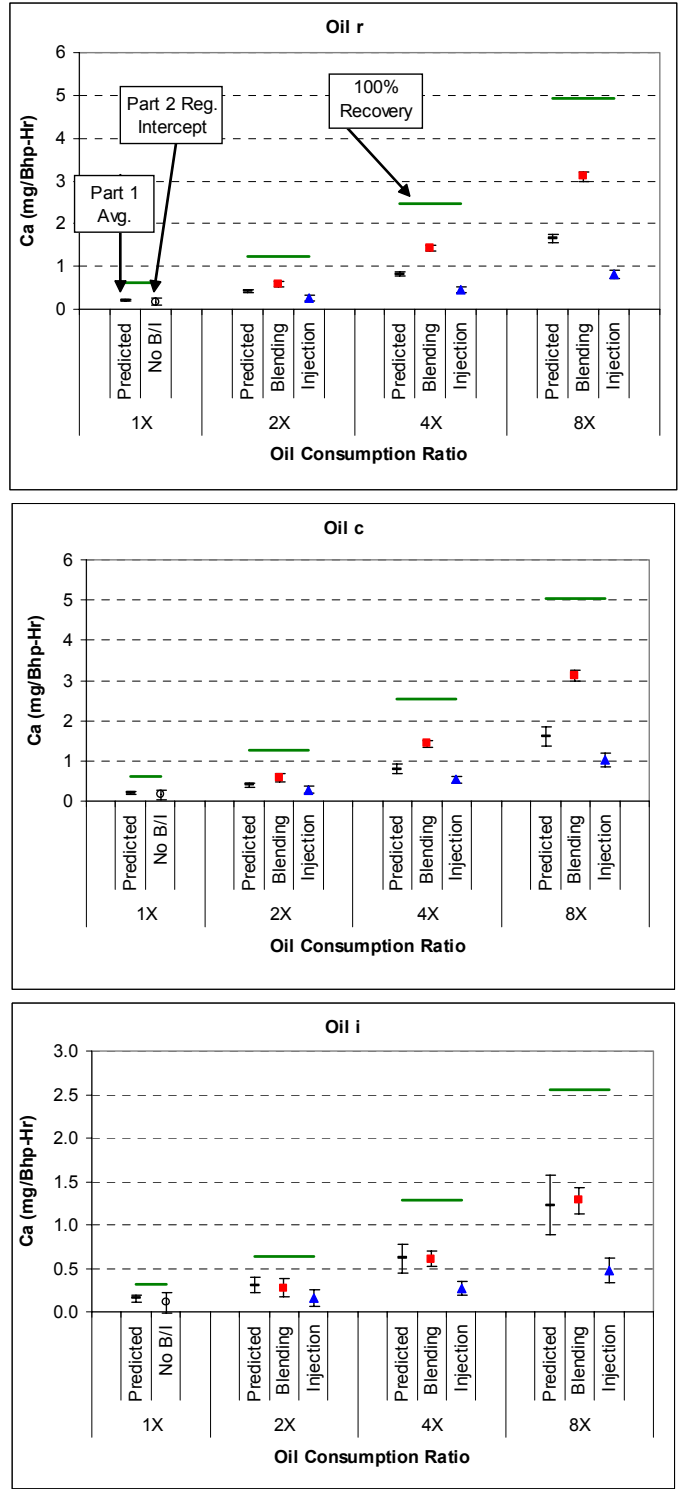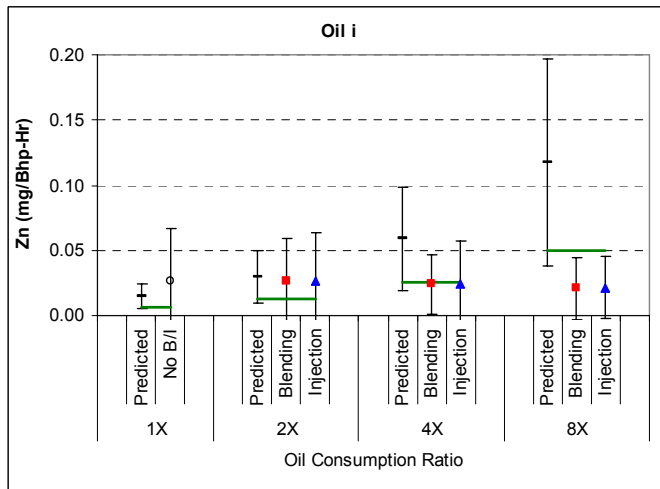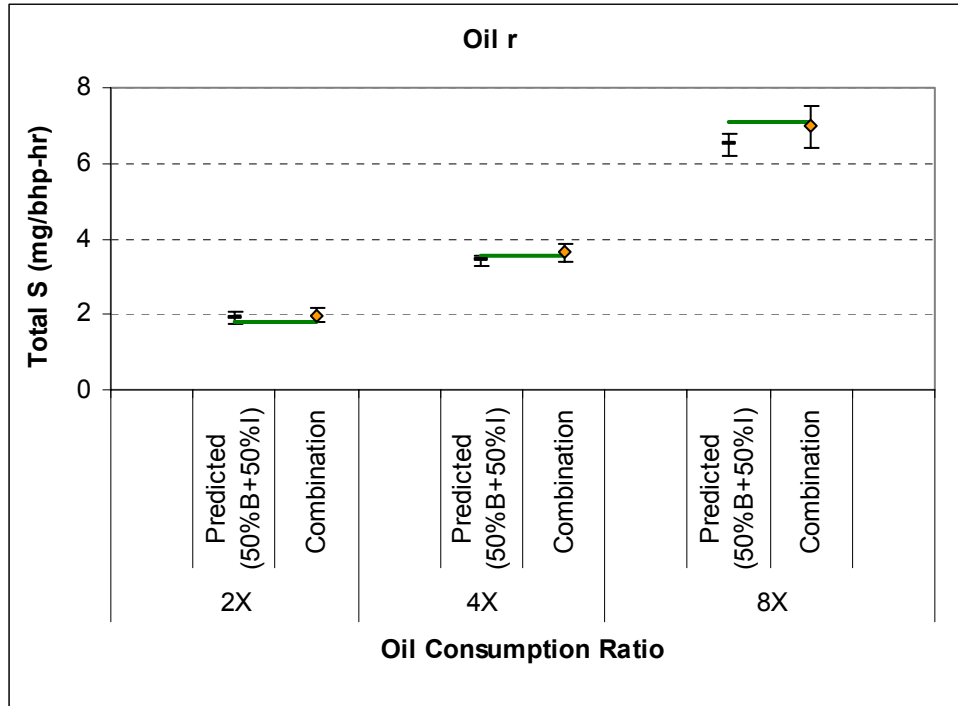
Figure 3.4-14.   Comparison of S emissions using combination (blending + injection) oil consumption method versus average S emissions using each method – with 95% confidence limits.

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Section 4: Conclusions

This study involved the most thorough examination of the impact of lubricant additives and base oils on diesel engine emissions ever undertaken. The results are helping to provide a basis for future lubricant specifications. The following are the major conclusions from the research:

Part 1 Conclusions

Impact of Oil Formulation on Gaseous and Total Particulate Emissions

- Oil formulation has statistically significant effects on nearly every emissions component:

- Both additives and base oils affect emissions

- There are interactions between the additive and base oil effects (i.e., the effects of additives are not the same for each base oil)\

- The most significant effects are on emissions of SO_2 and metals due to widely varying metal contents of additives and sulfur level in base oils.

- The magnitude of the effects on regulated emissions vary by component
  - HC emissions vary by $\pm25\%$
  - CO and NO_x vary by $\pm8\%$
  - PM emissions vary by $\pm15\%$.

- These findings support the motivating hypothesis for this study. That is, oil formulation for diesel engines using low sulfur fuel can have a significant impact on SO_2 emissions. It is known that SO_2 has a negative impact on the durability of certain advanced emission control technologies.

Impact of Oil Formulation on Emissions of Metals

- Metal emissions (S, Ca, Zn, P, and Mg) are highly correlated with predicted emissions based on the composition of oil (and fuel sulfur); however, recovery rates vary considerably (ranging from 27% for Mg to 127% for S) (Table 3.2-1).

- However, oil formulation is not always an accurate predictor of engine emissions:
  - S emissions from one oil (i2) were 8 to 10 time higher than predicted from the mass balance.
  - P emissions from all oils using additive ''c'' were 4 to 5 times higher than predicted.

  - 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 likely not the case. The recovery rate

is indicative of 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. However, it was beyond the scope of this study to investigate the specific fate of the various elements. The apparent recovery rate for sulfur is significant because it is perhaps the most scrutinized of the potential catalyst poisons, especially with respect to NO_x adsorber catalysts. It is important to note, though, that the nature of the sulfur compounds emitted is more important than the fact that they are emitted.

Impact of the Source of Sulfur in Oil on SO_2 Emissions

- Although the sulfur content of the oil is the primary predictor of SO_2 emissions (59% correlation), the results demonstrate that oils containing higher levels of Zn and Mo produce lower levels of SO_2 emissions. Adding Zn and Mo to the SO_2 prediction model increases the correlation from 59% to 74%.

- This finding offers additional evidence that the *source* of sulfur in the lubricant has an impact on the resultant emissions of SO_2. The oils with higher zinc content have a larger portion of the sulfur coming from 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_2 in the exhaust. This also suggests that chemical limits may need to include additional factors to maximize the ability to predict SO_2 emissions based on the oil analysis.

Part 2 Conclusions

Impact of Oil Consumption Method on Gaseous and Total Particulate Emissions

- 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 via injection of oil into the exhaust stream. However, if oil consumption is doubled by blending oil with the fuel, the impact on HC, CO, and PM emissions is negligible.
  – NO_x 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_x, and PM emissions when oil consumption is accelerated.
  – The relative increase in SO_2 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.

Impact of Oil Consumption Method on Metal Recovery Rates

- 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%).
  - Relative recovery rates for S range from 25% to 85% depending on the particular combination of test oil and acceleration method.

- Neither the blending approach nor the injection approach was adequate to simulate the sulfur emissions expected from the baseline testing. This disparity was especially great with Oils i2 and c2, which exhibited nonstandard behavior relative to the other products tested. This inability to accelerate oil consumption realistically—with respect to the measured emissions—left the project team little confidence in utilizing such a scheme for rapid catalyst aging. All Phase 2 tests (described in Section 5) were conducted without using artificial means to accelerate oil consumption.

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Section 5: Future Research Plans

A second phase of this study is currently underway. The primary objective of Phase 2 is to provide data to the industry that increases the collective knowledge base relative to the effects of lubricant on performance and durability of diesel emission control systems. Specifically, the project will focus on NO_x adsorber catalyst systems. Studies specific to lubricant effects on PM control technologies are being addressed in other programs. Resource constraints require that this study focus on only one technology, even though other NO_x control technologies (e.g., urea selective catalytic reduction) are also being considered for use in future engines.

The fluid matrix examined in Phase 2 is not as robust as was tested in Phase 1, due largely to the time and expense involved in durability testing. Instead, a set of oils has been blended that varies in sulfur, phosphorus, and ash content by varying the levels of zinc dialkyl-dithiophosphate (ZDDP) and detergent (calcium sulfonate, calcium salicylate, and calcium phenate) additives.

A Cummins ISB engine with EGR is being used for the second phase of testing. A full-flow NO_x adsorber catalyst and hydrocarbon dosing system has been integrated with the test engine. For each test, a new NO_x adsorber catalyst is installed. Each test is 400 hours in duration and includes emission evaluations at 100-hour intervals. All Phase 2 tests are being conducted with the 0.6-ppm S base fuel. Certain test oils have been 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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Section 6: References

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Appendix A: Statistical Method for Selecting Test Additives

The additive companies participating in this project made available 26 commercial and experimental packages for selection. In order to test representative additive packages in all four base oils listed in Section 2.1.5 and to evaluate the repeatability of the test and measurement process within resource constraints, it became necessary to limit the number of additive packages to be tested. It was decided that no more than 13 additive packages, including a reference package, would be tested. A statistical design was employed to select packages that would adequately span the range of properties of interest while balancing the contribution between additive suppliers. This section presents the statistical approach to selecting representative additive packages.

The candidate additive packages were labeled by uppercase letters A through Z. Specifications, in terms of ash level, sulfur, calcium, zinc, nitrogen, phosphorus, boron, chorine, molybdenum, and magnesium are provided in Table A-1, and selected pairwise plots of the constituents of these additive packages are presented in Figure A-1. Package U was selected for the reference oil because it is an existing product with a high ash level that had been tested extensively. Package U was blended only with the Group 2 base oil to serve as the reference oil and tested periodically to account for testing trends.

The additive package selection process was a two-step process. First, 12 of the 26 packages plus package U were chosen for testing with the Group 2 base oil. Then, six of the 12 chosen packages were selected for Group 2 duplicate testing, as well as for testing with the remaining three groups of base oils. The primary selection criterion was that the properties of the selected packages should span the practical ranges of elemental composition and ash levels shown in Table 2.1-2. To balance the representation from the participating companies, it was determined that:

1. At step 1, the single package provided by one of the companies and three packages from each of the remaining four companies would be selected, and
2. At step 2, at least one package from each company would be chosen.

Selection of Packages for Group 2 Testing

Two statistical approaches were employed to select representative additive packages for emissions testing based on their elemental compositions. Both approaches use principal components analysis (PCA) to transform the elemental compositions into nine new properties called principal components. Principal component analysis is a statistical technique for analyzing multidimensional data. The set of correlated variables are transformed to a new set of uncorrelated variables called principal components (Morrison 1976). In this case, the principal components (PCs) are uncorrelated linear combinations of the standardized element concentrations. Typically, they are listed in order of the amount of variability in the data that each explains. If the original properties (elemental concentrations) are highly correlated, the first few PCs will explain a high percentage of the variability. Pairwise plots of the first five PC

scores (the value of the PC variables) applied to the additive packages are shown in Figure A-2. The PRINCOMP procedure in the Statistical Analysis System (SAS©) software package was used to perform this analysis.

Figure A-1.   Additive contributions to composition of finished oils prepared with candidate additive packages.

| Additive Package	Ash Level (%)	S (ppm)	Ca (ppm)	Zn (ppm)	N (ppm)	P (ppm)	B (ppm)	Cl (ppm)	Mo (ppm)	Mg (ppm)
A	1.20	4950	3950	0	2000	600	0	<100	0	<50
B	0.00	5	0	0	950	670	0	0	0	0
C	0.23	725	415	225	1457	587	176	60	0	0
D	0.24	2440	0	1320	1045	1199	142	0	0	0
E	0.29	725	415	225	1397	205	393	60	0	0
F	0.30	20	870	0	2235	0	985	0	0	0
G	0.30	1080	435	590	2060	530	950	0	0	0
H	0.41	1404	1074	315	882	284	0	120	0	0
I	0.47	2205	959	288	819	278	109	120	0	139
J	0.54	2960	1200	770	940	700	0	0	0	0
K	0.60	4226	1748	0	0	0	0	100	0	0
L	0.60	0	1936	0	0	1153	0	100	0	0
M	0.75	2785	1820	860	1286	760	60	126	0	0
N	0.80	2400	400	1000	600	900	200	100	0	900
O	1.00	3200	600	1400	900	1200	200	200	0	1200
P	1.05	3327	2397	1190	911	1085	295	70	0	0
Q	1.15	3530	2360	1290	940	1180	130	0	90	270
R	1.20	4223	2915	1270	0	1149	0	100	0	0
S	1.31	961	3750	176	1045	161	142	0	0	0
T	1.20	0	3484	0	0	0	1099	100	0	0
U	1.35	4454	3412	1269	855	1156	0	80	0	0
V	1.40	2224	4128	0	1560	0	0	18	0	0
W	1.40	2921	3130	865	1137	788	143	0	284	277
X	1.44	3246	3130	1319	1182	1201	1235	0	0	277
Y	1.50	4500	800	1900	1200	1700	300	200	0	1700
Z	1.85	6590	4770	1560	970	1420	150	0	170	0
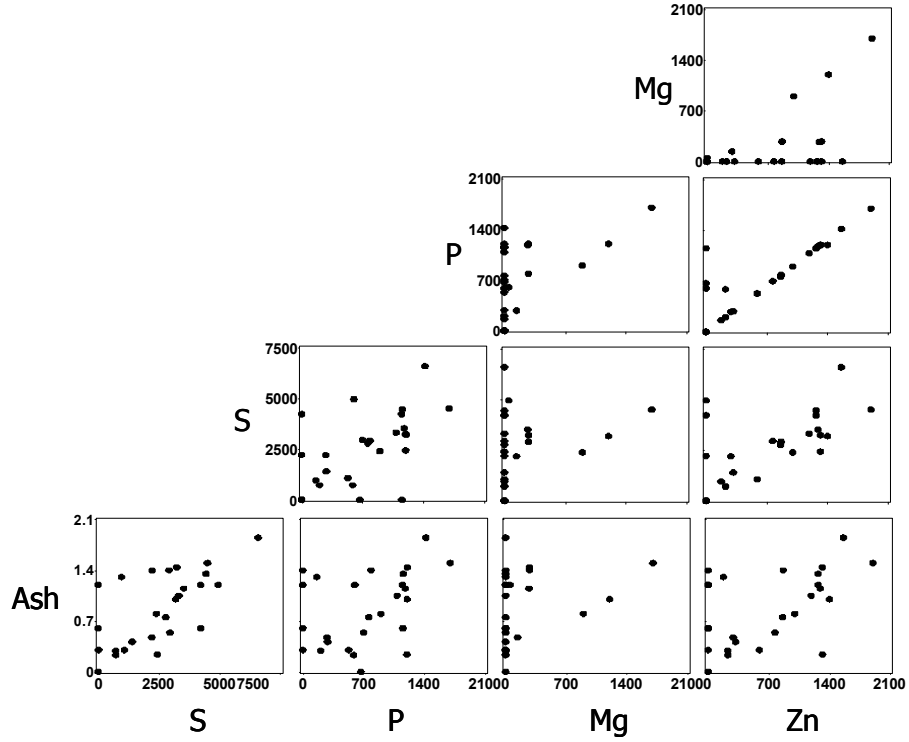
Figure A-1.   Selected pairwise plots of constituents (ppm S, P, Mg, Zn and % Ash).
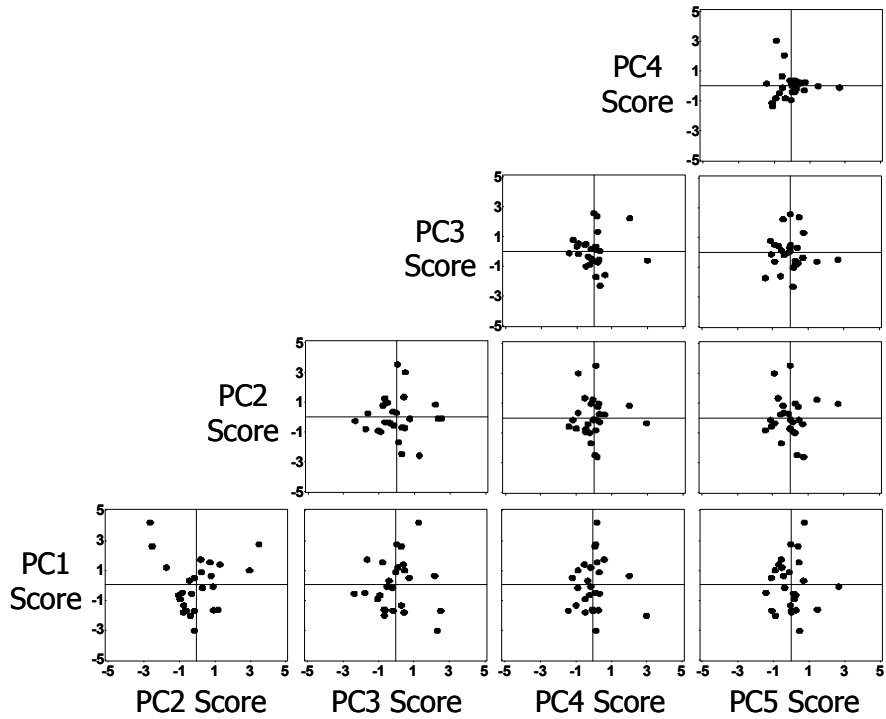

Figure A-2.   Selected pairwise plots of principal components scores.

Table A-2 lists the PC coefficients applied to standardized element concentrations for each PC along with the amount of variability explained. Notice that the first five PCs explain 88% of the total sample variance in the original system. Also presented in Table A-2 are simple "descriptions" of the PCs. This involves identifying the elements with the largest coefficients. Arbitrarily, we chose elements with coefficients greater than 0.3 in absolute value. Thus one can describe PC1 as S+Zn+P-N, PC2 as Ca+Mo-Cl-Mg, and so on. These descriptions might be helpful for interpreting the PC scores for each package in terms of some physical characteristics of the package.

The first approach (Approach #1) for selecting representative additive packages was adapted from a method developed by McAdams (2001). It employs a factorial design to selected principal components (PCs). The second approach (Approach #2) attempts to optimize the "distance" between selected packages based on all nine PCs. Both approaches are discussed in detail by Orban et al. (2003) and summarized below.

Table A-2. Principal component coefficients and contributions to total variance.

Constituent	PC1	PC2	PC3	PC4	PC5	PC6	PC7	PC8	PC9
S	0.453*	0.258	-0.106	0.034	0.404*	-0.187	-0.582*	-0.140	0.399*
Ca	0.085	0.529*	-0.236	0.521*	0.329*	0.030	0.490*	-0.074	-0.176
Zn	0.514*	0.032	0.262	0.028	-0.165	-0.243	-0.171	-0.017	-0.741*
N	-0.142	0.098	0.595*	-0.355*	0.645*	-0.015	0.155	0.209	-0.077
P	0.501*	0.003	0.157	-0.092	-0.268	-0.308*	0.490*	0.341*	0.439*
B	-0.160	-0.032	0.581*	0.710*	-0.148	0.033	-0.223	0.150	0.190
Cl	0.233	-0.501*	-0.295	0.214	0.329*	0.273	-0.060	0.600*	-0.119
Mo	0.175	0.498*	0.086	-0.200	-0.261	0.718*	-0.137	0.267	0.014
Mg	0.375*	-0.378*	0.230	0.055	0.118	0.468*	0.236	-0.600*	0.107
Contribution to Total Variance	3.001	1.921	1.394	0.822	0.744	0.621	0.232	0.182	0.083
Cumulative % Total Variance	33%	55%	70%	79%	88%	94%	97%	99%	100%
"Description"	S+Zn+P+Mg	Ca+Mo-Cl-Mg	N+B	Ca+B-N	S+Ca+N+Cl	-P+Mo+Mg	-S+Ca+P	P+Cl-Mg	S-Zn+P

* Indicates coefficient is >0.3 or <-0.3.

Approach #1

The first approach selects packages that approximate a factorial design in the first n PCs. It ensures optimal information on the effects of these PCs. In this study, the first three PCs were used to choose packages that approximate a three-factor design where the first factor has three levels and the other two factors have two levels, as shown in Table A-3. This yields 12 treatments (packages). However, package U, which was used in the reference oil, was also included.

Table A-3.   A three-factor design.

Treatment	Factor #1	Factor #2	Factor #3
1	-1	-1	-1
2	-1	-1	+1
3	-1	+1	-1
4	-1	+1	+1
5	0	-1	-1
6	0	-1	+1
7	0	+1	-1
8	0	+1	+1
9	+1	-1	-1
10	+1	-1	+1
11	+1	+1	-1
12	+1	+1	+1

The selection process begins by calculating principal component scores for all test oils. A PC score is the sum of the products of the PC coefficients (Table A-2) and the standardized elemental composition of the test oils. Next, a transformation was applied to map the PC scores into values between −1 and +1. Finally, the additive package whose transformed score has the smallest squared deviation from a target configuration is selected. This process is repeated for all target configurations in Table A-3. In the cases where a package has already been selected, the next available package was used. The resulting selection generated from Approach #1 was (A, F, G, K, L, N, O, U, V, W, X, Y, Z).

Approach #1 is intuitively appealing because it provides some flexibility in selecting the design. In this example, we used a full factorial design in three variables plus center points for factor #1. We could have chosen a fractional factorial design and used additional PC factors. However, there are two significant disadvantages of Approach #1. First, it produces a design that does not consider all of the PCs in the selection process. It is possible that the last few PCs, which were not used in the selection process, could have significant effects on the emissions. Secondly, there is no practical way to ensure that the selection will satisfy the criterion for balancing the representation from the participating companies. In an attempt to overcome these problems, Approach #2 was developed.

Approach #2

Another way to select packages with properties that span the practical ranges of elemental composition is to maximize the "distances" between design points. The approach we selected involves two steps. The first step is to maximize the minimal pairwise statistical distance for the combinations of 13 additive packages (including the reference package U). There can be multiple combinations that achieve this maximum minimum distance; so, the next step is to select from these combinations the one combination that has the maximum total pairwise distances between packages. Other constraints, such as balancing the number of packages selected from participating companies, are easily added.

This approach utilizes all nine PCs to make sure that no packages with similar properties are chosen. The pairwise statistical distance between package P1 and package P2 is calculated using the formula

$$\text{Distance} = \sqrt{\sum_{i=1}^{9} \frac{(x_i - y_i)^2}{s_{ii}}} \ ,$$

where $(x_1, x_2, \cdots, x_9)$ is the 9-dimensional PC scores for package P1, $(y_1, y_2, \cdots, y_9)$ is the 9-dimensional PC scores for package P2, and $s_{11}, s_{22}, \cdots, s_{99}$ are sample variances of PC scores.

For each combination of 13 packages containing U, the pairwise statistical distances were calculated and the minimal pairwise statistical distance was determined. Next, the combinations with balanced representation from participating companies that maximize the minimal distance were considered for the final selection. There were 951 combinations that met the initial selection criteria. Table A-4 displays the top five selections, sorted by total pairwise distance. The first two selections were very similar. The only difference was that Selection #1 contains package L but Selection #2 contains package T. Since package T may provide useful information on low-sulfur, high-ash oils, Selection #2 was chosen as the final selection for Approach #2.

Table A-4.   Results for selection approach #2.

Selection		1	2	3	4	5
Total Pairwise Distance		370.0	369.4	368.6	368.5	366.5
Minimal Pairwise Distance		2.22	2.22	2.22	2.22	2.22
Additive Package	1	A	A	A	A	A
	2	B	B	B	B	B
	3	C	C	C	C	E
	4	F	F	G	G	J
	5	K	K	K	K	K
	6	L	M	L	M	L
	7	M	T	M	T	M
	8	U	U	U	U	U
	9	V	V	V	V	V
	10	W	W	W	W	W
	11	X	X	X	X	X
	12	Y	Y	Y	Y	Y
	13	Z	Z	Z	Z	Z

Comparisons of the Two Approaches

The two approaches resulted in similar, but not identical solutions. Both approaches select packages A, F, K, U, V, W, X, Y and Z. Packages G, L, N and O were selected by Approach #1 but not by Approach #2. Packages B, C, M and T were selected by Approach #2 but not by Approach #1.

For this project we chose to use the selections derived from Approach #2. With Approach #1 there is no clear way to generate a selection that balances the representation from the participating companies. On the other hand, Approach #2 looked at all possible combinations of 13 packages that satisfy the selection criteria, then picked the best one.

Selection of Packages for Testing in
All Four Groups

A similar approach was used to select six additive packages for testing with all four base oil groups and for duplicate testing in Group 2, except we started with the selection of 13 packages (A, B, C, F, K, M, T, U, V, W, X, Y, Z) that were tested in Group 2. We also imposed the constraints that package U would not be included in the group of 6 (because the same reference oil was tested throughout the program) and at least one package had to be selected from each participating company. Also, because of the importance of generating information about the effects of ash and sulfur, the technical oversight committee requested that the selection include packages B (lowest ash and sulfur) and Z (highest ash and sulfur). The resulting selection was (A, B, T, M, Y, Z).

Once the 13 additive packages were selected, the order of testing was randomized – with the constraint that the six packages selected for testing in all four base oils, would be tested first. Identification labels were reassigned to the lowercase letters a through l and r, as shown in Table A-5. The experimental design (Table 2.1-5) and all of the findings from this study are presented using the lowercase letters to identify the packages.

Table A-5.   Test package IDs for selected candidate packages.

	Candidate Package												
	T	B	A	Y	Z	M	X	W	K	V	F	C	U
Test Package ID	a*	b*	c*	d*	e*	f*	g	h	i	j	k	l	r++

*   Packages tested in all four base oils
++   Reference package
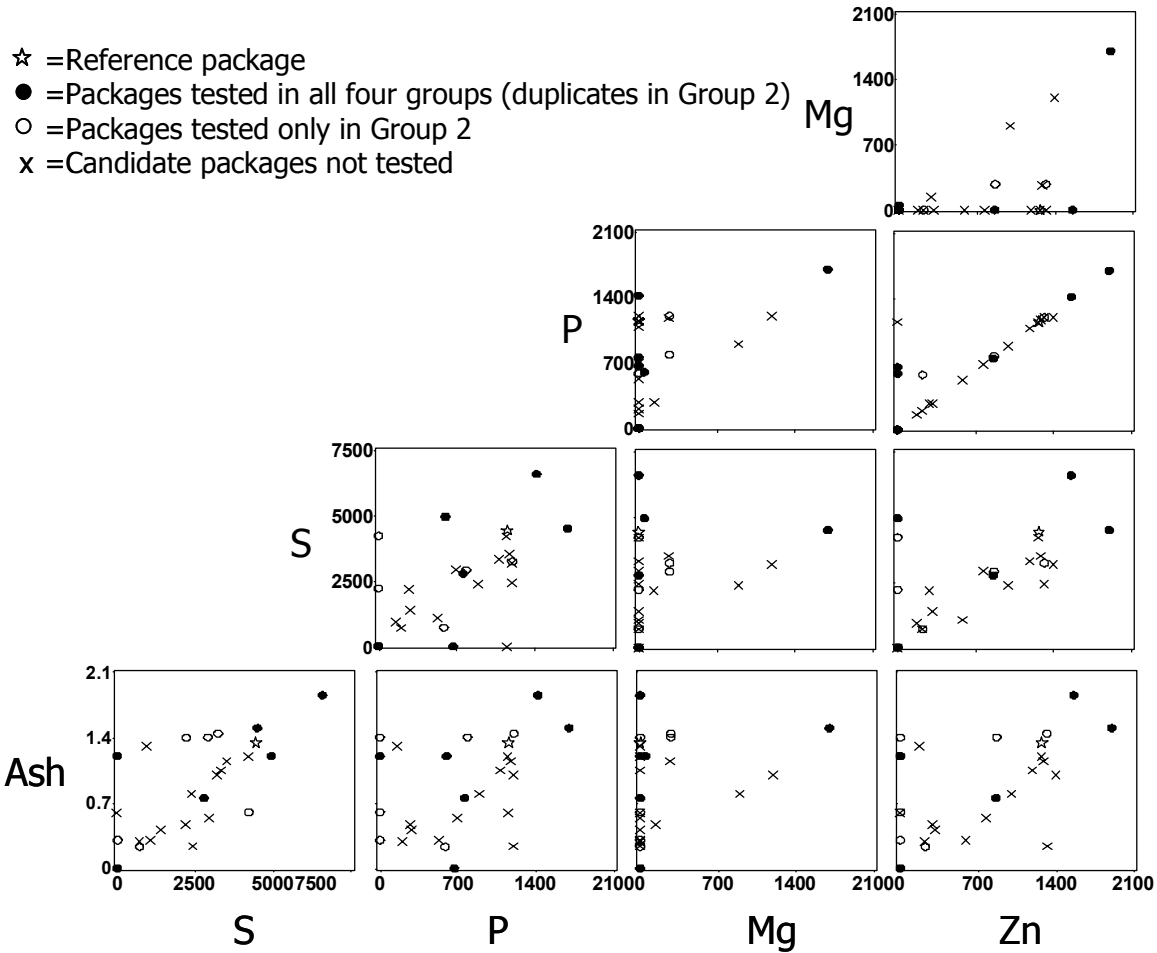
Figure A-3.   Selected additive packages constituents (ppm S, P, Mg, Zn and % Ash).

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Appendix B: Measured Properties of Test Oils

Table B-1. Average measured properties of Group 1 test oils.

Element/Property	Additive Package							
	a	a*	b	c	c*	d	e	f
Number of Samples	1	1	1	1	1	1	1	1
Ash Level (%)	1.25	1.24	0.18	1.39	1.39	1.45	1.87	0.89
S, ppm	3610	3400	4160	6570	6880	7390	8930	5680
Ca, ppm	3307	3605	121	3992	4066	983	4785	2193
Zn, ppm	194	1	216	147	1	1885	1542	940
P, ppm	185	1	836	712	694	1760	1413	865
B, ppm	1194	1330	31	4	3	257	145	70
Mo, ppm	1	1	1	1	1	1	163	19
Mg, ppm	193	20	207	10	10	1672	16	6
Viscosity, 40°C	96	96	104	99	102	108	102	101
Viscosity, 100°C	14	13	14	14	15	14	14	14
Total Base Number	11	11	2	10	11	10	12	6
Si, ppm	3	5	1	4	6	2	7	1
Na, ppm	6	6	5	5	5	5	5	5
K, ppm	5	5	5	5	5	5	5	5
Fe, ppm	3	2	2	9	2	5	5	3
Pb, ppm	1	1	1	1	1	1	1	1
Cu, ppm	1	1	1	1	1	1	1	1
Cr, ppm	1	1	1	1	1	1	1	1

* Unused oil

Table B-2.   Average measured properties of Group 2 test oils.

Element/Property	Additive Package													
	a	b	c	c⁺	d	e	f	g	h	i	i⁺	j	k	l
Number of Samples	2	2	2	6	2	2	2	1	1	2	6	1	1	1
Ash Level (%)	1.22	0.07	1.21	1.18	1.42	1.78	0.83	1.41	1.35	0.62	0.68	1.38	0.33	0.2
S, ppm	515	245	3620	3593	4780	5785	2635	3250	2920	3500	3368	1980	310	520
Ca, ppm	3536	80	3526	3460	953	4524	2038	3183	3141	1787	1936	4027	952	497
Zn, ppm	40	71	56	7	1819	1509	902	1292	855	35	106	53	13	197
P, ppm	53	738	626	526	1712	1414	824	1226	814	34	99	52	13	533
B, ppm	1263	20	4	1	263	144	67	1009	171	3	1	1	718	241
Mo, ppm	1	1	2	1	1	158	11	1	274	4	1	1	2	1
Mg, ppm	19	68	9	9	1670	33	9	268	298	12	6	12	3	2
Viscosity, 40°C	114	101	99	98	106	108	109	106	108	106	107	104	106	108
Viscosity, 100°C	15	14	14	14	14	14	14	14	15	14	14	14	14	14
Total Base Number	11	1	9	8	10	11	6	10	11	5	5	11	5	2
Si, ppm	3	1	4	3	4	5	3	4	7	1	1	2	5	1
Na, ppm	15	5	5	5	5	5	5	5	5	15	14	5	5	5
K, ppm	5	6	5	5	7	6	7	7	8	6	5	8	623	91
Fe, ppm	6	2	4	7	4	5	4	6	6	3	5	6	3	3
Pb, ppm	2	1	2	1	1	1	1	1	1	1	1	1	1	4
Cu, ppm	1	1	1	1	1	1	1	1	1	1	1	1	1	1
Cr, ppm	1	1	1	1	1	1	1	1	1	1	1	1	1	1

+ Part 2 Tests

Table B-3.   Average measured properties of Group 3 test oils.

Element/Property	Additive Package					
	a	b	c	d	e	f
Number of Samples	1	1	1	1	1	1
Ash Level (%)	1.23	0.17	1.22	1.45	1.66	0.82
S, ppm	450	510	4140	4790	5380	2250
Ca, ppm	3570	425	3546	977	4263	2062
Zn, ppm	24	142	9	1911	1368	775
P, ppm	24	750	669	1777	1329	710
B, ppm	1267	1	2	268	128	197
Mo, ppm	1	1	1	1	146	1
Mg, ppm	19	1	19	1677	14	7
Viscosity, 40°C	93	92	87	93	95	92
Viscosity, 100°C	15	14	14	14	14	14
Total Base Number	11	1	9	10	10	6
Si, ppm	4	1	4	6	2	3
Na, ppm	16	5	5	5	5	5
K, ppm	5	5	5	5	5	5
Fe, ppm	6	6	4	4	5	3
Pb, ppm	1	1	1	1	1	1
Cu, ppm	1	1	1	1	1	1
Cr, ppm	1	1	1	1	1	1

Table B-4. Average measured properties of Group 4 test oils.

Element/Property	Additive Package									
	a	b	b*	c	c*	d	d*	e	e*	f
Number of Samples	1	1	1	1	1	1	1	1	1	1
Ash Level (%)	1.28	0.17	0	1.41	1.38	1.43	1.45	1.82	1.9	0.78
S, ppm	1200	530	70	4400	4140	4760	4820	6210	6210	2400
Ca, ppm	3215	414	2	4053	4040	1179	911	4671	4864	1885
Zn, ppm	279	141	1	181	5	1817	1901	1510	1575	856
P, ppm	261	746	719	748	696	1710	1769	1415	1449	782
B, ppm	1151	2	1	18	3	241	274	140	154	84
Mo, ppm	1	1	1	19	1	1	1	160	167	1
Mg, ppm	282	8	1	9	9	1531	1730	17	17	9
Viscosity, 40°C	100	92	95	87	90	89	90	91	94	86
Viscosity, 100°C	15	14	15	14	15	14	14	14	15	14
Total Base Number	11	2	1	10	10	10	10	12	13	6
Si, ppm	1	1	1	4	5	4	10	5	11	2
Na, ppm	8	5	5	5	5	5	5	5	5	5
K, ppm	5	5	5	5	5	5	5	5	5	5
Fe, ppm	3	7	1	4	2	3	2	5	3	3
Pb, ppm	1	2	1	1	1	1	1	1	1	1
Cu, ppm	1	1	1	1	1	1	1	1	1	1
Cr, ppm	1	1	1	1	1	1	1	1	1	1

* Unused Oil

Table B-5. Average measured properties of reference oil.

Element/Property	Reference Oil (Group 2)				
	Pre-aging	Post-aging	Average Pre & Post	Unused	Part 2
Number of Samples	13	7	20	1	15
Ash Level (%)	1.35	1.37	1.36	1.38	1.37
S, ppm	3969	3864	3933	4080	4036
Ca, ppm	3423	3473	3441	3474	3506
Zn, ppm	1205	1198	1203	1252	1170
P, ppm	1131	1077	1112	1173	1113
B, ppm	21	9	17	1	1
Mo, ppm	2	2	2	1	1
Mg, ppm	12	14	12	11	12
Viscosity, 40°C	106	108	107	110	104
Viscosity, 100°C	14	14	14	15	14
Total Base Number	9	8	9	10	9
Si, ppm	2	3	3	6	3
Na, ppm	5	5	5	5	5
K, ppm	6	6	6	5	5
Fe, ppm	3	37	15	2	9
Pb, ppm	1	1	1	1	1
Cu, ppm	1	1	1	1	1
Cr, ppm	1	1	1	1	1

Appendix C: Oil Injection System Design

As described in Section 3.4, Part 2 of this study included an investigation of various means for increasing engine oil consumption including blending oil into the fuel, injecting oil into the exhaust manifold, and a combination of the two approaches. This appendix provides details of the oil injection system design.

Figure C-1 displays a close-up view of the injector body. The main body is a 0.25" outer diameter stainless steel tube. It is attached to a pipe fitting which screws directly into the exhaust manifold (Figure C-2). Oil was introduced into the injector body via a 0.125" tube and compressed air was fed into the end of the tube. The oil was housed in a cylindrical bomb under nitrogen pressure (Figure C-3) and dripped into the injector body where it was swept by the compressed air supply into the exhaust manifold. The nitrogen pressure applied to the oil ranged from 8 to 22 psi, depending on the exhaust back pressure at each test mode. A solenoid valve was used to pulse the oil at 1 Hz to give the appropriate mass flow rate. A rotameter regulated the compressed air flow to 2 ft^3/hr, which was 1% of the total exhaust flow rate.

Figure C-1. Oil Injector Body used in Part 2 experiments.

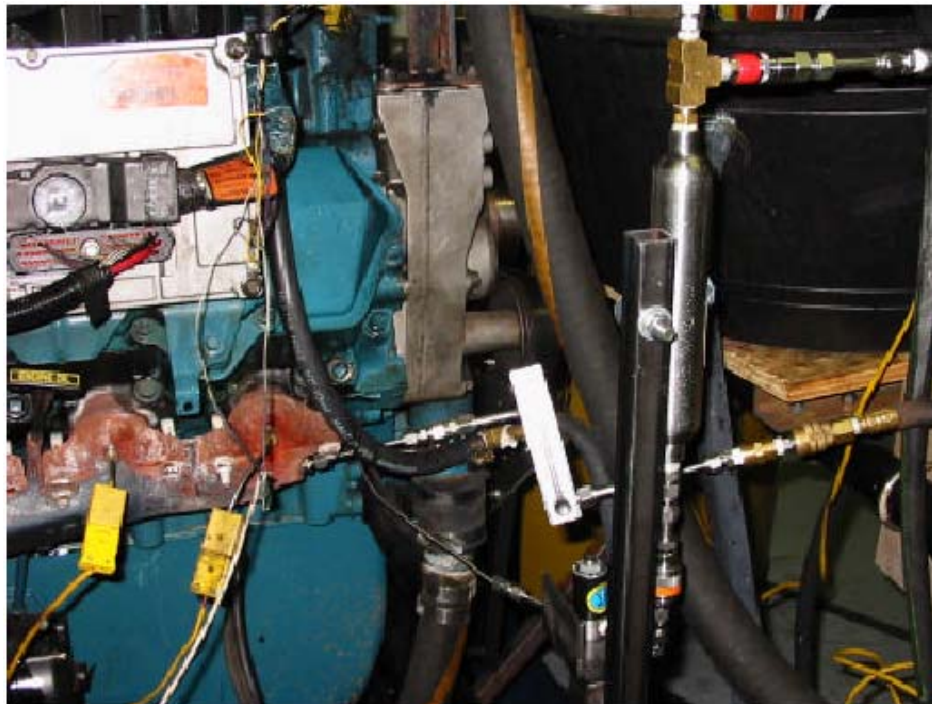*Figure C-2. Oil injector body attached to exhaust manifold.*

Figure C-3. Oil injection system with oil reservoir.

Appendix D: Analysis of the Consistency in Reference Oil Test Results

Throughout the study, a reference oil, consisting of additive "r" in a Group 2 base oil, was tested at regular intervals in order to assess the consistency of test and measurement processes. The reference oil evaluations were performed in duplicate after every third evaluation of test oils. Furthermore, every other reference oil evaluation was followed by a 40-minute oil aging sequence, then a second set of duplicate emissions tests. (See the experimental design matrix in Table 2-7.) This section presents an analysis of trends in the reference oil results and discusses how we made adjustments to test results when it was demonstrated that deviations in the measurement or testing procedures affected the results.

For each emission species of interest, the emission results of the reference oils were plotted against the test date and statistically analyzed to determine if there were systematic changes in the emissions measurements over the six-month data collection period for Part 1. In some cases, it was possible to identify specific causes for the changes. For example, in February 2002, about halfway through the testing program, a new batch of fuel was delivered. Other changes might be due to engine wear or measurement issues.   If it was demonstrated that there was a statistically significant trend or a sudden change in the reference oil results, appropriate statistical adjust-ments were made to all of the corresponding emissions data. Laboratory test procedures were reviewed in an attempt to identify the factors affecting the results.

Two types of testing trends were observed in the reference oils: a sudden change in emissions for tests conducted after February 2002 and linear trends throughout the entire test period. Separate statistical models were fitted to the data for each situation. If the emissions results from the reference oil tests show a sudden change for a particular species after February 2002, the following model was fitted.

$$E_i = \beta_0 + \beta_1 \times I_i + e_i, \qquad\qquad \text{(Model 1)}$$

where E_i is the measured emissions of that species on the ith reference oil test, β_0 (the intercept) is the average emissions before February 2002, β_1 (the slope) is the change in emissions after February 2002, I_i is an indicator variable ($I_i = 1$ if the ith test was conducted after February 2002, otherwise, $I_i = 0$), and e_i = random error component. Hypotheses testing was conducted determine if there was a statistically significant change (i.e., $\beta_1 = 0$). If so, the following adjustment was made to the measured emissions of that species to remove the testing trend:

$$\text{Adjusted } E_i = E_i - B_1 \times I_i,$$

where B_1 is the estimate of the slope (β_1) in Model 1. Figure D-1 illustrates a drop in SO_2 emissions for the reference oils after February 2002, and Figure D-2 shows the effect of applying the adjustment described above to all SO_2 test results, including those obtained with the reference oil.
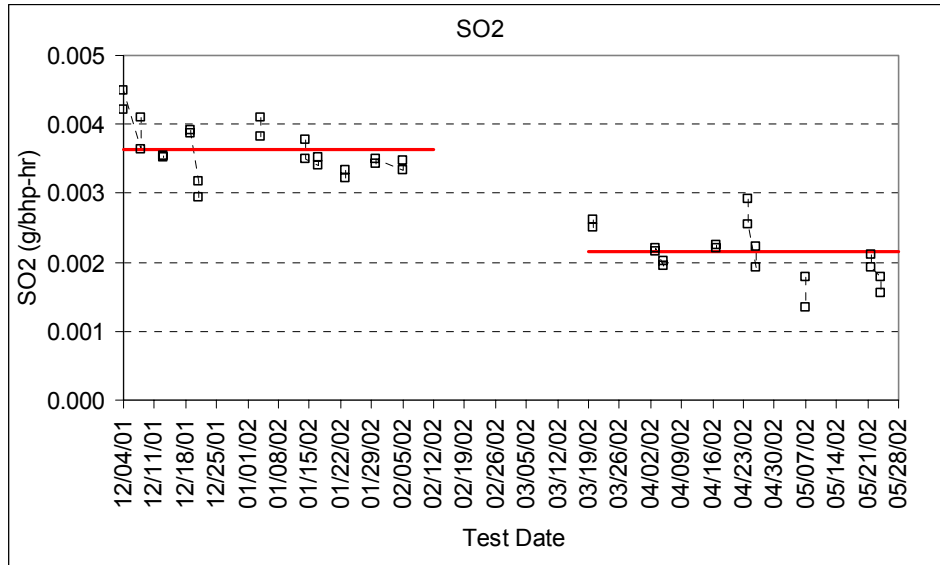
Figure D-1. A drop in SO₂ emissions for the reference oils tested after February 2002.
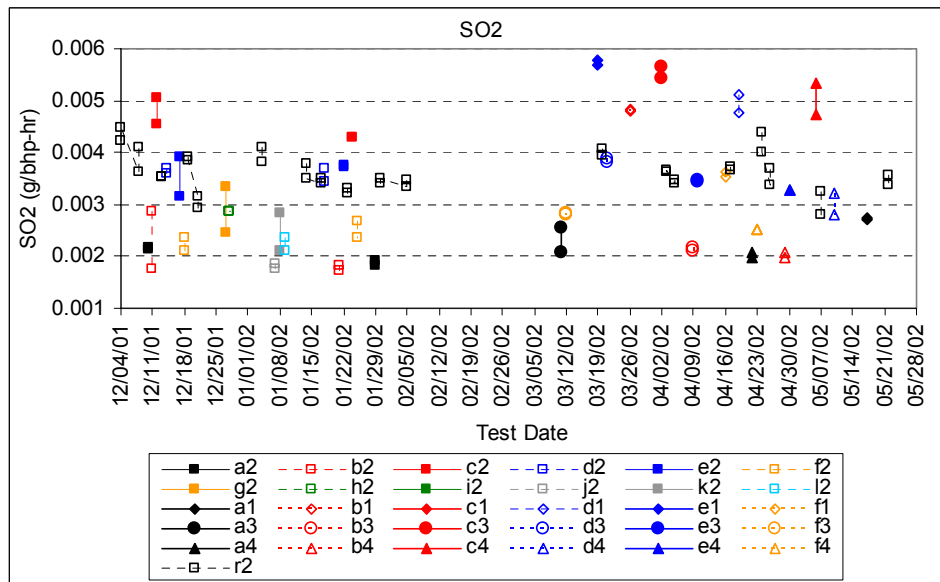
Figure D-2. SO₂ emissions adjusted for trends in the reference oil results.

When linear trends in the reference oil results were evident, the following model was fitted:

$$E_i = \beta_0 + \beta_1 \times Day_i + e_i, \qquad \text{(Model 2)}$$

where E_i is the measured emissions on the ith reference oil test, β_0 = the intercept, β_1 = the slope, Day_i is the number of test days prior to the ith reference oil test, and e_i = random error

component. Hypotheses testing was conducted to test if β_1 is different from zero (i.e., if there is a significant trend). If the trend was statistically significant for a particular emission species, the following adjustment was made to that species to remove the testing trend:

$$\text{Adjusted } E_i = E_i - B_1 \times Day_i,$$

where B_1 is the estimate of the slope (β_1). Figure D-3 illustrates a downward trend in Ca emissions for the reference oils. Figure D-4 shows the Ca emissions adjusted for the trend.
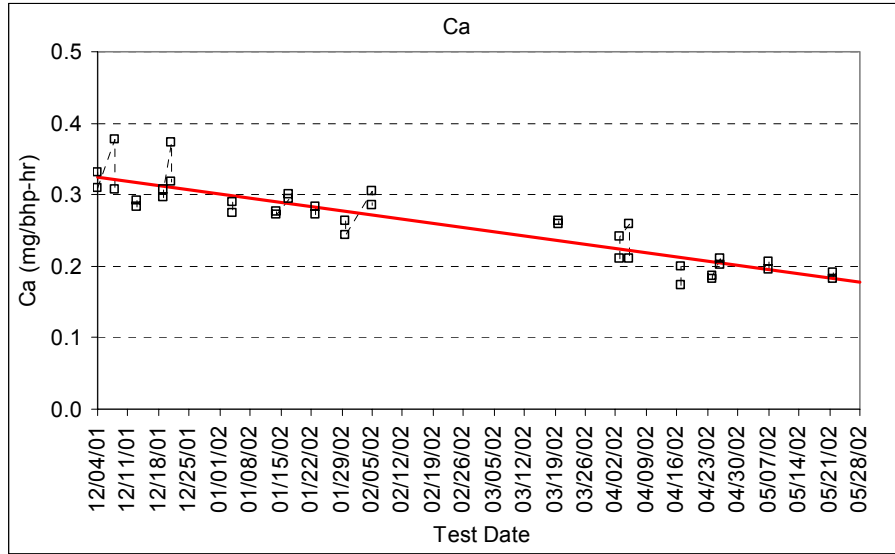

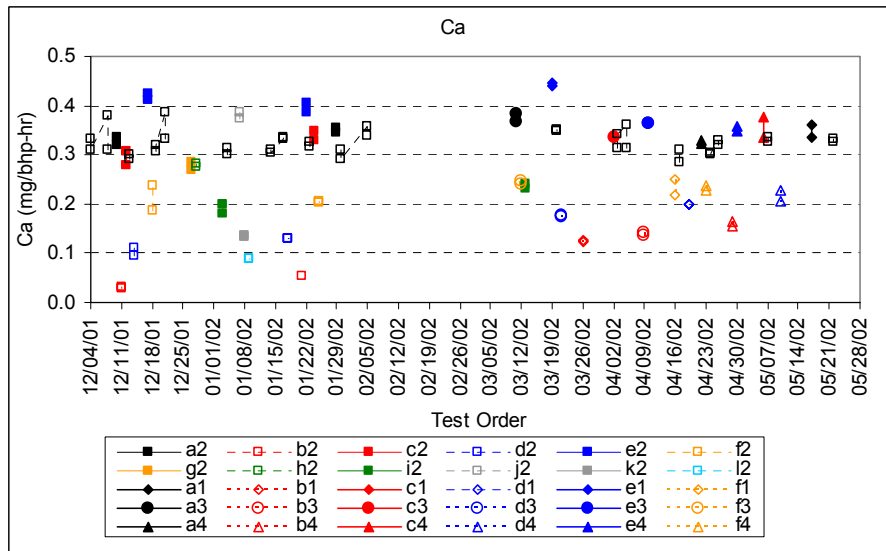*Figure D-3.   Downward trend in Ca emissions for the reference oils.*

Figure D-4.   Ca emissions adjusted for trends in the reference oil results.

This type of analysis was performed on all of the emissions species. Adjustments were made to the test data only if we found statistically significant trends in the reference oil results. The adjustments were made prior to carrying out statistical comparisons of additive and base oil effects on emissions. Table D-1 summarizes the findings of the trend analysis on the reference oil results.

Table D-1.   Summary of analysis on testing trends in the reference oils.

Emission	Unit	n	Model Fitted	Estimate Intercept	Estimate Slope	Findings
HC	(g/bhp-hr)	36	1	-	-	No significant trends.
CO	(g/bhp-hr)	38	1	0.595*	-0.049*	Significant trends: Different intercepts.
NO$_x$	(g/bhp-hr)	22	1	-	-	Too many step changes for data collected after February 2002; excluded data collected after February 2002.
SO$_2$	(g/bhp-hr)	38	1	0.004*	-0.001*	Significant trends: Different intercepts.
TPM	(g/bhp-hr)	38	1	-	-	No significant trends.
S	(mg/bhp-hr)	38	1	0.172*	-0.094*	Significant trends: Different intercepts.
Ca	(mg/bhp-hr)	38	2	0.324*	-0.001*	Significant trends: Linear trend.
Zn	(mg/bhp-hr)	38	2	0.124*	0.000*	Significant trends: Linear trend.
P	(mg/bhp-hr)	38	1	-	-	No significant trends.
Mo	(mg/bhp-hr)	38	1	-	-	No significant trends.
Si	(mg/bhp-hr)	37	1	0.840*	-0.492*	Significant trends: Different intercepts.
K	(mg/bhp-hr)	38	1	-	-	No significant trends.
Fe	(mg/bhp-hr)	38	1	-	-	No significant trends.
Pb	(mg/bhp-hr)	38	1	-	-	No significant trends.
Cu	(mg/bhp-hr)	37	1	0.007*	-0.005*	Significant trends: Different intercepts.
Cr	(mg/bhp-hr)	38	1	0.004*	0.005*	Significant trends: Different intercepts.
Cl	(mg/bhp-hr)	NA	-	-	-	Large Variations.
Mg	(mg/bhp-hr)	NA	-	-	-	Large Variations.
Na	(mg/bhp-hr)	NA	-	-	-	Large Variations.

NA = Not Analyzed.
*Significantly different from 0

Appendix E: Estimated Average Emissions of Gases and Particulate Matter Components with 95% Confidence Limits

Table E-1.   Average emissions of gases and particulates – with 95% confidence intervals – for each combination of lubricant additive package and base oil.

Additive Package	Base Oil Group	HC (g/bhp-hr)	CO (g/bhp-hr)	NOx (g/bhp-hr)	SO2 (g/bhp-hr)	TPM (g/bhp-hr)	Hydrated SO4 (g/bhp-hr)	SOF (g/bhp-hr)
a	1	0.0601 (0.0541, 0.066)	0.6249 (0.5923, 0.6575)		0.0027 (0.0023, 0.0032)	0.05 (0.0438, 0.0561)	0.0009 (-0.0005, 0.0023)	0.0047 (-0.0064, 0.0158)
a	2	0.0615 (0.0556, 0.0674)	0.6199 (0.5969, 0.643)	3.4188 (3.3381, 3.4994)	0.002 (0.0017, 0.0023)	0.051 (0.0467, 0.0554)	0 (-0.001, 0.0011)	0.0047 (-0.0032, 0.0126)
a	3	0.0636 (0.0577, 0.0696)	0.6164 (0.5838, 0.649)		0.0023 (0.0019, 0.0028)	0.0475 (0.0413, 0.0536)	0 (-0.0014, 0.0014)	0.0151 (0.004, 0.0263)
a	4	0.0576 (0.0517, 0.0635)	0.6498 (0.6172, 0.6824)		0.002 (0.0016, 0.0025)	0.0502 (0.0441, 0.0564)	0 (-0.0014, 0.0014)	0.0002 (-0.011, 0.0113)
b	1	0.0562 (0.0503, 0.0621)	0.5893 (0.5567, 0.6219)		0.0048 (0.0044, 0.0053)	0.0575 (0.0513, 0.0636)	0 (-0.0014, 0.0014)	0.0088 (-0.0023, 0.02)
b	2	0.0559 (0.0517, 0.0601)	0.5507 (0.5241, 0.5773)	3.3197 (3.239, 3.4003)	0.002 (0.0017, 0.0024)	0.0525 (0.0475, 0.0576)	0 (-0.0011, 0.0012)	0.0019 (-0.0072, 0.0109)
b	3	0.0579 (0.052, 0.0638)	0.5864 (0.5538, 0.619)		0.0021 (0.0017, 0.0026)	0.0574 (0.0512, 0.0636)	0.0004 (-0.0011, 0.0018)	0.0019 (-0.0092, 0.0131)
b	4	0.0526 (0.0467, 0.0585)	0.5584 (0.5258, 0.591)		0.002 (0.0016, 0.0025)	0.0549 (0.0488, 0.0611)	0 (-0.0014, 0.0014)	0.0045 (-0.0066, 0.0157)
c	1	0.0545 (0.0486, 0.0604)	0.665 (0.6324, 0.6976)		0.006 (0.0055, 0.0064)	0.0561 (0.05, 0.0623)	0.0018 (0.0003, 0.0032)	0.0044 (-0.0068, 0.0155)
c	2	0.0609 (0.055, 0.0669)	0.5658 (0.5428, 0.5889)	3.5148 (3.4341, 3.5954)	0.0045 (0.0042, 0.0049)	0.0518 (0.0475, 0.0562)	0.0005 (-0.0005, 0.0015)	0.0047 (-0.0032, 0.0125)
c	3	0.0514 (0.0455, 0.0573)	0.6134 (0.5808, 0.646)		0.0055 (0.0051, 0.006)	0.0564 (0.0502, 0.0626)	0 (-0.0014, 0.0014)	0.0107 (-0.0004, 0.0218)
c	4	0.0473 (0.0413, 0.0532)	0.5907 (0.5581, 0.6233)		0.005 (0.0046, 0.0055)	0.0559 (0.0497, 0.0621)	0.0001 (-0.0014, 0.0015)	0.0031 (-0.008, 0.0142)
d	1	0.0401 (0.0342, 0.046)	0.6307 (0.5981, 0.6633)		0.0049 (0.0045, 0.0054)	0.0581 (0.0519, 0.0643)	0 (-0.0014, 0.0014)	0.0004 (-0.0107, 0.0115)
d	2	0.0577 (0.0535, 0.0619)	0.5664 (0.5433, 0.5894)	3.3347 (3.254, 3.4153)	0.0036 (0.0033, 0.0039)	0.0519 (0.0476, 0.0563)	0.0006 (-0.0004, 0.0016)	0.0002 (-0.0076, 0.0081)
d	3	0.0504 (0.0445, 0.0563)	0.5721 (0.5395, 0.6047)		0.0038 (0.0034, 0.0043)	0.0535 (0.0473, 0.0596)	0 (-0.0014, 0.0014)	0.0041 (-0.007, 0.0153)
d	4	0.0471 (0.0412, 0.0531)	0.568 (0.5354, 0.6006)		0.003 (0.0026, 0.0034)	0.048 (0.0418, 0.0542)	0 (-0.0014, 0.0014)	0.004 (-0.0071, 0.0151)
e	1	0.0501 (0.0441, 0.056)	0.6045 (0.5719, 0.6371)		0.0057 (0.0053, 0.0062)	0.0534 (0.0472, 0.0596)	0 (-0.0014, 0.0014)	0.01 (-0.0011, 0.0211)
e	2	0.0539 (0.048, 0.0598)	0.6027 (0.5797, 0.6258)	3.4187 (3.3381, 3.4994)	0.0036 (0.0033, 0.0039)	0.047 (0.0427, 0.0514)	0.0002 (-0.0008, 0.0012)	0.0015 (-0.0063, 0.0094)
e	3	0.0517 (0.0458, 0.0576)	0.5685 (0.5359, 0.6011)		0.0035 (0.003, 0.0039)	0.0506 (0.0444, 0.0567)	0.005 (0.0036, 0.0065)	0.0206 (0.0095, 0.0318)
e	4	0.0529 (0.047, 0.0588)	0.5699 (0.5373, 0.6025)		(0.0028, 0.0037)			
f		(0.054, 0.0658)	(0.581, 0.6463)		0.0036 (0.0031, 0.004)	0.06 (0.0538, 0.0662)	0 (-0.0014, 0.0014)	0.0017 (-0.0094, 0.0128)
f	2			3.4238 (3.3432, 3.5045)	0.0024			
f	3	0.0723 (0.0664, 0.0782)	0.5998 (0.5672, 0.6324)		0.0028 (0.0024, 0.0033)	0.0548 (0.0486, 0.0609)	0 (-0.0014, 0.0014)	0.0313 (0.0202, 0.0424)
f			0.595 (0.5624, 0.6276)		0.0025 (0.0021, 0.003)	0.0502 (0.0441, 0.0564)	0 (-0.0014, 0.0014)	0.0021 (-0.0091, 0.0132)
g	2	0.0543 (0.0484, 0.0602)	0.649 (0.6164, 0.6816)	3.323 (3.2089, 3.4371)	0.0029 (0.0025, 0.0034)	0.0449 (0.0387, 0.0511)	0 (-0.0014, 0.0014)	0.0008 (-0.0103, 0.0119)
h	2	0.0662 (0.0603, 0.0721)	0.6057 (0.5731, 0.6383)	3.1742 (3.0602, 3.2883)	0.0029 (0.0024, 0.0033)	0.0435 (0.0373, 0.0496)	0.0001 (-0.0014, 0.0015)	0.0035 (-0.0076, 0.0146)
i	2	0.0653 (0.0612, 0.0695)	0.5886 (0.5655, 0.6116)	3.2064 (3.0923, 3.3204)		0.0526 (0.0482, 0.057)	0.0003 (-0.0007, 0.0013)	0.0145 (0.0067, 0.0224)
j	2	0.0439 (0.0379, 0.0498)	0.5981 (0.5655, 0.6307)	3.3318 (3.2178, 3.4459)	0.0018 (0.0014, 0.0023)	0.0423 (0.0361, 0.0485)	0 (-0.0014, 0.0014)	0.0017 (-0.0094, 0.0128)
k	2	0.0622 (0.0563, 0.0681)	0.6086 (0.576, 0.6412)	3.3964 (3.2824, 3.5105)	0.0025 (0.002, 0.0029)	0.047 (0.0408, 0.0532)	0 (-0.0014, 0.0014)	0 (-0.0111, 0.0112)
l	2	0.0536 (0.0477, 0.0595)	0.588 (0.5554, 0.6206)	3.0377 (2.9236, 3.1517)	0.0022 (0.0018, 0.0027)	0.0526 (0.0464, 0.0588)	0 (-0.0014, 0.0014)	0.0023 (-0.0088, 0.0135)
r	2 (Pre-Aging)	0.0573 (0.0556, 0.059)	0.594 (0.585, 0.6031)	3.2758 (3.231, 3.3205)	0.0037 (0.0036, 0.0038)	0.0509 (0.0492, 0.0526)	0.0003 (0, 0.0007)	0.0045 (0.0014, 0.0076)
r	2 (Post-Aging)	0.0564 (0.0539, 0.0589)	0.5984 (0.5851, 0.6117)	3.2914 (3.2344, 3.3484)	0.0035 (0.0033, 0.0036)	0.0541 (0.0516, 0.0566)	0 (-0.0005, 0.0006)	0.0067 (0.0022, 0.0113)

Table E-2. *Average emissions of target elements – with 95% confidence intervals – for each combination of lubricant additive package and base oil.*

Additive Package	Base Oil Group	S (mg/bhp-hr)	Ca (mg/bhp-hr)	Zn (mg/bhp-hr)	P (mg/bhp-hr)	Mo (mg/bhp-hr)
a	1	0.1696 (0.1437, 0.1954)	0.3481 (0.3222, 0.374)	0.0646 (0.0516, 0.0777)	0.072 (0.0447, 0.0994)	0 (-0.0013, 0.0013)
	2	0.1773 (0.159, 0.1956)	0.339 (0.3207, 0.3572)	0.0295 (0.0203, 0.0388)	0.0252 (0.0059, 0.0446)	0.0009 (0, 0.0019)
	3	0.1722 (0.1463, 0.198)	0.3751 (0.3492, 0.401)	0.039 (0.0259, 0.0521)	0.0493 (0.022, 0.0766)	0 (-0.0013, 0.0013)
	4	0.1551 (0.1293, 0.181)	0.3262 (0.3003, 0.352)	0.054 (0.0409, 0.067)	0.0508 (0.0234, 0.0781)	0 (-0.0013, 0.0013)
b	1	0.1991 (0.1732, 0.2249)	0.1248 (0.0989, 0.1507)	0.047 (0.034, 0.0601)	0.0973 (0.07, 0.1246)	0.0004 (-0.0009, 0.0018)
	2	0.1788 (0.1577, 0.1999)	0.0383 (0.0172, 0.0594)	0.0228 (0.0121, 0.0335)	0.0511 (0.0288, 0.0734)	0.0009 (-0.0002, 0.002)
	3	0.1482 (0.1224, 0.174)	0.1396 (0.1138, 0.1655)	0.0483 (0.0352, 0.0614)	0.1054 (0.0781, 0.1327)	0 (-0.0013, 0.0013)
	4	0.153 (0.1271, 0.1788)	0.1612 (0.1353, 0.187)	0.0537 (0.0406, 0.0667)	0.0991 (0.0718, 0.1264)	0 (-0.0013, 0.0013)
c	1					
	2	0.2052 (0.1869, 0.2234)	0.3158 (0.2975, 0.3341)	0.0195 (0.0102, 0.0287)	0.3935 (0.3741, 0.4128)	0.0008 (-0.0001, 0.0018)
	3	0.1951 (0.1585, 0.2316)	0.3352 (0.2986, 0.3718)	0.0425 (0.024, 0.061)	0.3503 (0.3117, 0.389)	0 (-0.0019, 0.0019)
	4	0.1886 (0.1627, 0.2144)	0.357 (0.3311, 0.3828)	0.0581 (0.045, 0.0711)	0.4824 (0.4551, 0.5097)	0 (-0.0013, 0.0013)
d	1	0.1879 (0.1621, 0.2137)	0.1991 (0.1732, 0.225)	0.1637 (0.1506, 0.1767)	0.2507 (0.2234, 0.278)	0.0014 (0.0001, 0.0028)
	2	0.1997 (0.1814, 0.218)	0.1166 (0.0984, 0.1349)	0.1749 (0.1656, 0.1841)	0.2621 (0.2428, 0.2814)	0.0009 (0, 0.0019)
	3	0.1738 (0.148, 0.1997)	0.1754 (0.1496, 0.2013)	0.155 (0.1419, 0.168)	0.2482 (0.2209, 0.2755)	0.0004 (-0.001, 0.0017)
	4	0.1688 (0.143, 0.1946)	0.2169 (0.191, 0.2427)	0.1513 (0.1383, 0.1644)	0.2477 (0.2203, 0.275)	0 (-0.0013, 0.0013)
e	1	0.2054 (0.1795, 0.2312)	0.4424 (0.4166, 0.4683)	0.1234 (0.1103, 0.1365)	0.1594 (0.1321, 0.1867)	0.0069 (0.0056, 0.0082)
	2	0.1959 (0.1776, 0.2141)	0.4069 (0.3886, 0.4251)	0.1266 (0.1174, 0.1359)	0.1848 (0.1655, 0.2041)	0.0108 (0.0098, 0.0117)
	3	0.1691 (0.1432, 0.1949)	0.3638 (0.338, 0.3897)	0.1122 (0.0991, 0.1252)	0.1639 (0.1366, 0.1912)	0.0007 (-0.0007, 0.002)
	4	0.1553 (0.1295, 0.1812)	0.3524 (0.3265, 0.3783)	0.1126 (0.0996, 0.1257)	0.1494 (0.1221, 0.1767)	0.0057 (0.0043, 0.007)
f	1	0.19 (0.1641, 0.2158)	0.2345 (0.2086, 0.2603)	0.0784 (0.0653, 0.0914)	0.0764 (0.0491, 0.1037)	0.0008 (-0.0006, 0.0021)
	2	0.1766 (0.1583, 0.1949)	0.2079 (0.1896, 0.2262)	0.0775 (0.0682, 0.0867)	0.1008 (0.0815, 0.1202)	0.0016 (0.0006, 0.0025)
	3	0.1964 (0.1706, 0.2223)	0.2437 (0.2179, 0.2696)	0.0774 (0.0644, 0.0905)	0.0784 (0.0511, 0.1057)	0 (-0.0013, 0.0013)
	4	0.1589 (0.133, 0.1847)	0.2321 (0.2063, 0.258)	0.082 (0.0689, 0.0951)	0.0866 (0.0593, 0.1139)	0 (-0.0013, 0.0013)
g	2	0.1851 (0.1592, 0.2109)	0.2777 (0.2518, 0.3036)	0.1278 (0.1147, 0.1408)	0.1969 (0.1696, 0.2242)	0.0013 (0, 0.0026)
h	2	0.1423 (0.1165, 0.1682)	0.2777 (0.2518, 0.3035)	0.0749 (0.0618, 0.0879)	0.1319 (0.1046, 0.1592)	0.0165 (0.0152, 0.0179)
i	2	0.2675 (0.2492, 0.2858)	0.2132 (0.1949, 0.2314)	0.0308 (0.0216, 0.04)	0.022 (0.0027, 0.0413)	0.0018 (0.0009, 0.0028)
j	2	0.1898 (0.1639, 0.2156)	0.3793 (0.3534, 0.4051)	0.0253 (0.0123, 0.0384)	0.0349 (0.0076, 0.0622)	0.0004 (-0.001, 0.0017)
k	2	0.178 (0.1521, 0.2038)	0.1346 (0.1088, 0.1605)	0.045 (0.0319, 0.058)	0.0278 (0.0005, 0.0551)	0.0012 (-0.0001, 0.0025)
l	2	0.2066 (0.1808, 0.2325)	0.0888 (0.0629, 0.1147)	0.1084 (0.0954, 0.1215)	0.0491 (0.0218, 0.0764)	0.0005 (-0.0008, 0.0019)
r	2 (Pre-Aging)	0.1764 (0.1693, 0.1836)	0.3156 (0.3084, 0.3227)	0.123 (0.1194, 0.1266)	0.2163 (0.2088, 0.2239)	0.0002 (-0.0001, 0.0006)
	2 (Post-Aging)	0.1618 (0.1512, 0.1723)	0.3417 (0.3311, 0.3522)	0.1268 (0.1215, 0.1322)	0.1836 (0.1724, 0.1947)	0.0002 (-0.0003, 0.0008)

Table E-3.   Average emissions of trace elements – with 95% confidence intervals – for each combination of lubricant additive package and base oil.

Additive Package	Base Oil	Si (mg/bhp-hr)	K (mg/bhp-hr)	Fe (mg/bhp-hr)	Pb (mg/bhp-hr)	Cu (mg/bhp-hr)	Cr (mg/bhp-hr)
a	1	0.8412 (0.6448, 1.0376)	0.0037 (-0.0026, 0.0099)	0.0435 (0.0126, 0.0745)	0.0035 (0.001, 0.006)	0.0075 (-0.0036, 0.0185)	0.0071 (0.0031, 0.0111)
a	2	1.048 (0.9091, 1.1868)	0.0031 (-0.0013, 0.0075)	0.0354 (0.0135, 0.0573)	0.0013 (-0.0005, 0.0031)	0.0062 (-0.0017, 0.014)	0.002 (-0.0009, 0.0048)
a	3	1.2579 (1.0615, 1.4543)	0.0009 (-0.0053, 0.0072)	0.0153 (-0.0156, 0.0463)	0.0011 (-0.0014, 0.0036)	0.0073 (-0.0038, 0.0184)	0 (-0.004, 0.004)
a	4	(0.6093, 1.0021)	0.006 (-0.0003, 0.0122)	0.0392 (0.0083, 0.0702)	0.0018 (-0.0007, 0.0043)	0.0061 (-0.005, 0.0172)	0.0044 (0.0004, 0.0085)
b	1	0.8937 (0.6973, 1.0901)	0 (-0.0063, 0.0062)	0.0382 (0.0073, 0.0692)	0.0039 (0.0014, 0.0064)	0.0085 (-0.0025, 0.0196)	0.0018 (-0.0023, 0.0058)
b	2	1.0929 (0.9325, 1.2533)	0.0033 (-0.0019, 0.0083)	0.0381 (0.0128, 0.0633)	0.0006 (-0.0014, 0.0026)	0.0061 (-0.0029, 0.0152)	0.0028 (-0.0005, 0.0061)
b	3	0.8487 (0.6523, 1.0451)	0.0044 (-0.0018, 0.0107)	0.0397 (0.0087, 0.0706)	0.001 (-0.0015, 0.0035)	0.0077 (-0.0034, 0.0188)	0.0038 (-0.0002, 0.0079)
b	4	0.8171 (0.6207, 1.0135)	0.0017 (-0.0046, 0.0079)	0.028 (-0.003, 0.0589)	0.0042 (0.0017, 0.0067)	0.0073 (-0.0038, 0.0184)	0.0026 (-0.0014, 0.0066)
c	1						
c	2	0.8869 (0.748, 1.0258)	0.0001 (-0.0043, 0.0045)	0.0546 (0.0327, 0.0765)	0.0002 (-0.0016, 0.0019)	0.0066 (-0.0013, 0.0144)	0.0029 (0.0001, 0.0057)
c	3	0.5723 (0.2946, 0.8501)	0 (-0.0088, 0.0088)	0.0284 (-0.0154, 0.0722)	0.0023 (-0.0013, 0.0058)	0.0062 (-0.0095, 0.0219)	0.0018 (-0.0039, 0.0075)
c		0.7034 (0.507, 0.8998)	0.0009 (-0.0053, 0.0072)	0.0487 (0.0178, 0.0797)	0.0015 (-0.001, 0.004)	0.0061 (-0.005, 0.0172)	0.0101 (0.0061, 0.0142)
d	1	0.8729 (0.6765, 1.0693)	0.0065 (0.0003, 0.0128)	0.045 (0.014, 0.0759)	0.0029 (0.0004, 0.0054)	0.0079 (-0.0032, 0.019)	0.0058 (0.0018, 0.0098)
d	2	0.9013 (0.7624, 1.0402)	0 (-0.0044, 0.0044)	0.0255 (0.0036, 0.0474)	0.0001 (-0.0016, 0.0019)	0.0153 (0.0074, 0.0231)	0.0019 (-0.0009, 0.0048)
d	3	(0.7217, 1.1145)	0.0067 (0.0005, 0.013)	0.0276 (-0.0034, 0.0585)	0.0046 (0.0021, 0.0071)	0.0084 (-0.0027, 0.0195)	0.0039 (-0.0001, 0.0079)
d	4	0.7115 (0.5151, 0.9079)	0 (-0.0063, 0.0062)	0.0304 (-0.0006, 0.0614)	0.0006 (-0.0019, 0.003)	0.0068 (-0.0043, 0.0178)	0.0022 (-0.0018, 0.0062)
	1	1.0868 (0.8904, 1.2832)	0.0018 (-0.0044, 0.0081)	0.013 (-0.018, 0.044)	0.0035 (0.001, 0.006)	0.007 (-0.0041, 0.0181)	0.0024 (-0.0016, 0.0064)
	2	0.8598 (0.721, 0.9987)	0.0002 (-0.0042, 0.0046)	0.0357 (0.0138, 0.0576)	0.0002 (-0.0016, 0.002)	0.0056 (-0.0023, 0.0134)	0.0033 (0.0004, 0.0061)
	3	0.8672 (0.6708, 1.0636)	-0.0028, 0.0097)	0.0244 (-0.0065, 0.0554)	0.0011 (-0.0014, 0.0036)	0.0065 (-0.0046, 0.0176)	0.0026
	4	0.808 (0.6116, 1.0044)	0.0001 (-0.0062, 0.0063)	0.0275 (-0.0035, 0.0585)		0.0066 (-0.0045, 0.0177)	0.0041 (0.0001, 0.0081)
f	1	0.8903	(-0.0063, 0.0062)	0.0276 (-0.0034, 0.0585)	0.0021 (-0.0004, 0.0046)	0.0058 (-0.0053, 0.0169)	0.004 (0, 0.008)
f	2	1.0256 (0.8867, 1.1644)	0.0022 (-0.0022, 0.0067)	0.035 (0.0131, 0.0569)	0.0013 (-0.0005, 0.003)	0.0053 (-0.0026, 0.0131)	0.0037 (0.0009, 0.0065)
f	3	1.3507 (1.1543, 1.5471)	0.0033 (-0.0029, 0.0096)	0.0116 (-0.0194, 0.0425)	0.0051 (0.0026, 0.0076)	0.0061 (-0.005, 0.0172)	0.0001 (-0.0039, 0.0041)
f	4	0.8408 (0.6444, 1.0372)	0.0036 (-0.0027, 0.0098)	0.0413 (0.0104, 0.0723)	0.0004 (-0.0021, 0.0029)	0.006 (-0.0051, 0.0171)	0.0064 (0.0024, 0.0105)
g	2	0.8879 (0.6915, 1.0843)	0.0013 (-0.0049, 0.0076)	0.0298 (-0.0012, 0.0607)	0.0013 (-0.0012, 0.0038)	0.0201 (0.009, 0.0312)	0.003 (-0.0011, 0.007)
h	2	0.8455 (0.6491, 1.0419)	0.0007 (-0.0056, 0.0069)	0.0353 (0.0043, 0.0662)	0 (-0.0025, 0.0025)	0.0075 (-0.0036, 0.0186)	0.0019 (-0.0022, 0.0059)
i	2	1.148 (1.0091, 1.2868)	0.0014 (-0.003, 0.0058)	0.0301 (0.0082, 0.052)	0.0028 (0.001, 0.0046)	0.0065 (-0.0013, 0.0143)	0.0016 (-0.0012, 0.0045)
j	2	0.8501 (0.6537, 1.0465)	0 (-0.0063, 0.0062)	0.042 (0.0111, 0.073)	0.0008 (-0.0017, 0.0033)	0.0102 (-0.0009, 0.0213)	0.0056 (0.0016, 0.0096)
k	2	0.9178 (0.7214, 1.1142)	0.0633 (0.057, 0.0695)	0.0442 (0.0132, 0.0752)	0.003 (0.0005, 0.0055)	0.0454 (0.0343, 0.0565)	0.0033 (-0.0007, 0.0073)
l	2	1.0121 (0.8157, 1.2085)	0.0053 (-0.001, 0.0115)	0.0491 (0.0181, 0.08)	0.0081 (0.0056, 0.0106)	0.1194 (0.1083, 0.1305)	0.0051 (0.001, 0.0091)
r	2 (Pre-Aging)	0.8339 (0.7795, 0.8884)	0.0027 (0.0009, 0.0044)	0.0384 (0.0298, 0.047)	0.0015 (0.0008, 0.0022)	0.007 (0.0039, 0.0101)	0.0036 (0.0025, 0.0047)
r	2 (Post-Aging)	0.8551 (0.7713, 0.9388)	0.0039 (0.0013, 0.0064)	0.0606 (0.048, 0.0733)	0.001 (0, 0.0021)	0.0068 (0.0023, 0.0114)	0.0054 (0.0037, 0.007)

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Appendix F: PAH Results

This appendix presents results from the analysis of PM-bound PAHs, which was included as part of the overall APBF-DEC assessment of potentially toxic emissions. However, these data were not inherent to the lubricants project experimental design. As a result, they were not screened for statistical outliers, nor were they statistically analyzed. They are presented here only for reference.
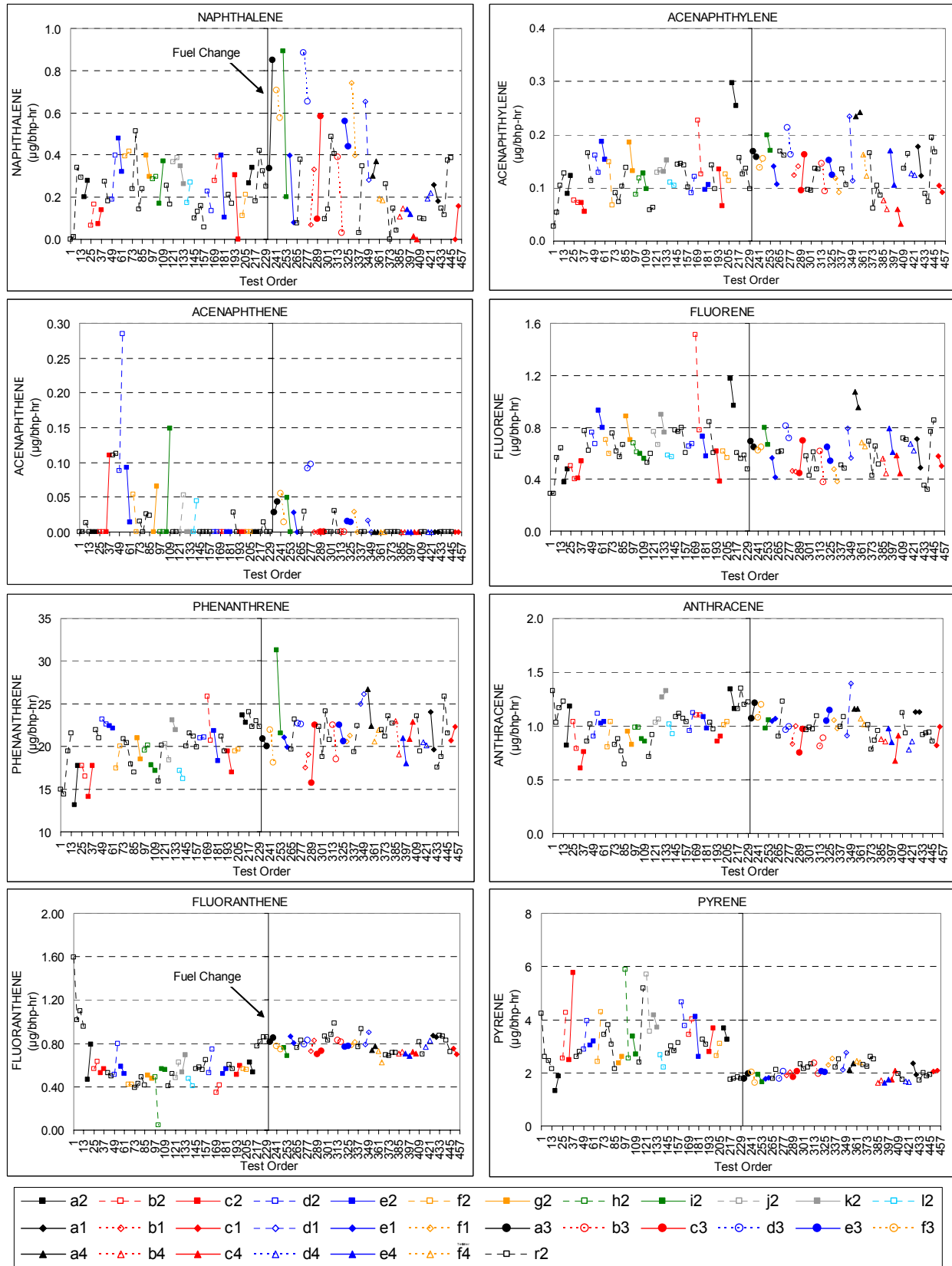
Figure F-1. Plots of particulate-bound emissions of PAHs versus test order – by test oil.
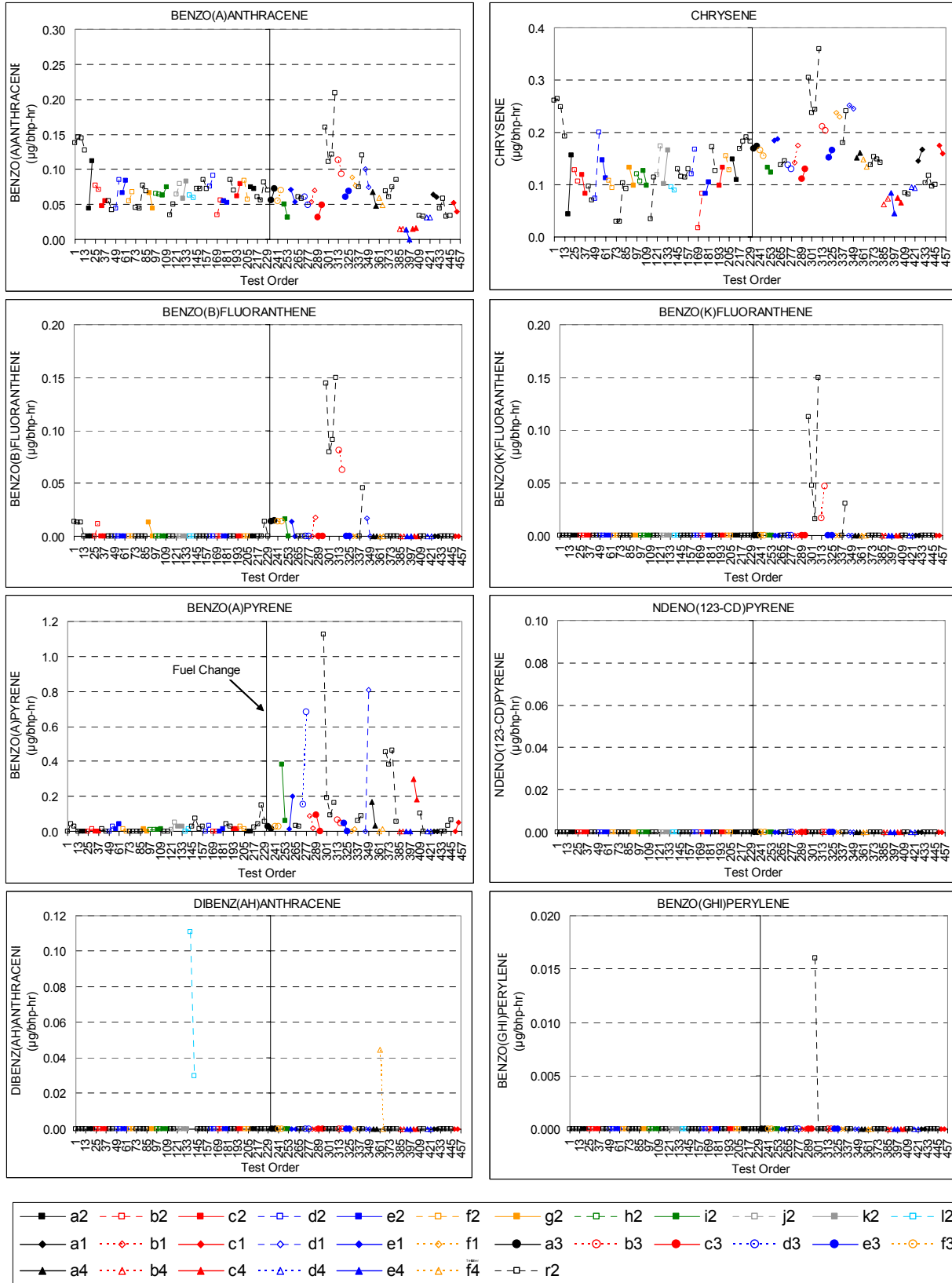
Figure F-1. Plots of particulate-bound emissions of PAHs versus test order – by test oil (continued).

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