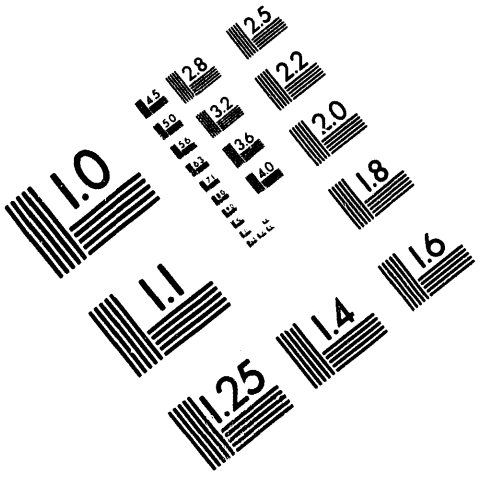
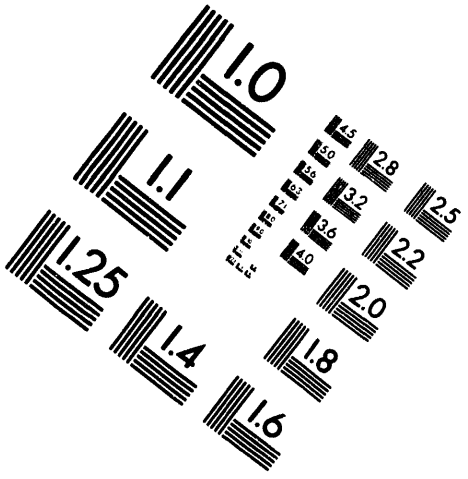


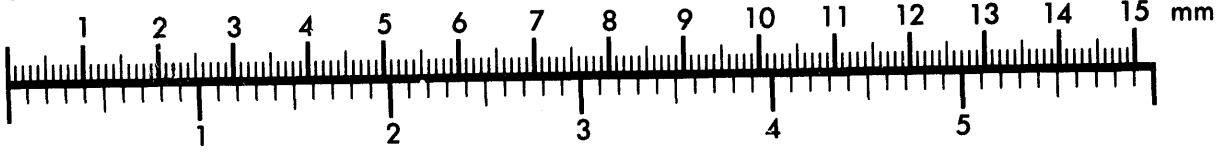
Association for Information and Image Management

1100 Wayne Avenue, Suite 1100
Silver Spring, Maryland 20910

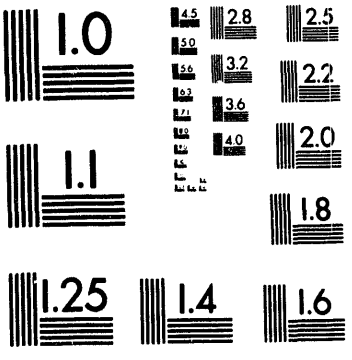
301/587-8202

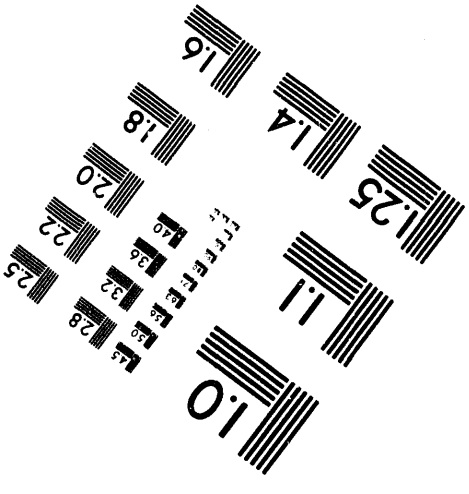


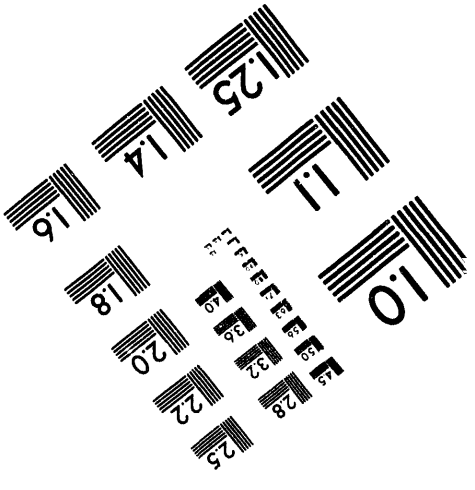
Centimeter



Inches







MANUFACTURED TO AIIM STANDARDS
BY APPLIED IMAGE, INC.

1 of 1

DOE/NASA/0373-12
NASA CR-191197

Development of High Temperature Liquid Lubricants For Low-Heat Rejection Heavy Duty Diesel Engines

P.D. Wiczynski
T.A. Marolewski
Cummins Engine Company, Inc.
Columbus, Indiana 47201

March 1993

Prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Lewis Research Center
Contract DEN3-373
Under Interagency Agreement DE-AI01-91CE50306

for
U.S. DEPARTMENT OF ENERGY
Office of Transportation Technologies
Washington, D.C. 20545

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

TABLE OF CONTENTS

	Page
LIST OF FIGURES	4
LIST OF TABLES	7
1.0 SUMMARY	9
2.0 INTRODUCTION	10
2.1 Previous LHR Lubricant Development	11
2.2 Present Program Objectives and Scope	12
3.0 TASK I - LUBRICATION SPECIFICATIONS/ENGINE REQUIREMENTS	12
4.0 TASK II - LABORATORY TESTING AND DEVELOPMENT	13
4.1 Laboratory Bench Tests	16
4.2 Initial Tests	17
4.2.1 Base Stock Deposit	17
4.2.2 Lubricant Deposits	19
4.2.3 Modified Bench Tests	20
4.3 Lubricant Formulation	23
4.3.1 Lubricant Deposit Composition	23
4.3.2 Additive Package Development	24
4.3.3 High Viscosity Index Base Stocks	30
4.4 Wear Studies of Aromatic Ester Based Lubricants	33
4.5 Evaluation of Acid Absorbing Media For Chemically Reactive Filter	37
4.6 Subcontracted Lubricant Evaluation and Development	40
4.6.1 Penn State Lubricant Evaluation and Development	40
4.6.2 NIST Lubricant Evaluation and Development	44
5.0 TASK III - HIGH TEMPERATURE LUBRICANT ENGINE TESTING	47
5.1 Engine Test	47
5.2 Initial Lubricant Tests	48
5.3 Results From Initial Lubricant Tests	50
5.4 Final Lubricant Tests	57

Page

6.0 CONCLUSIONS 65

7.0 RECOMMENDATIONS 66

8.0 ACKNOWLEDGEMENTS 67

9.0 APPENDICES 68

9.1 Cummins L10 Engine Cylinder Head Cooling Modifications 68

9.2 Lubricant Engine Test Data Summary 69
9.2.1 Engine Test HTL-1 69
9.2.2 Engine Test HTL-2 70
9.2.3 Engine Test HTL-3 70
9.2.4 Engine Test HTL-4 71
9.2.5 Engine Test HTL-5 73

LIST OF FIGURES

Figure No.	Description	Page
4.1	Lubricant Deposit Temperature Profile for SDL-1 vs. SDL-3	21
4.2	Deposit Temperature Profile Reproducibility of SDL-3	22
4.3	Base Stock Deposit Temperature Profile Aromatic Ester vs. Polyol Ester	23
4.4	Falex Panel Coker Deposit Composition	24
4.5	Lubricant Stability Characteristics: Alcor Deposition Test	25
4.6	Lubricant Deposit Temperature Profile Effect of Additives and Viscosity	29
4.7	Lubricant Deposit Temperature Profile of Aliphatic Diester and Aromatic Ester	31
4.8	Effect of Aryl ZDP on Wear Scar	35
4.9	Effect of Alkyl ZDP on Wear Scar	36
5.1	Average Oil Consumption (Initial Engine Operating Conditions)	51
5.2	HTL-1: Viscosity and Soot Increase	52
5.3	HTL-2: Viscosity and Soot Increase	52
5.4	HTL-3: Viscosity and Soot Increase	53
5.5	TBN Decrease (Initial Engine Operating Conditions)	53
5.6	Ring Wear (Initial Engine Operating Conditions)	54

Figure No.	Description	Page
5.7	Iron Concentration Increase (Initial Engine Operating Conditions)	54
5.8	Lead Concentration Increase (Initial Engine Operating Conditions)	55
5.9	Piston Ring Groove Deposit Ratings (Initial Engine Operating Conditions)	55
5.10	Piston Land Deposit Ratings (Initial Engine Operating Conditions)	56
5.11	Steady State Particulate Emissions (Initial Engine Operating Conditions)	56
5.12	Soot Increase (Final Engine Operating Conditions)	59
5.13	Average Oil Consumption (Final Engine Operating Conditions)	59
5.14	40°C Viscosity Increase (Final Engine Operating Conditions)	60
5.15	100°C Viscosity Increase (Final Engine Operating Conditions)	60
5.16	TBN Decrease (Final Engine Operating Conditions)	61
5.17	Ring Wear (Final Engine Operating Conditions)	61
5.18	Iron Concentration Increase (Final Engine Operating Conditions)	63

Figure No.	Description	Page
5.19	Lead Concentration Increase (Final Engine Operating Conditions)	62
5.20	Piston Ring Groove Deposit Ratings (Final Engine Operating Conditions)	63
5.21	Piston Land Deposit Ratings (Final Engine Operating Conditions)	63
5.22	Steady State Particulate Emissions (Final Engine Operating Conditions)	64
9.1	Cylinder Head Cooling Schematic	68

LIST OF TABLES

Table No.	Description	Page
2.1	Reference LHR Lubricants	11
4.0	Lubricant Formulations Investigated	15
4.1	Summary of Base Stock Deposition Studies: Falex Panel Coker	18
4.2	Base Stock Study: Alcor Deposition Test	19
4.3	Effects of Base Stock and Additive Package on Lubricant Deposition Characteristics: Falex Panel Coker and Alcor	20
4.4	Lubricant Formulation Studies: Alcor Deposition Test	26
4.5	Aromatic Diester Formulation Studies: Alcor Deposition Test	27
4.6	Aromatic Ester Formulation Studies and Effect of Viscosity: Alcor Deposition Test	28
4.7	Ester Stability Comparison and Deposit Performance of Improved Viscosity Index Lubricant, HTL-2: Alcor Deposition Test	32
4.8	Composition of HTL-2	33
4.9	Wear Properties of SDL-2 and SDL-3: Four Ball Wear Tests	34
4.10	ZDP Activity in Aromatic Ester Formulations: Four-Ball Wear Tests	37
4.11	Evaluation of Sulfur Dioxide Adsorbing/ Characteristics of Various Filter Media (@ 25°C)	38
4.12	Effect of Filter Media on Sulfur Dioxide Treated Lubricants	39

Table No.	Description	Page
4.13	Effect of Zeolite H on Additive Levels of Experimental-4	40
4.14	Determination of Lubricant Deposit Formation With the Microoxidation Test	41
4.15	Determination of Lubricant Retention and Deposit Formation With the Microoxidation Test	42
4.16	Effect of Copper Additive on Lubricant Retention and Deposit Formation: Microoxidation Test at 250°C	43
4.17	Effect of Copper Additive on Lubricant Retention and Deposit Formation: Microoxidation Test at 250°C	44
4.18	Lubricant Deposit and Oxidation Stability	45
4.19	Performance Comparison of HTL-2 and HTL-5	46
5.1	L10 Multi-Cylinder Engine Specifications	47
5.2	Engine Test Components	48
5.3	Initial Engine Operating Conditions	48
5.4	Cylinder Kit Temperatures at Initial Test Conditions	49
5.5	Initial Test Lubricant Properties	50
5.6	Final Engine Operating Conditions	57
5.7	Cylinder Kit Temperatures at Final Test Conditions	58
5.8	Final Test Lubricant Properties	58

1.0 SUMMARY

The objective of this Department of Energy (DOE) program was to develop a liquid lubricant that will allow advanced diesel engines to operate at top ring reversal temperatures approaching 500°C and lubricant sump temperatures approaching 250°C. In order to achieve those goals, the following technologies were considered critical to the development of the high temperature lubricant.

1) Development of appropriate bench and rig tests to guide the lubricant formulation.
2) Development of additive packages with low ash levels to avoid abrasive oxide formation or possibly use chemically active filtration to remove corrosive by-products and augment the additive package.
3) Control of high temperature wear over a broad temperature range
4) Control of deposits through base stocks with high temperature stability that decompose with minimum residue.
5) Viscosity control over a wide operating temperature range.

The lubricant development program started with literature studies and surveys to benchmark the state of the art and projected advances for diesel engines and lubricants. Concurrent with the generation of this background information, laboratory work was initiated on the identification of bench tests to be used for selecting and refining lubricants for high temperature engine tests.

The lubricants evaluated in an earlier Cummins-Akzo project were used as references to calibrate these bench tests. In addition, Akzo independently funded programs at Penn State and NIST to develop bench tests and high temperature lubricants to supplement the DOE-funded work. Procedures were developed with the Falex panel coker to measure deposit characteristics using the reference lubricants as bench marks. The Alcor deposition test was selected as a general measure of lubricant stability and dispersancy. Bench test correlation with the reference lubricants was also achieved at Penn State with the microoxidation test and at NIST with the two-peak differential scanning calorimetry (DSC) deposit test.

Base stock screening was initiated early in the program. All bench tests identified the aromatic esters used in the best reference lubricant as having the lowest deposit forming characteristics. Lubricant formulation studies centered on this class of base stock with the ultimate goal of achieving an ashless additive package. Previous Cummins-Akzo engine tests identified ash generating additives as deposit precursors in the upper ring belt. While the need for acid scavenging additives in the test lubricants is minimized by the use of low sulfur fuel, an external acid absorbing filter was investigated as an alternative to standard ash generating overbased detergents. Since it was anticipated that the external filter system would not be completed prior to the initial engine test series, prototype high temperature formulations were low ash rather than ashless, incorporating a minimum level of overbased detergent to prevent corrosion. Bench tests indicated that this compromise would still yield lubricants with significantly lower deposits.

While engine test development was proceeding, lubricant formulation studies continued with the objective of achieving a low deposit, multi-grade high temperature lubricant. This option was explored because of the poor viscosity index of the aromatic esters. There was a concern that their poor low temperature properties would make them impractical and their low viscosity at high bearing temperatures would not provide adequate lubrication. Formulations were developed based on blends of aromatic esters and other high viscosity index esters. These blends were fortified with additive packages derived from the work at Akzo, Penn State and NIST.

Baseline engine tests were performed on a fully instrumented production L10 using a premium 15W-40 petroleum based lubricant. The cylinder liners were not cooled and the oil sump was held at 150°C. The engine was overfueled to increase operating temperatures. Templug data indicated a top land temperature of 397°C. The test was terminated after 40 hours due to low oil pressure. This was later found to be a mechanical rather than oil related problem. However, the significant ring/liner distress observed showed that this lubricant was not providing adequate lubrication. Inadequate lubrication may have been related to excessive volatility as indicated by an oil consumption rate of over 4.4 kg/hr. The rate of soot increase in the oil was approximately 1% for every 10 hours of engine operation due to the overfueling to achieve the higher component temperatures. High levels (8%) of soot in the oil made analysis of the lubricants impossible. Engine operating conditions were modified to reduce the soot level of the oil. Connecting rod bearing problems necessitated a reduction of sump temperature to 120°C. The top land was at 362°C. The oil soot level was reduced to 1% after 100 hours with these changes.

The final two test lubricants were 15W-40 multi-grade oils utilizing the same base stock system. One employed a low ash additive package with organo-copper inhibitor. The other used an additive package developed in conjunction with NIST. Both lubricants completed the 100 hour test with satisfactory wear except for the piston pin bushing. This may be attributed to restricted oil flow to the area in an attempt to reduce oil cooling of the piston undercrown.

In summary, these lubricants functioned satisfactorily at the highest operating temperatures attainable within the design constraints of the test engine. The lubricants demonstrated a marginal increase in sump temperature capability, approximately 15°C, and an increase in top ring reversal temperature over commercial engine operation.

2.0 INTRODUCTION

The primary factors driving improvements in the design of heavy duty diesel engines today include improved fuel economy, reduced emissions, and higher reliability. The low heat rejection (LHR) diesel engine offers the potential for addressing all of these requirements by reducing the heat loss to the cooling system and raising in cylinder operating temperatures. The LHR engine will reduce fuel consumption by increasing the thermal efficiency of combustion and by providing for higher temperature exhaust. Turbo compounding can be used to extract the energy from this exhaust which is currently being wasted as heat dissipated through the engine cooling system. However, substantial engine system design challenges must be overcome before the benefits of the LHR engine can be realized. The development of new high temperature lubricants must be considered as an integral factor in the design and construction of this new class of diesel engines.

2.1 Previous LHR Lubricant Development

In the early 1980's Cummins was conducting an in cylinder materials development program using a multi-cylinder, uncooled Cummins NTC 250 engine. In the course of that program, various synthetic lubricants developed by Akzo were evaluated under high temperature operating conditions. A generic description of the lubricants used in this initial study is given in Table 2.1.

Table 2.1

Reference LHR Lubricants

	SDL-1	SDL-2	SDL-3
Base Stock	Polyol Ester	Aromatic Ester	Aromatic Ester
Additive Package	Conventional	Conventional	Conventional + Triaryl Phosphate Ester
SAE Grade	15W-30	30	30

The polyol ester lubricant, SDL-1, provided satisfactory service up to a top ring reversal (TRR) of 340°C. An engine test with this lubricant at a TRR of 400°C had to be terminated after 50 hours. Heavy deposits were formed on the piston lands in this test. When the experimental lubricant SDL-2 based on an aromatic ester was evaluated under these conditions, the test again had to be terminated after a short time. However, in this case, the lubricant provided satisfactory deposit control, but excessive ring liner wear caused a dramatic increase in blow by. Since these tests were being conducted with a variety of ceramic ring/liner wear couples, the conventional antiwear additive used in SDL-2 was assumed to be not functional with these new materials.

The additive package of SDL-2 was fortified with a triarylphosphate ester antiwear additive. This new lubricant designated SDL-3, was found to provide satisfactory antiwear protection with the ceramic wear couples used in this program. SDL-3 was found to function at a TRR as high as 450°C. Low levels of deposits were observed with SDL-3 under these conditions. Chemical analysis of the deposits revealed that they could be attributed to the ash containing additives in the conventional additive package. Very low levels of carbon were detected in these upper cylinder deposits. This was attributed to the intrinsic property of the aromatic base stock to decompose cleanly at the high temperatures of this engine test. This property also suggested that the aromatic esters would contribute less to the lubricant contribution of particulate emissions.

2.2 Present Program Objectives and Scope

The major program tasks conducted in the present project are:

Task I - Lubricant Specifications and Engine Requirements
Task II - High Temperature Liquid Lubricant Formulations/Bench Testing
Task III - Single Cylinder Engine High Temperature Lubricant Testing
Task IV - (Option) Multicylinder Engine Lubricant Validation

The objective of this program was to develop a lubricant which would function satisfactorily in a single cylinder test engine at a TRR of 500°C and an oil sump temperature of 250°C. A Cummins NTC 250 single cylinder engine was available for this work and plans were made at the beginning of the contract to fit the engine for high temperature tests. At a Task 1 review held early in the program, DOE/NASA requested that a more current engine, the Cummins L10, be used for testing in this program. This would allow coordination with the In-Cylinder Components Program also funded by DOE/NASA under Contract DEN3-375 which already used a single cylinder version of this engine. Mechanical problems with the design and operation of an L10 single cylinder engine at the severe operating conditions required for this program eventually forced redirection of effort to the use of a modified multi-cylinder L10 which was essentially the Optional Task IV.

The primary objective of the laboratory phase of lubricant development was the selection of base stocks and additives which would give formulations which generate the lowest level of deposits in appropriate screening tests. These lubricants were also to incorporate all other functionals required for operation under the severe conditions of this program, including, oxidation stability, wear control, corrosion prevention, elastomer compatibility, and adequate high and low temperature viscosity properties. The original intent of the contract was to create an interactive testing program alternating between laboratory and engine tests using the latter to refine the former. Difficulties in engine operation at high temperature resulted in time and funding constraints precluded the use of this approach. The optimum fluids as determined by a series of laboratory bench tests were selected for evaluation in the multi-cylinder L10 engine tests.

3.0 TASK I - LUBRICATION SPECIFICATIONS/ENGINE REQUIREMENTS

Task I involved the identification of State-Of-The-Art (SOA) diesel engines and liquid lubricants, specification of the requirements of advanced Low-Heat-Rejection (LHR) Heavy Duty Diesel (HDD) engines and lubricants, and documentation of existing commercial and governmental liquid lubricant research and testing programs which addressed advanced high temperature lubrication requirements for LHR and HDD and turbine engines. The major results of these surveys and investigations are paraphrased in the following paragraphs of this section.

Major driving forces for commercial diesel engines include engine durability (in excess of 500,000 miles before overhaul), reliability, and fuel economy. These attributes impact operating costs which influence customer purchasing decisions. Technologies used to achieve these goals by diesel engine companies include the use of electronic fuel injection for better control of injection

resulting in increased fuel efficiency. There is also a major trend to increase injection pressures in the 151 MPa (22,000 psi) range. Charge air cooling is also used to increase operating performance, increase fuel efficiency, and to decrease emissions.

Future commercial diesel engine systems must continue to emphasize market-driven technology demands which include improved durability (target 750,000 to 1 million miles), improved reliability, improved fuel economy (target 0.28 to 0.25 lbs/bhp-hr), and meeting legislated emissions requirements. One interesting outcome of the survey was that there was significant interest in the long term future use of one fluid (oil) for cooling and lubrication.

In surveys regarding high temperature lubrication, temporary and permanent viscosity loss are important performance factors for conventional engines and are a critical concern for engines operating at higher temperatures. Broad multi-grade oils are formulated with high levels of polymers to meet SAE viscosity grade requirements; consequently, these products exhibit significant temporary and permanent viscosity loss in tests which simulate engine load bearing areas. The extreme mechanical and thermal environment of the advanced diesel engines will severely restrict or preclude the use of polymeric VI improvers.

A variety of chemical tests were performed to categorize products based on the generic types of additive chemistry common to all crankcase lubricants. These tests provided information on overbased detergents (TBN, detergent metals content, and sulfated ash), nitrogen-based dispersants (nitrogen content), and ZDP anti-wear additives (phosphorus and zinc content). The overall oxidation stability of the lubricants was also defined with the Thin Film Oxygen Uptake Test (TFOUT). As expected, heavy duty diesel lubricants were found to have higher loadings of overbased detergents than gasoline engine oils. They also had higher levels of nitrogen dispersants and marginally higher levels of ZDP. All classifications of lubricants had similar TFOUT break time distributions. This survey did not provide any indication that it would be possible to operate at the program goals with commercially available mineral oil or synthetic products. The survey showed that commercial lubricants use very similar additive chemistry which has already been demonstrated to have serious deficiencies in this severe environment.

4.0 TASK II - LABORATORY TESTING AND DEVELOPMENT

Lubricant formulation research was conducted in three major lubricant categories during the course of this program. These included commercial lubricants (the SDL series), experimental lubricants (the EXP series), and finally the lubricant formulations that were evaluated in engine tests (the HTL series). The program approach was to use the SDL series to develop bench tests and screening tests to differentiate lubricant performance. Further improvement made to the formulations were evaluated in the EXP series which were later modified for engine test evaluation. These lubricants for engine test were designated as HTL lubricants. Table 4.0 summarizes these lubricant formulations. Aromatic Ester A and Aromatic Ester B used in the lubricant formulations were aromatic diesters.

Since the foundation of a high temperature lubricant is the base stock, this program was initiated with a general evaluation of base stock categories using bench tests and test conditions which were standard for high temperature lubricants at Akzo. The base stocks used in the SDL series of lubricants were incorporated as internal controls to verify trends.

The next phase of the work entailed additive selection and lubricant formulation. Akzo bench tests were employed for this formulation development. However, independent arrangements were also made with Penn State and NIST to supplement Akzo testing and lubricant formulation using their bench test procedures and additive expertise. In this way, a broader range of experience was brought to bear in the program. The Penn State and NIST efforts were phased in after Akzo formulation work was under way.

Following the identification of appropriate test conditions, additive selection and formulation work commenced with the objective of optimizing a low ash additive package that minimized deposit formation and maximized stability. The Falex panel coker was used to study deposits and the Alcor deposition test was used to measure stability and dispersancy.

Due to concerns about the poor viscosity index (VI) of the aromatic esters, the next phase of the lubricant development effort shifted back to base stock evaluations. The objective was to identify high VI base stocks to use with the aromatic esters to provide adequate lubricant film thickness at high operating temperatures. A polymeric ester was identified as a co-base stock to be used with the aromatic ester. The resulting lubricant meets 15W-40 multigrade viscosity criteria.

One of the objectives of this program was to reduce and ultimately eliminate the ash containing additives which contribute to deposits. An external acid absorbent in the form of a chemical filter was envisioned to supplement the additive package. A variety of absorbent media were evaluated in slurry tests for acid removal efficiency and additive compatibility.

Tabel 4.0

Lubricant Formulations Investigated

Designation	Base Stock	Additive Package*	SAE Grade
SDL-1	Polyol Ester A	Conventional	15W-30
SDL-2	Aromatic Ester A	Conventional	30W
SDL-3	Aromatic Ester A	Conventional + Triaryl Phosphate Ester	30W
EXP-1	Aromatic Ester A	1.5%A+0.75% ODP+1%SDL+* 0.75% Aryl ZDP+5% Aryl Phosphate	30W
EXP-2	Aromatic Ester A	1.5%A+1% ODC+2%SDC+* 0.75% Aryl ZDP+5% Aryl Phosphate	30W
EXP-3	Aromatic Ester A	1.5%A+1% ODC+3%SDC+* 0.75% Aryl ZDP+5% Aryl Phosphate	30W
EXP-4	Aromatic Ester A	1.5%A+1% ODC+3%SDC+* 0.75% Aryl ZDP+5% Aryl Phosphate	40W
HTL-1	Premium Mineral Based Lubricant		15W-40
HTL-2	Aromatic Ester B (34%) +Polymeric Ester	Low Ash + VI Improver (polymeric)	15W-40
HTL-3	Aromatic Ester B	Low Ash +Organo-Copper Inhibitor	40W
HTL-4	Aromatic Ester B (34%) +Polymeric Ester	Low Ash +VI+Organo-Copper Inh.	15W-40
HTL-5	Aromatic Ester B (34%) +Polymeric Ester	NIST additive +VI Improver	15W-40

* A= Aromatic Amine Antioxidants
OD = Overbased Detergent
SD= Succinimide Dispersant
C,L,P = Variations of Detergents and Dispersants

4.1 Laboratory Bench Tests

The primary objective of this project is to develop lubricants which minimize deposit formation in the upper cylinder area of a low heat rejection engine. Four laboratory screening tests were selected for evaluation of this property. This selection was based on previous Akzo experience in the development of synthetic lubricants for gas turbines and compressors and on literature references to high temperature lubricant development. These four laboratory tests were:

a) the Falex panel coker test
b) the Alcor deposition test
c) the Penn State microoxidation test
d) the NIST DSC two peak deposit test

The Falex panel coker test was used extensively in the development of synthetic compressor lubricants by Akzo. With proper selection of test conditions it was found to correlate well with the deposit forming characteristics of lubricants on the exit valves of reciprocating compressors. These high temperature, thin film conditions were thought to correspond well with the environment of the upper cylinder area of the diesel engine. This test also provides a great deal of flexibility in time and temperature exposure of the lubricant to hot surfaces and the degree of lubricant replenishment. In the Falex panel coker test, a rotating steel comb or splasher is immersed in a reservoir of the test fluid. When activated the splasher throws droplets of fluid on a heated, inclined steel plate which forms part of the reservoir enclosure. The frequency of splashing, speed of the splasher, and the temperature of the plate can be independently varied. The weight of deposits on the plate after a given test time relates to the deposit forming tendencies of the fluid.

The Alcor deposition test is an accepted lubricant standard for evaluating the deposit characteristics of synthetic gas turbine lubricants. As opposed to the thin film oxidation characteristics of the Falex panel coker, the Alcor deposition test would correspond more to the engine components that are continuously wetted with a lubricant film. The Alcor deposition test also provides information on bulk fluid properties such as viscosity and acid number buildup which relate to the oxidation and thermal stability of the lubricant. The Alcor test configuration provides for the circulation of heated fluid at a constant flow rate through an annulus formed by a ten inch steel tube and an electrically heated steel rod. Prior to exposure to the heated rod, air is injected into the fluid. The flexibility of the test extends to test duration, tube temperature, and the air flow injected into the oil. Fluid physical properties are determined after the test. The degree of change is indicative of the fluid stability. Dispersancy is determined by deposit weight and a visual rating of the rod.

The Penn State microoxidation test developed by Professor Elmer Klaus has been shown to correlate well with a variety of engine test data bases for predicting oil life and engine cleanliness. In addition to lubricant deposit forming characteristics, this test can also be used for determining lubricant stability and lubricant evaporation loss. The test is very flexible. Test time, test temperature, and the nature of the catalytic surface in contact with the lubricant can all be varied.

Specifically a small amount of fluid (20-40 mg), is exposed to high temperature and air as a thin film on a metal specimen shaped like a shallow, flat-bottomed dish. Changes in fluid molecular weight and the volatility of the sample relate to fluid stability. Residual deposit weight on the metal specimen provide a measure of fluid cleanliness.

Dr. Hsu and Dr. Perez at NIST have developed a deposit screening test based on differential scanning calorimetry (DSC). Roughly 0.8 mg of the lubricant is placed in a titanium pan covered by a lid with three small holes. The pan is then placed in the DSC under 100 psi oxygen. The test temperature is raised steadily at 2 degrees per minute up to 340°C, then the heating rate is increased to 10 degrees per minute. The first part of heating generates a peak corresponding to heat of oxidation. The second part of heating generates a peak corresponding to heat of combustion of the deposits formed in the first period. The ratio of the two peaks has been used to correlate with deposit forming tendency of lubricants. Several different test conditions have evolved to provide better correlation for lubricants of various quality levels.

4.2 Initial Tests

4.2.1 Base Stock Deposit

A series of deposit tests were conducted to establish conditions which differentiate deposit forming characteristics on various lubricant base stocks. The Falex panel coker and Alcor deposition tests were used in this study. For the Falex panel coker, test time, panel temperature, and splasher speed were varied to maximize the differences among the base stocks tested. Among the various conditions evaluated, a three hour test performed with a panel temperature of 315°C and a splasher rate of 700 RPM at a sump air flow of 850 cc/min provided the best separation of these base stocks.

As shown in Table 4.1, aromatic esters and aliphatic diesters show the lowest level of deposits compared to other base stocks including polyol esters, poly-alpha-olefins, or super refined mineral oil of comparable viscosity. Within each class of base stock, the level of deposit seems to be viscosity (or possible volatility) dependent to some degree. The aromatic ester A used in SDL-2 and SDL-3 exhibited the best overall deposit performance in this initial series of base stock studies.

Table 4.1

Summary of Base Stock Deposition Studies

Falex Panel Coker

	100°C Viscosity (Cst)	Average Deposits [3 tests] (mg)
Aromatic Ester A (SDL-2,3 base stock)	9.7	22
Aromatic Ester B	17	31
Aliphatic Diester	5.6	25
Polyol Ester A (SDL-1 base stock)	8.5	39
Polyol Ester B	4.7	43
Polyol ester C	16	102
Poly-alpha-olefin	10	46
Super Refined Mineral Oil	12	55

A series of base stock evaluations was also conducted with the Alcor deposition test (Table 4.2). Alcor deposition tests were conducted at a flow rate of 300 ml/min at an upper tube temperature of 370 C for 24 hours with an air volume of 1000 ml/min. As in the case of the panel coker study there was a clear distinction between a polyol ester and the aromatic ester. The poly-alpha-olefin and the aliphatic diester exhibited very similar deposition characteristics which were intermediate between the aromatic ester and the polyol ester.

It should be noted that these base stocks were evaluated without antioxidants and therefore experience significant thermal and oxidative breakdown in the test as indicated by the high viscosity increase. The deposit rating not only reflects the inherent deposit forming tendency of the undergraded base stock, but also the formation and deposition of base stock degradation products. This test also pointed to the use of the aromatic ester as the optimum base stock for minimizing high temperature deposits.

Table 4.2

Base Stock Study
Alcor Deposition Test

Base Stock	% Viscosity Change	TAN Change	Tube Deposits (mg)	Tube* Rating
Polyol Ester A (SDL-1 Basestock)	624	8.2	112	120
Aromatic Ester A (SDL-2,3 Basestock)	662	1.4	11	63
Aliphatic Diester	386	29.7	76	109
Poly-alpha-olefin (10 cSt)	440	5.6	58	92

*0 = Clean

4.2.2 Lubricant Deposits

Newly formulated lubricants were evaluated under the same conditions that were used to differentiate base stocks with the Falex panel coker test. The results from tests performed on two of these formulations are shown in Table 4.3. In these tests the same conventional additive package was used to treat polyol esters and aromatic esters. The inclusion of the additive package essentially masked the differences observed between these two base stocks and increased the total deposits for the panel coker test. A similar lack of differentiation in deposit characteristics for these two formulations was observed with the Alcor deposition test.

Table 4.3

Effects of Base Stock and Additive Package on Lubricant Deposition Characteristics

Falex Panel Coker and Alcor

	Falex mg/deposits	Alcor % Viscosity Change	Alcor TAN Change	Alcor Deposits (mg)	Tube Rating
Polyol Ester A (SDL-1 Basestock)	39	624	8.2	112	120
Polyol EsterA + Additive Package A	51	68.7	5.1	0	18
Aromatic Ester A (SDL-2,3 Basestock)	22	662	1.4	11	63
Aromatic Ester A + Additive Package A	60	27.3	3.9	6.8	21

4.2.3 Modified Bench Tests

At this point the decision was made to use SDL-1 and SDL-3 as reference oils to identify test conditions for the Falex panel coker and/or the Alcor deposition test which would allow distinguishing the performance of these two oils in accord with their performance in high temperature LHR engine tests. A variety of changes were made in the operating conditions of the Alcor deposition test including changes in tube temperature, the amount and relative humidity of air introduced into the fluid, and the volume of fluid in the test reservoir. None of the changes in the Alcor deposition test conditions were found to be suitable for the objectives of this program.

Changes made in Falex panel test conditions included test panel temperature, rate of splasher rotation, and changing to an intermittent splasher mode. This last change proved to be the most effective for discriminating the deposit characteristics of SDL-1 and SDL-3. Using a 10 second on/60 second off cycle in the panel coker, SDL-1 exhibited dramatically higher deposits than SDL-3. The information available from this test procedure was further amplified by performing one hour deposit tests for each lubricant at a series of temperatures, thus generating a temperature/deposit profile for each lubricant. The results for this test using SDL-1 and SDL-3 are shown in Figure 4.1. This test was found to be very reproducible as shown by the results for three replicate runs on SDL-3 in Figure 4.2.

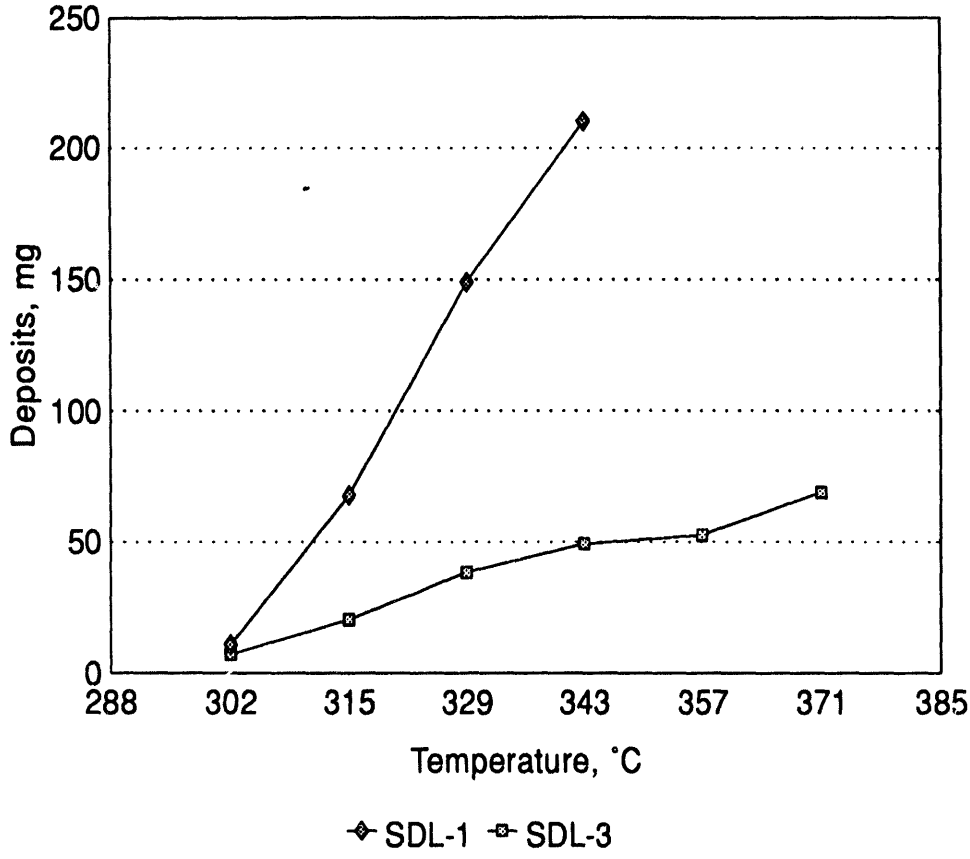


Figure 4.1

Lubricant Deposit Temperature Profile for SDL-1 vs. SDL-3

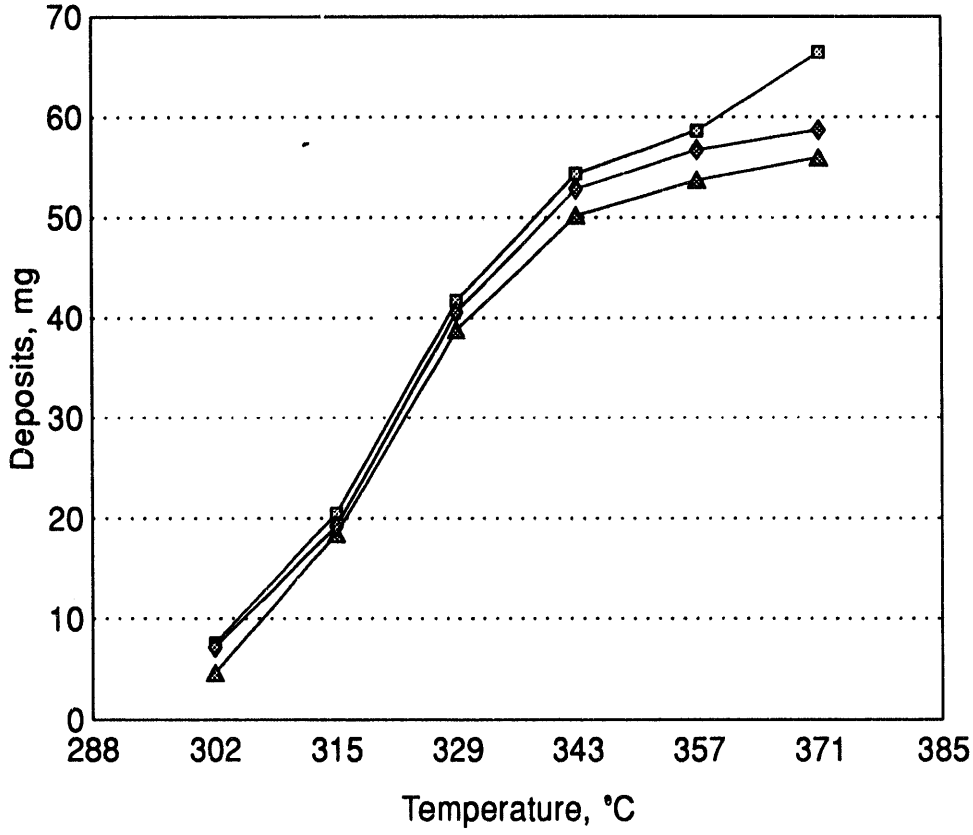


Figure 4.2

Deposit Temperature Profile Reproducibility of SDL-3

Base stocks used in SDL-1 (Polyol Ester) and SDL-3 (Aromatic Ester) were evaluated under the conditions described in the previous section. The results of these tests shown in Figure 4.3 indicate that these new conditions further amplify the differences observed earlier between these two types of base stocks. Furthermore, these tests illustrate that a conventional diesel detergent inhibitor additive package significantly contributes to the deposits formed in a lubricant formulated with the aromatic ester. This is evident from the amount of deposits formed from the aromatic ester lubricant SDL-3 at 371°C versus the amount of deposits formed at the same temperature of the aromatic ester by itself. Based on these results, improvements of the deposit performance of SDL-3 should be feasible by optimizing the additive package for this property.

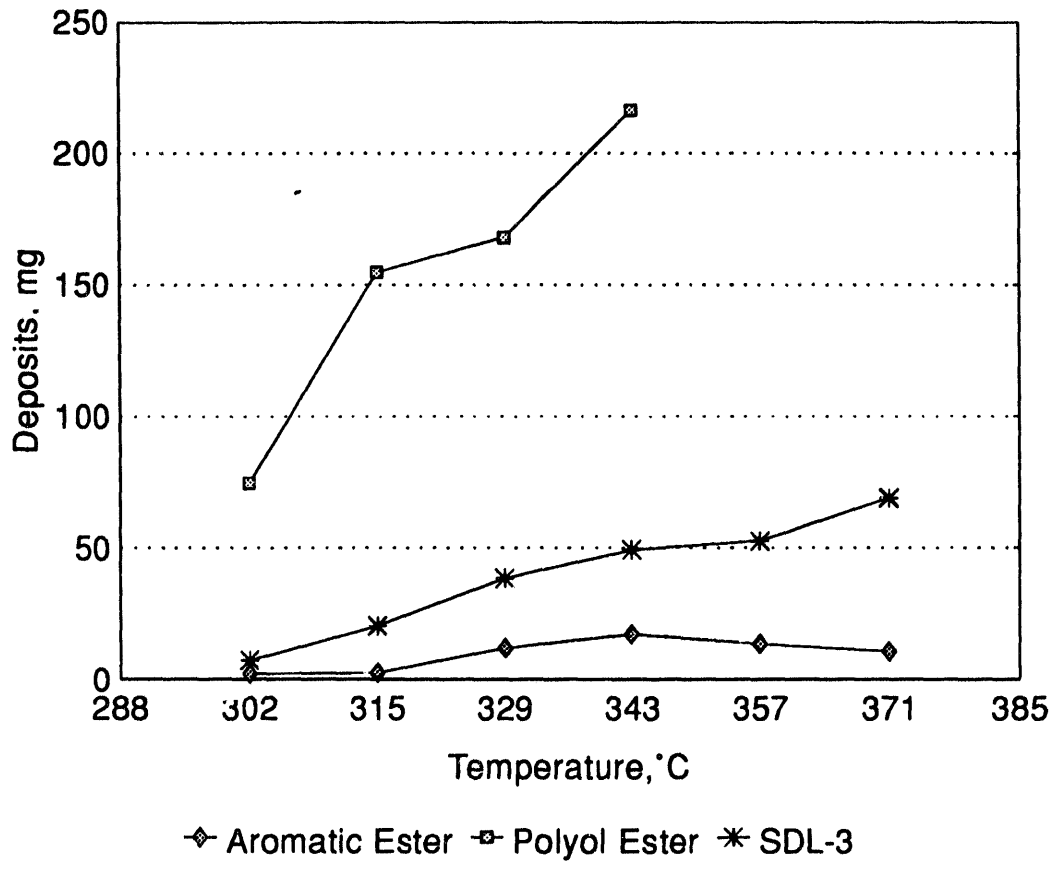


Figure 4.3

Base Stock Deposit Temperature Profile
Aromatic Ester vs. Polyol Ester vs SDL-3

4.3 Lubricant Formulation

4.3.1 Lubricant Deposit Composition

The compositions of the deposits formed in the Falex panel coker test of the reference lubricants SDL-1 and SDL-3 were analyzed by Scanning Electron Microscopy and Energy Dispersive X-ray Analysis (SEM EDAX). The level of elemental constituents attributable to the additive package was much higher in the deposits from the aromatic ester lubricant SDL-3 then from the polyol ester lubricant SDL-1. Conversely, the deposits from SDL-1 had a much higher level of carbon. This is consistent with the observations made in the high temperature LHR engine tests described earlier. Ring land deposits from SDL-1 were found to be primarily carbonaceous, while the ring land deposits and cylinder liner deposits from SDL-3 were primarily composed of metal oxides from the degradation of the additive package. The EDAX traces shown in Figure 4.4 illustrate

these differences. This points to the need to reduce the ash level of lubricants designed for high temperature diesel operation.

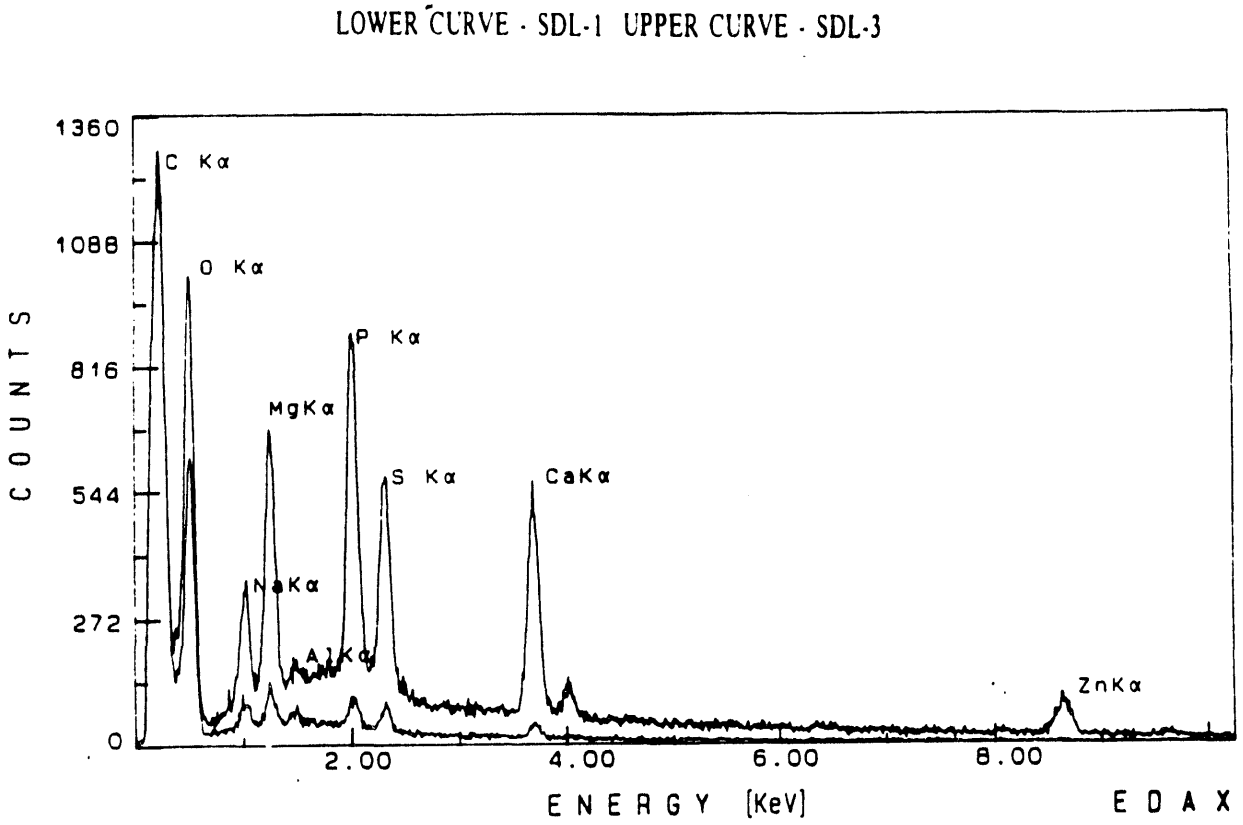


Figure 4.4

Falex Panel Coker Deposit Composition

4.3.2 Additive Package Development

Formulation studies were initiated using the Falex panel coker test for thin film deposit characterization and the Alcor deposition test for bulk fluid property changes such as viscosity and total acid number. The Alcor deposition test was also used as a measure of the lubricants dispersancy characteristics as revealed by the overall tube deposit rating. The use of the Alcor test to rate fluid stability is illustrated in the relative viscosity increase of SDL-1, SDL-3 and HTL-1, the premium SAE-15W-40 petroleum based lubricant used in the engine tests

(Figure 4.5). The relative deposit characteristics of these fluids as measured by the Alcor test are also shown in Figure 4.5.

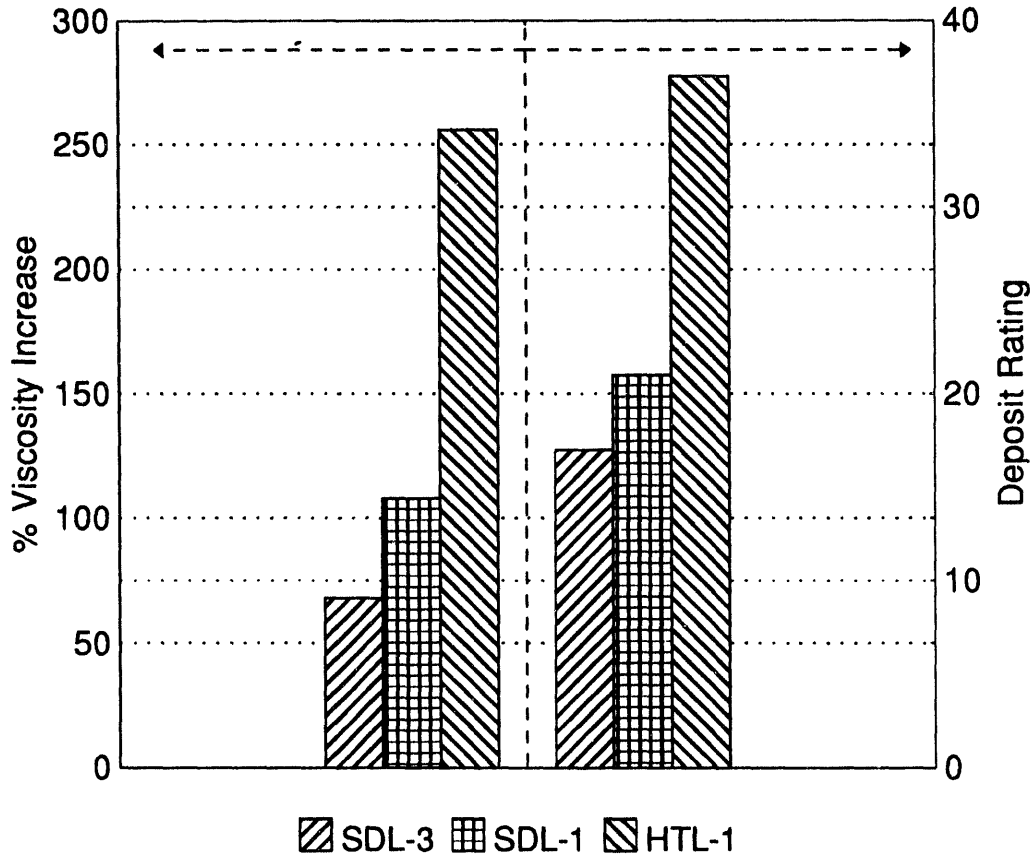


Figure 4.5

Lubricant Stability Characteristics
Alcor Deposition Test

Formulation work on the aromatic ester was initiated with the selection of the antioxidant system. Since the target is a high temperature lubricant, antioxidants were selected from those typically used in high temperature gas turbine lubricants, i.e., aromatic amines. A combination of these aromatic amine antioxidants was tested at two different concentrations in the aromatic diester base stock. A triarylphosphate ester was also incorporated into the system at a 5% level. This additive was used in all formulation studies based on the original finding from the LHR engine tests that a phosphate ester is needed with the aromatic diesters to prevent wear problems at the ring liner interface. The Alcor deposition test results at antioxidant treat levels of 3% and 1.5% illustrate the importance of selecting the proper additive concentration (Table 4.4). The

antioxidant can be deleterious at excessive levels. The higher antioxidant level offers no advantage in viscosity control and contributes to deposits.

Table 4.4

Lubricant Formulation Studies

Alcor Deposition Test

	% Viscosity Change	TAN Change	Tube Deposits(mg)	Tube Rating
Aromatic Ester A + 1.5% Aromatic Amine Antioxidants	9.6	0.9	2.8	35
Aromatic Ester A + 3% Aromatic Amine Antioxidants	17.5	1.1	10.4	50

Following the identification of the proper antioxidant level, formulation work continued on the development of a low ash additive package for the aromatic ester base stock used in SDL-3. Other conventional diesel lubricant additives were added sequentially to the blend of aromatic ester/antioxidant /triarylphosphate. After the addition of each additive, the blend was tested in the Alcor deposition test to determine the impact of the additive on fluid performance. The results of this series of deposition tests are shown in Table 4.5. This table graphically illustrates the deleterious effect of the overbased detergent on the stability of the fluid as shown by the significant viscosity change. However, the incorporation of a small amount of overbase was considered to be necessary to prevent corrosion and control the build up of acid byproducts in the lubricant. A low level of zinc diaryldithiophosphate was also found to be necessary as a coantioxidant.

Table 4.5

Aromatic Ester Formulation Studies

Alcor Deposition Test

Additives In Aromatic Ester A Plus 5% Phosphate Ester	% Viscosity Change	TAN Change	Tube Deposits(mg)	Tube Rating
1.5% Aromatic Amine Antioxidants	9.6	0.9	2.8	35
0.75% Overbased Detergent P	366	31.2	56.6	81
1.5% Aromatic Amine Antioxidants + 0.75% Overbased Detergent P	53.3	10.4	12.1	59
1.5% Aromatic Amine Antioxidants + 0.75% Overbased Detergent P + 1.0% Succinimide Dispersant L	32.9	6.1	10.5	38
1.5% Aromatic Amine Antioxidants + 0.75% Overbased Detergent P + 1.0% Succinimide Dispersant L + 0.75% Aryl ZDP	15.3	2.3	10.7	36

Commercial overbased detergents and dispersants were also evaluated (Table 4.6). The combination of overbased detergent (C) and dispersant (C) provided the best overall performance in the Alcor deposition test. The thin film deposits characteristics of experimental fluids 1, 2 and 3 are illustrated in the Falex panel coker results shown in Figure 4.6. While the overall performance of the fluids with 2% dispersant was marginally better in both deposit tests, it was decided to proceed with the higher level of dispersant in experimental fluid 3 to ensure adequate sludge and soot dispersion capacity.

Table 4.6

Aromatic Ester Formulation Studies and Effect of Viscosity

Alcor Deposition Test

Formulation	% Viscosity Change	TAN Change	Tube Deposits(mg)	Tube Rating
Experimental - 1 (Exp-1) 1.5% Aromatic Amine Antioxidants + 0.75% Overbased Detergent P + 1.0% Succinimide Dispersant L + 0.75% Aryl ZDP 5.0% Aryl Phosphate	15.3	2.3	10.7	36
Experimental - 2 (Exp-2) 1.5% Aromatic Amine Antioxidants + 1.0% Overbased Detergent C + 2.0% Succinimide Dispersant C + 0.75% Aryl ZDP 5.0% Aryl Phosphate	9.7	1.0	nil	19
Experimental - 3 (Exp-3) 1.5% Aromatic Amine Antioxidants + 1.0% Overbased Detergent C + 3.0% Succinimide Dispersant C + 0.75% Aryl ZDP 5% Aryl Phosphate	13.1	1.2	5.4	28
Experimental - 4 (Exp-4) (40 wt version of Experimental 3)	19.0	3.0	7.7	19

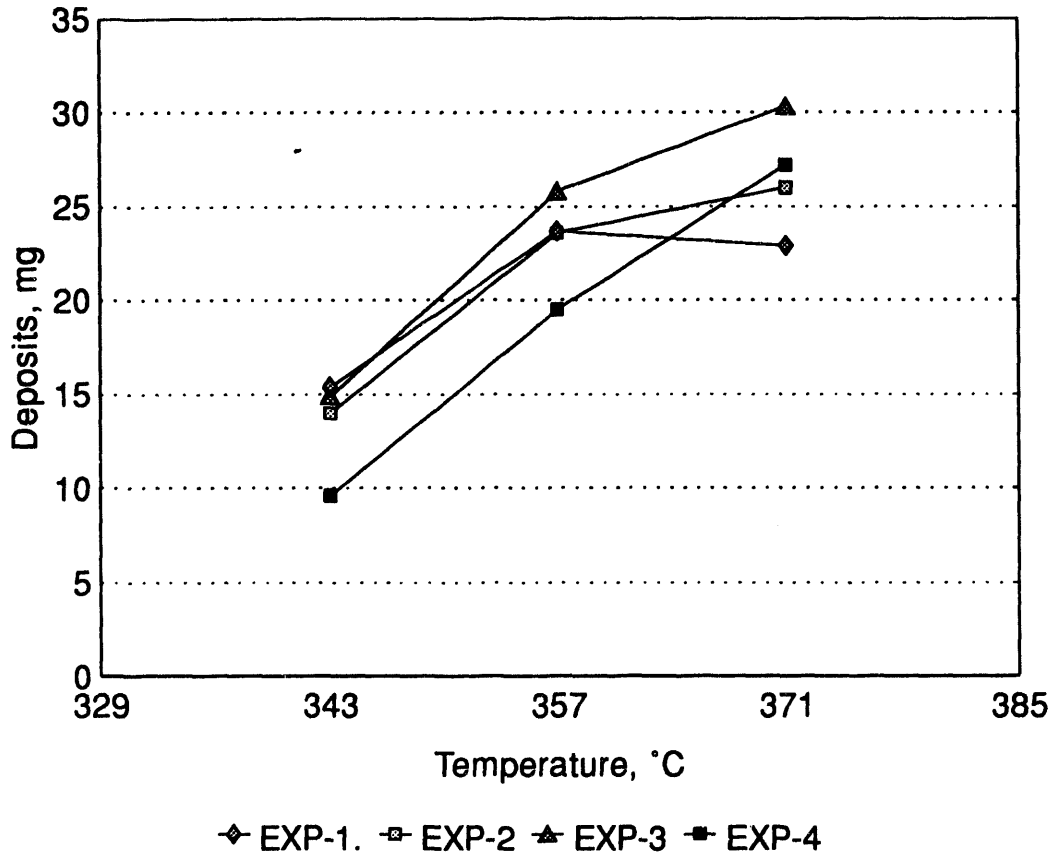


Figure 4.6

Lubricant Deposit Temperature Profile Effect of Additives and Viscosity

At this stage in the formulation work, studies were extended into higher viscosity analogs of the aromatic ester used in formulating SDL-3. SDL-3 is an SAE-30 weight lubricant. It has a very poor viscosity index of 85. This not only indicates poor low temperature properties, but the rapid change in viscosity with temperature also implies rapid thinning at high temperature. Since one of the objectives of this program is running with a very hot sump, there was concern that a 30 weight oil would not provide sufficient film thickness to protect the main and connecting rod bearings of the test engine. A 40 weight version of Experimental 3, designated Experimental 4, was formulated with aromatic ester B and subjected to the Alcor deposition test and the Falex panel coker test. No significant differences in performance were observed in changing to the more viscous base stock (Table 4.6 and Figure 4.6).

4.3.3 High Viscosity Index Base Stocks

While the aromatic ester base stocks may provide the best deposit performance at high temperatures for the LHR engine, the poor viscometric properties could be an insurmountable barrier to the wide spread use of aromatic ester based commercial lubricants. Moreover, their ability to lubricate at high temperatures because of the rapid fall off of viscosity was yet to be resolved by engine tests with high temperature sump conditions. For this reason, studies commenced on combinations of aromatic esters with other base stocks with better viscometric properties. This was done with the recognition that such mixtures would probably compromise the exceptional deposit control characteristics of the aromatic esters exhibited in laboratory bench tests.

The original base stock screening work performed using the panel coker test revealed that aliphatic diesters approached aromatic esters in thin film deposit control in the test (Table 4.1, Section 4.2.1). However, most representatives of this class of ester are relatively low in viscosity. The commercial availability of a new long chain dicarboxylic acid provided the opportunity to test a more viscous diester, specifically the ditridecyl ester of dodecane dicarboxylic acid. A Falex panel coker test of this aliphatic diester was somewhat encouraging (Figure 4.7). The ester formed a low level of deposits and the deposits tended to flake off the panel after cooling. This behavior was not observed with the aromatic ester.

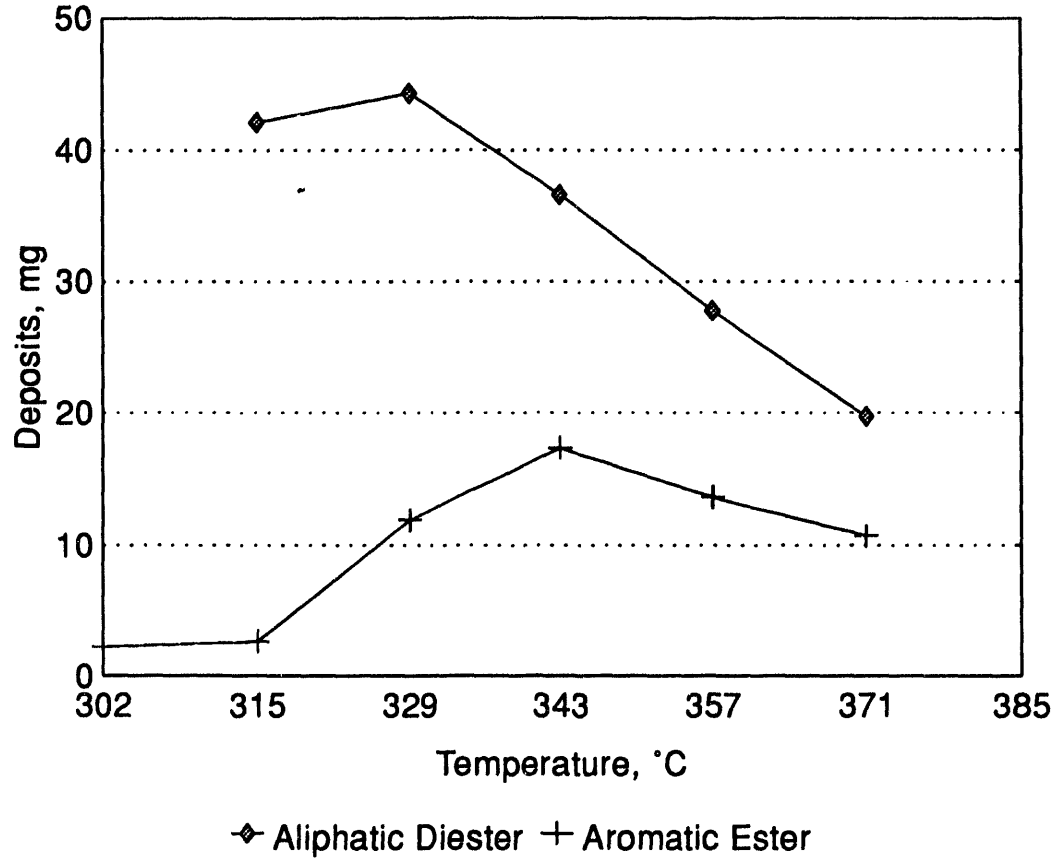


Figure 4.7

Lubricant Deposit Temperature Profile of Aliphatic and Aromatic Ester

While the panel coker results for this aliphatic diester were encouraging, stability problems were encountered in the Alcor deposition test (Table 4.7). A simple aliphatic diester formulation which incorporated antioxidants and a phosphate ester showed extensive deterioration in the Alcor test compared to an aromatic ester formulation tested under the same conditions.

Table 4.7

Ester Stability Comparison

Alcor Deposition Test

Formulation: 1.5% Antioxidants

5.0% Aryl Phosphate

Base Stock	% Viscosity Change	TAN Change	Tube Deposits(mg)	Tube Rating
Aromatic Ester	9.6	0.9	2.8	35
Aliphatic Diester	116	14.8	83	99
	Deposit Performance of Improved Viscosity Index Lubricant, HTL-2			
HTL-2	-7	1.9	2.5	22
EXP-4	19	3.0	7.7	19

Following the evaluation of a number of other low deposit base stocks, a polymeric ester which has a 100°C viscosity of 6.0 cSt was found to provide the best balance of low deposits and stability when used in conjunction with the aromatic ester (Table 4.7). The same low ash additive package described in connection with EXP-4 was used in this formulation, designated HTL-2. The composition of HTL-2 is shown in Table 4.8. A low level of a polymeric VI improver was also incorporated in this formulation to achieve a SAE 40 weight viscosity.

In addition to thickening the oil, this polymeric VI improver was also incorporated for its potentially beneficial effect in controlling oil consumption. This hypothesis was based on the observation that lubricants with polymeric viscosity index improvers exhibit less oil consumption in the Cummins NTC 400 engine test than straight grade SAE 40 lubricants. This is observed in spite of the fact that the multi-grade viscosity improved lubricants are formulated with more volatile base stocks than the straight grade SAE 40 weight lubricant. Therefore, the polymer appears to be controlling a significant mechanism of oil consumption in this case.

Table 4.8

Composition of HTL-2

COMPONENT	WEIGHT %
Aromatic Ester B	34.0
Polymeric Ester	53.73
Viscosity Index Improver	1.0
Triaryl Phosphate Ester	5.0
Aromatic amine antioxidants	1.5
Succinimide Dispersant C	3.0
Overbased Detergent C	1.0
Aryl ZDP	0.75
Copper Deactivator	0.02

4.4 Wear Studies of Aromatic Ester Based Lubricants

Another objective of this program was to evaluate the surface interaction of antiwear additives with various substrates. This objective was included to develop a better understanding of the wear phenomena observed in the original engine tests conducted with SDL-2 and SDL-3. Reiterating test results described in the introduction of this report, SDL-2 exhibited high wear in an NTC 250 engine test to the extent that the test had to be terminated after 50 hours. After the addition of a triarylphosphate ester antiwear additive to the formulation, the new formulation designated SDL-3 functioned satisfactorily for extended periods of time at high temperature with various ceramic wear couples.

Wear tests were conducted with a Four-Ball wear test to gain a better understanding of the wear phenomena observed in engine tests.A significant difference was observed between SDL-2 and SDL-3 under the test conditions described in Table 4.9. Wear tests were conducted at 600 rpm, 54°C, 40 Kg for a one hour time period.

Table 4.9

Wear Properties of SDL-2 and SDL-3

Four-Ball Wear Tests

Test Conditions: 600 RPM, 54°C, 40 Kg, 1 hour

Formulation	Wear Scar (mm)
SDL-3	0.29
SDL-2	0.42

SDL-3 exhibited negligible wear compared to SDL-2. Based on this test, further studies were conducted on the interaction of classical antiwear additives such as aryl and alkyl ZDP in the aromatic ester base stock.

In these base stock studies wear scar results shown in Figures 4.8 and 4.9, the response of aromatic ester, polyol ester, and mineral oil were compared following the addition of various levels of aryl ZDP and alkyl ZDP.

These studies showed that classical aryl and alkyl ZDP antiwear additives are ineffective in aromatic esters. The relative effectiveness of aryl ZDP in mineral oil is also apparent in Figure 4.8. While aryl ZDP is known to be less efficient than alkyl ZDP (even at a relatively low treat level of 0.75%), a significant reduction in wear is observed with mineral oil. Polyol esters are also found to be much more amenable to treatment with ZDP than the aromatic esters.

Therefore, some of the wear problems that were observed in tests conducted with SDL-2 may be related to an incompatibility with the classical ZDP antiwear additives used in the formulation as opposed to the high temperature conditions or unusual wear couples. Finally, Table 4.10 shows wear test results for a low ash blend based on aromatic ester compared to the aromatic ester results. These wear tests again illustrate the ineffectiveness of aryl ZDP in aromatic ester formulations.

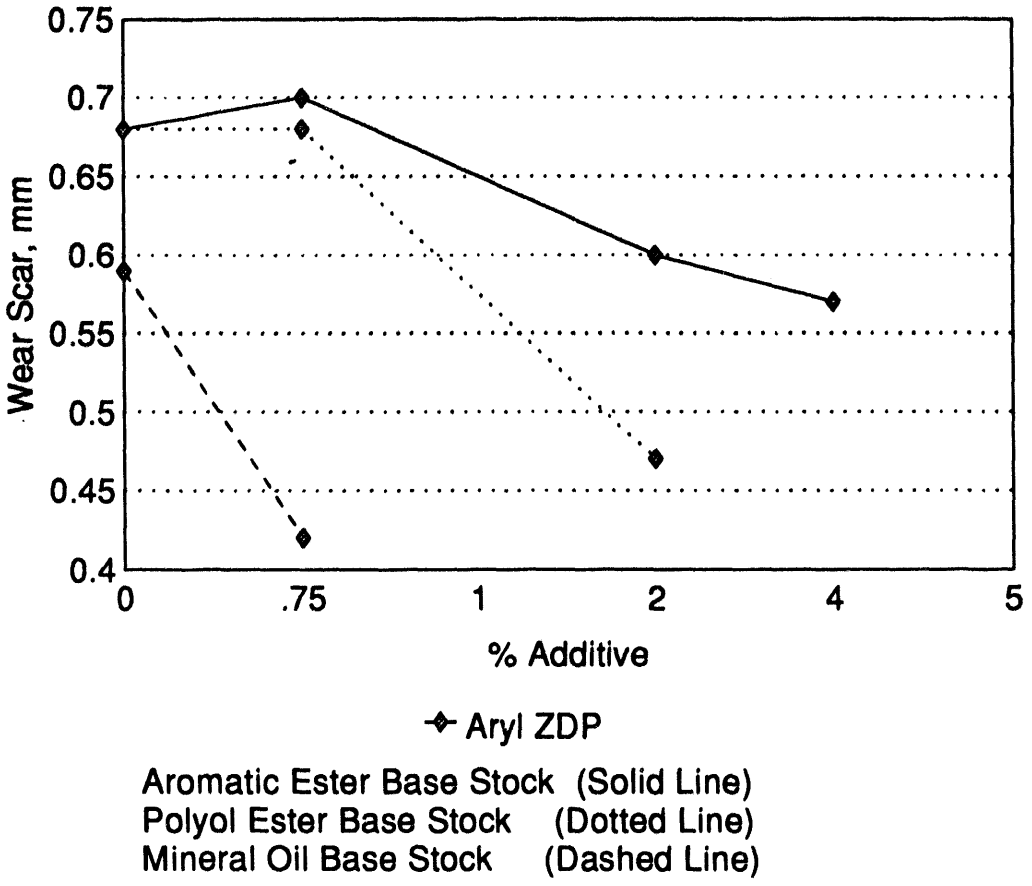


Figure 4.8

Effect of Aryl ZDP on Wear Scar

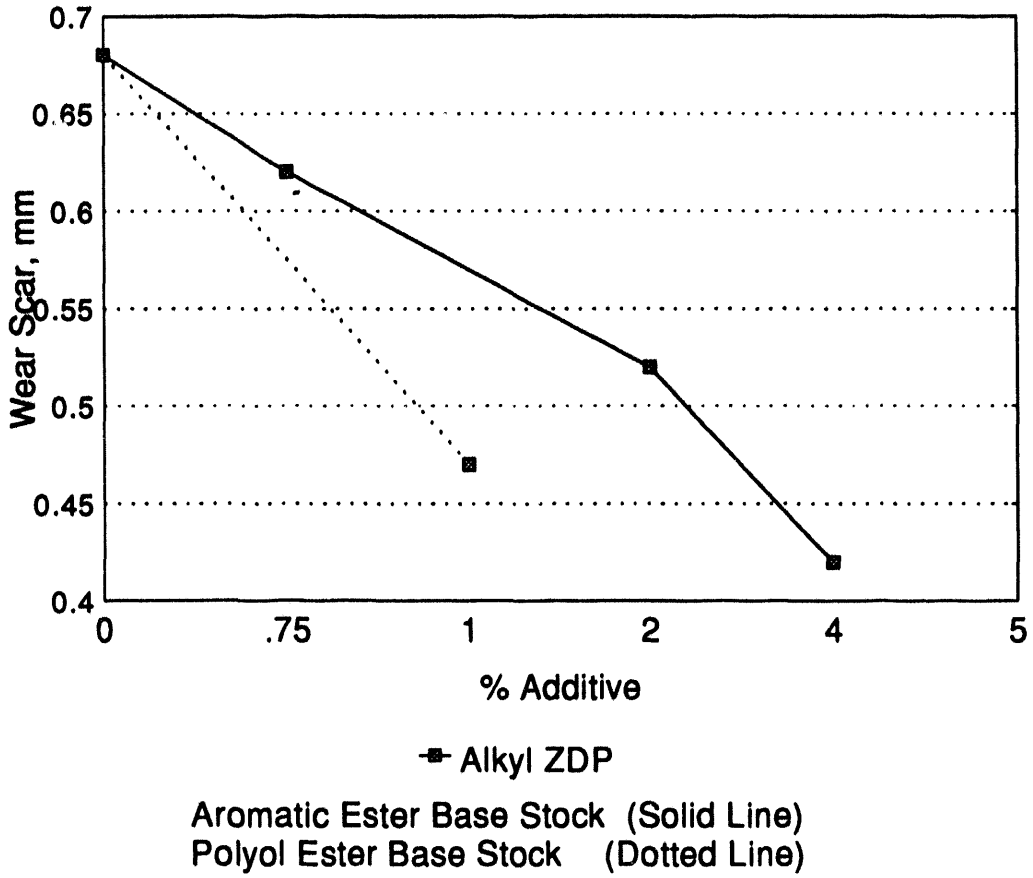


Figure 4.9

Effect of Alkyl ZDP on Wear Scar

Table 4.10

ZDP Activity in Aromatic Ester Formulations

Four-Ball Wear Tests

Test Conditions: 600 RPM, 54°C, 40 Kg, 1 hour

Low Ash Base Blend:
Aromatic Ester
1.5% Antioxidants
1.0% Overbased Detergent C
2.0% Succinimide Dispersant C

	Wear Scar (mm)	Percent Additive Aryl ZDP	Treatment Phosphate Ester
Aromatic Ester	0.68	-	-
Aromatic Ester	0.42	-	5.0
Base Blend	0.65	-	-
Base Blend	0.59	0.75	-
Base Blend	0.34	-	5.0
Base Blend	0.34	0.75	5.0

As in the base stock studies, a phosphate ester antiwear additive was much more efficient in reducing the wear of this aromatic ester based formulation.

4.5 Evaluation of Acid Absorbing Media for Chemically Reactive Filter

The primary source of deposits in the original engine tests performed on SDL-3 were metallic based additives such as the overbased detergent and the ZDP present in the formulation. One of the primary thrusts of this program was to reduce and eventually eliminate these ash generating additives. Even though current emissions regulations are significantly decreasing fuel sulfur levels, acid by-products from fuel and lubricant degradation must be controlled in the lubricant to prevent corrosion of engine components. Formulations discussed to this point in this report incorporate low levels of overbased detergent to control acids generated from low sulfur fuel. To achieve the ultimate targets of ashless formulation, one possible option for replacing the overbase is to incorporate an external filter which would continuously treat acidic materials from the lubricant. This section of the report describes studies conducted to identify and evaluate appropriate media for such an acid absorbing filter.

For screening the filter medium, aromatic ester B was saturated with sulfur dioxide gas at 25°C and was treated with 10 wt% of each candidate absorbent for 48 hours. The acid removal

capabilities of the absorbent media are contained in Table 4.11. Two primary types of media were evaluated in this initial study, zeolites and clays. The zeolites were found to be most active for removing the sulfur dioxide. Zeolites A, B, and C were selected for further study.

Table 4.11

Evaluation of Sulfur Dioxide Adsorbing/Characteristics of Various Filter Media (@ 25°C)

Filter Medium	Acid Removing Capacity mg SO_2/g solid
Zeolite A	75
Zeolite B	68
Zeolite C	56
Zeolite D	34
Zeolite E	33
Zeolite F	27
Zeolite G	26
Clay A	22
Clay B	14
Clay C	14
Clay D	9

Since these absorbents would be used in a chemically aggressive system at high temperatures, it was critical that the filter materials do not contaminate the lubricant with metallic reaction products. Experiments were conducted with Zeolites A, B and C at a 10 wt. % level in aromatic ester B which incorporated sufficient sulfur dioxide to achieve a TAN of 11 mg KOH/g. Each slurry was maintained at 175°C for 72 hours in seal tubes. At the end of the test period, the fluids were filtered and analyzed for total metal content. Zeolite C showed negligible introduction of metals into the fluid. Zeolite A incorporated 50 ppm total metals and Zeolite B incorporated 300 ppm of total metals. Based on this work further evaluations were limited to Zeolites A and C.

Filter media evaluation studies continued with new and used samples of EXP-4, the low ash lubricant based on aromatic ester B. Filter media slurry tests were conducted on EXP-4 containing 0.19 wt. % sulfur dioxide (Table 4.12). This level of sulfur dioxide incorporation raised the acid number of the fluid by 1.7 mg KOH/g. Treatment with 10 wt % of Zeolites A and C at 25°C for 24 hours significantly reduced the total acid number of the fluid in each case. Zeolite A continued to show the best overall performance.

Table 4.12

Effect of Filter Media on Sulfur Dioxide Treated Lubricants

	EXP-4	
	TAN	TBN
Initial	0.5	3.7
Addition of 0.19 wt % SO_2	2.2	0.8
Treated with 10 wt % Zeolite A	1.4	0.7
Treated with 10 wt % Zeolite C	1.7	0.8

Filter media were also evaluated in a sample of EXP-4 which was run in a Cummins L-10 engine under normal operating conditions. This was done to determine the efficiency of acid removal for the media under more typical conditions where the used fluid incorporates fuel by-products and soot. The total acid number of the used lubricant in this evaluation was 4.0 mg KOH/g. In addition to Zeolite A a new media designated Zeolite H was used to reduce the acid number of this test fluid. The evaluations were conducted using a 10 wt% slurry of each zeolite at 85°C for 48 hours. Zeolite A reduced the acid number of the used oil by 1.4 mg KOH/g and Zeolite H reduced the acid number by 1.8 mg KOH/g. This demonstrates that the decomposition products of the lubricant and the soot in the used oil do not dramatically deactivate the surface of the zeolite.

The interaction of Zeolite H with various additive components of EXP-4 was evaluated by selectively deleting individual components from the total formulation and measuring the acid number and base number following treatment with 10 wt% of Zeolite H at 75°C for 72 hours (Table 4.13). The results of these experiments demonstrate that Zeolite H does not remove the acidic additives such as the arylamine antioxidants or the aryl ZDP antiwear additive. Some depletion of the overbase is observed. These studies demonstrate that Zeolite H is a viable candidate for the active media of an external acid absorbing filter.

Table 4.13

Effect of Zeolite H on Additive Levels of EXP-4

	TAN mg KOH/g Initial	after 72 hrs	TBN mg KOH/g Initial	after 72 hrs
EXP-4	0.37	0.37	3.7	3.3
EXP-4:				
without anti-wear additive	0.13	0.13	3.5	3.2
without overbase	0.34	0.34	1.0	0.8
without dispersant	0.37	0.21	2.3	2.2
without anti-oxidant	0.31	0.31	3.5	3.3

4.6 Subcontracted Lubricant Evaluation and Development

4.6.1 Penn State Lubricant Evaluation and Development

As discussed in the introduction, a research contract was funded at Penn State with Dr. Elmer Klaus to assist in the development of high temperature lubricants for this program. Dr. Klaus developed the Penn State microoxidation test. Over the years this test has been correlated with gasoline and diesel engine lubricant performance both as a measure of lubricant stability and deposit forming characteristics.

The program with Penn State was initiated with an evaluation of the deposit forming characteristics of SDL-1 and SDL-3. The microoxidation test easily distinguished these two reference oils (Table 4.14). This test also confirmed the desirability of reducing the ash level of the lubricant as exemplified by the lower deposits observed for EXP-4 compared to SDL-3.

Table 4.14

Determination of Lubricant Deposit Formation With the Microoxidation Test

Time (min)	Weight % Deposits		
@ 250°C	SDL-1	SDL-3	EXP-4
40	17.0	1.3	1.4
60	21.5	1.6	0.6
80	32.0	-	1.9
120	-	-	5.3
@ 275°C			
30	21.0	7.3	1.7
60	28.9	10.9	6.4

In addition to describing the deposit forming tendencies of lubricants, the microoxidation test can also be used to evaluate the evaporation characteristics of lubricants as related to base stock volatility and the generation of volatile components via fluid degradation. The lubricant retention characteristics of SDL-1, SDL-3 and EXP-4 are shown in Table 4.15. The higher temperature determination points to a potential volatility problem with the aromatic ester used in SDL-3 and EXP-4. SDL-1 utilizes a less volatile base stock component which is reflected in the remaining liquid at 275°C. This volatility could significantly amplify the anticipated oil consumption problems at the high sump and ring liner temperatures projected for this program.

Table 4.15

Determination of Lubricant Retention and Deposit Formation With the Microoxidation Test

	60 min @ 250°C		60 min @ 275°C	
	Remaining Liquid,%	Deposits, Wt %	Remaining Liquid, %	Deposits, Wt %
SDL-1	21.5	70.4	28.9	42.5
SDL-3	1.6	66.1	10.9	12.4
EXP-4	0.6	70.7	6.4	11.5

New additive technology developed by Dr. Klaus to control lubricant oxidation and deposit tendencies was applied to the high temperature lubricants developed in this program. This additive technology entails the addition of soluble copper salts to the lubricant at a level sufficient to achieve a concentration of 2000 ppm of copper. This approach has been demonstrated in the microoxidation test to significantly reduce the rate of fluid oxidation and deposit formation. This also results in a reduced rate of fluid loss due to evaporation by controlling the formation of volatile lubricant by-products. The effect of this additive treatment on EXP-4 is illustrated in Table 4.16. The addition of the copper additive dramatically increases the amount of retained fluid under the conditions of the microoxidation test. It also significantly reduces the already low deposit forming characteristics of this lubricant. The copper containing modified EXP-4 is designated HTL-3.

Table 4.16

Effect of Copper Additive on Lubricant Retention and Deposit Formation

Microoxidation Test at 250°C

	EXP-4		HTL-3 (EXP-4+2000 ppm Cu)	
Time (Hrs)	Remaining Liquid,%	Deposits, Wt %	Remaining Liquid, %	Deposits, Wt %
3	9.5	12.8	67.9	2.4
4	4.7	13.2	-	-
6	-	-	42.2	9.4

A similar favorable result on lubricant retention and deposit formation was observed with the addition of the copper additive to HTL-2 (Table 4.17). HTL-2 is defined in Table 4.0.

The copper modified lubricant designated HTL-4 showed significantly improved oxidation stability as indicated by the increased fluid retention and reduced deposit forming tendencies.

Table 4.17

Effect of Copper Additive on Lubricant Retention and Deposit Formation

Microoxidation Test at 250°C

TIME (Hrs)	HTL-2		HTL-4 (HTL-2 + 2000 ppm Cu)	
	Remaining Liquid,%	Deposits, Wt %	Remaining Liquid, %	Deposits, Wt %
0.5	68.6	1.3	83.0	0.4
1.0	40.3	5.3	64.7	5.6
2.0	19.9	11.0	48.1	7.0
3.0	1.2	23.7	36.5	12.4
4.0	0	18.7	19.4	13.1
6.0	0	18.9	6.7	15.1

4.6.2 NIST Lubricant Evaluation and Development

A research contract was also funded at NIST by Akzo to supplement the development of high temperature diesel lubricants under the direction of Dr. Steve Hsu and Dr. Joseph Perez. The program with NIST was constructed to take advantage of that additive expertise and integrate it into the current lubricant development project.

As in the Akzo internal evaluation, NIST first evaluated the deposit characteristics and oxidation stability of SDL-1, SDL-3 and a premium petroleum diesel lubricant, HTL-1. The deposit characteristics were determined using the NIST "two-peak" DSC deposit test. Oxidation stability was determined by pressure DSC.

The deposit characteristics of petroleum based and synthetic reference lubricants are separated well by the two peak deposit test (Table 4.18). The distinction between SDL-1 and SDL-3 is less dramatic than with the panel coker or the microoxidation test. The relative oxidation stability of the reference lubricants are shown in this Table at the following test conditions: 220°C, 550 PSI Oxygen, Steel Pan. The lower oxidation stability for SDL-3 versus SDL-1 is related to the less stable aromatic ester versus the polyol ester in SDL-1.

Table 4.18

Lubricant Deposit and Oxidation Stability

Lubricant	NIST Two Peak Deposit, %	Isothermal PDSC Induction Time (min.)
HTL-1	26.1	1.1
SDL-1	6.6	9.9
SDL-3	5.0	6.5
EXP-3		12
EXP-4		22

Following these evaluations of the program reference oils, NIST initiated formulation studies starting with the low ash additive package incorporated in to EXP-4. This work resulted in a combination of Akzo and NIST additives to yield a lubricant EXP-5 with significantly enhanced oxidation stability as measured by the pressure DSC method also shown in Table 4.18. The Akzo additive package was supplemented with an overbased salicylate detergent and additional high temperature antioxidants.

The additive package used in EXP-5 was incorporated into the base stock system developed for HTL-2, the multi-grade alternative to the aromatic ester used in EXP-5. This was done to provide an alternative additive system to the high copper formulation used in HTL-4. This new multi-grade lubricant incorporating the combination of NIST and Akzo additive technology is designated HTL-5. A comparison of the performance of HTL-2 and HTL-5 is shown in Table 4.19.

Table 4.19

Performance Comparison of HTL-2 and HTL-5

	HTL-2	HTL-5
Panel Coker, mg @		
315°C	6.7	1.1
343°C	41	9.5
371°C	53	91
Alcor Deposition Test		
Viscosity Change, %	6.9	11.5
TAN Charge, mg KOH/g	1.9	2.7
Tube Deposits, mg	2.5	15.9
Tube Rating	22	36

5.0 TASK III - HIGH TEMPERATURE LUBRICANT ENGINE TESTING

Laboratory bench testing can provide valuable guidance to screen out inappropriate lubricant base stocks and additives; however, no combination of tests can simulate the complex chemical and physical interactions which occur in a fired engine. For example, thin film deposit tests measure lubricant oxidation and metal catalyzed polymerization, but these tests do not approach the severe environment of the upper cylinder area where a thin lubricant film is repeatedly exposed to combustion gases at very high temperatures and pressures. Simple laboratory tests do not simultaneously subject fluids to the high flash temperature of heavily loaded contact areas while circulating lubricant through a wide variety of temperature zones. Only engine tests can ultimately tell the lubricant formulator if test protocols and new base stock or additive approaches are viable for operating systems. This is particularly true for the extreme conditions attempted in this program.

Five lubricants were selected for engine testing (HTL) series). HTL-1 was a premium mineral base lubricant used as a baseline. HTL-2 through 5 were lubricants formulated for the tests based on the results of the laboratory work described in Section 4. The tests were run on a modern multi-cylinder engine modified to attain higher temperature.

5.1 Engine Test

Test Hardware - The L10 engine used for lubricant evaluation is described in Table 5.1.

Table 5.1

L10 Multi-Cylinder Engine Specifications

Model	1988 LTA10-350*
No. of Cylinders	6 (In-line)
Displacement	10 liters (611 in^3)
Bore	125 mm (4.92 in.)
Stroke	136 mm (5.35 in.)
Compression Ratio	17.1
Rated Power	257 kW (350 hp) @ 2100 rpm
Torque Peak	1288 N-m (950 lb-ft) @ 1300 rpm

*Cummins P-T Fuel Injection System

Description of the engine test components is presented in Table 5.2.

Table 5.2

Engine Test Components

- 1991 Production Articulated Steel Crown Pistons.
- Production 1st, 2nd and Oil Control Rings.
- High Temperature Capacity Production Bearings.
- Production Cast Iron Liners.

As shown in Table 5.2, production aluminum pistons were replaced with production articulated steel crown pistons. The articulated pistons were used to accommodate target elevated cylinder kit temperatures. Details of engine modifications made to achieve elevated cylinder kit temperatures are presented in the Appendix 9.1 at the end of the paper.

5.2 Initial Lubricant Tests

The lubricants were evaluated in an engine test run for 100 hours at the initial operating conditions presented in Table 5.3. Representative cylinder kit temperatures achieved at the initial test conditions are shown in Table 5. 4.

Table 5.3

Initial Engine Operating Conditions

Engine Speed	1200 ± 2 rpm
Engine Torque	1630 N-m
Fuel Rate	43.5 kg/hr ± 0.4 kg/hr
Oil Sump Temperature	149 ± 2°C
Intake Manifold Air Temperature	66 ± 4°C
Cylinder Head Coolant Temperature	93 ± 2°C

Table 5. 4

Cylinder Kit Temperatures at Initial Test Conditions

Location	Temperature (°C)
Piston:	
Center of Top Land§	400
Back of Top Ring	335
Groove	
Back of Second Ring	280
Groove	
Center Undercrown	305
Top Compression Ring	330
Liner:	
Top¶	310
Middle	300
Bottom	230

§Piston temperatures measured with Templugs.
¶Liner temperatures measured with J-type thermocouples.

During each test, oil was continuously replaced as it was consumed with a weigh-tank/feeder system. A sample was taken from the oil sump at least once every 12 hours. Each oil sample was evaluated on-site for viscosity increase. The samples were sent off-site for TAN, TBN and metals analyses.

To determine lubricant contribution to particulate, exhaust particulate measurements were made at three points during each test: at 20 hours, at 50 hours and at 100 hours. These steady-state measurements were made at 1300 rpm (peak torque speed) full load and 1800 rpm (near rated speed) full load.

The lubricants tested are defined in Table 4.0. These oils included HTL-1 a multi-grade mineral oil (15W-40), HTL-2 a multi-grade synthetic lubricant (15W-40) and HTL-3 a straight-grade synthetic lubricant (SG 40). The initial test lubricant properties are presented in Table 5.5.

Table 5.5

Initial Test Lubricant Properties

	HTL-1	HTL-2	HTL-3
Viscosity @ 40°C (cSt)	117	107	152
Viscosity @ 100°C (cSt)	14.5	13.5	12.9
Viscosity Index	124	123	84
TAN (mg KOH/g)	2.3	0.44	0.73
TAN (mg KOH/g)	6.7	5.9	4.2

5.3 Results From Initial Lubricant Tests

The engine test to evaluate lubricant HTL-1 did not run for the planned 100 hours. The engine was shut down after 40 engine hours at which time the engine oil pressure had decreased from 241 kPa to 138 kPa. Initially, the low oil pressure was thought to be due to a loss in oil viscosity. However, post-test Brookfield, kinematic and high temperature/high shear analyses indicated that the oil viscosity had increased rather than decreased. Upon engine teardown and inspection, it was concluded that the loss in oil pressure was due to wear debris which caused sticking of the oil pressure regulator piston and the high pressure bypass valve. As a result of this finding, the bypass valve was blocked shut. For subsequent tests, the engine oil pressure was controlled by an external, manual valve.

The test of lubricant HTL-2 ran for 100 hours. During engine teardown and inspection, the entire inside of the engine was observed to be covered with a heavy coating of tenacious black sludge. The coating could not be removed by washing the engine parts in mineral spirits. It was necessary to put the parts through a hot dip process for satisfactory cleaning.

The test of lubricant HTL-3 ran for only 50 engine hours. At 50 hours, the engine had been shutdown for a weekend break. During engine restart and warm-up the turbocharger failed. The turbocharger shaft was found to be broken. The turbocharger was replaced and the engine restarted on warm-up. During the second warm-up the engine developed a low power situation. Investigation turned up another failed turbocharger. After failing a third turbocharger as well as the #3 connecting rod bearing, the test was terminated.

Post-test inspection of the first failed turbocharger turned up a plugged thrust bearing oil supply drilling. The drilling was found to be plugged with wear debris. The combination of wear debris and an extremely hot turbocharger environment (turbine casing and exhaust manifold glowed red in a lighted test cell) served to accelerate the failure of the oil starved thrust bearing.

The average oil consumption for each oil is shown in Figure 5.1. Lubricant HTL-1 (mineral, 15W-40) had the worst oil consumption while lubricant HTL-2 (synthetic, 15W-40) had the best oil consumption. These levels of oil consumption are more than an order of magnitude greater than oil consumption observed in similar engines under typical operating conditions (~0.2 kg/hr).

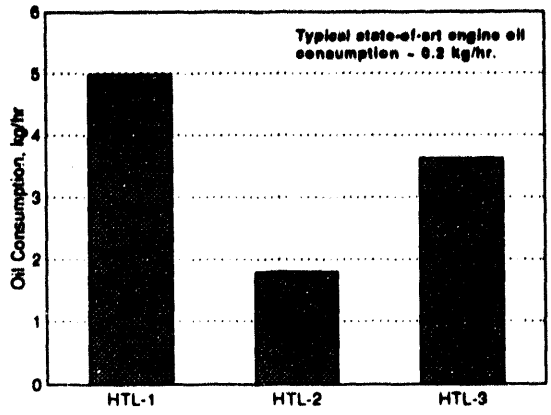


Figure 5.1

Average Oil Consumption
(Initial Engine Operating Conditions)

Figures 5.2, 5.3, and 5.4 show the viscosity and soot increases measured for each of the test lubricants. Note that for each lubricant the rate of soot increase was virtually identical (1% soot/10 test hours). The high soot (8% soot at 100 hours for lubricant HTL-2) concentration made it difficult to differentiate whether the viscosity increases were due to oil degradation or to the presence of the soot. Methods for separating the soot from the oil were investigated. No method was found which could satisfactorily remove the soot without affecting the oxidation products of the oil. The final conclusion was that future lubricant tests would have to be run with the engine combustion process adjusted to reduce the amount of soot introduced into the oil.

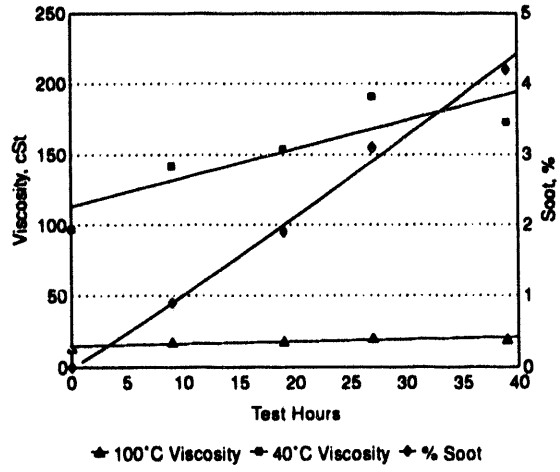


Figure 5.2

HTL-1
Viscosity and Soot Increase

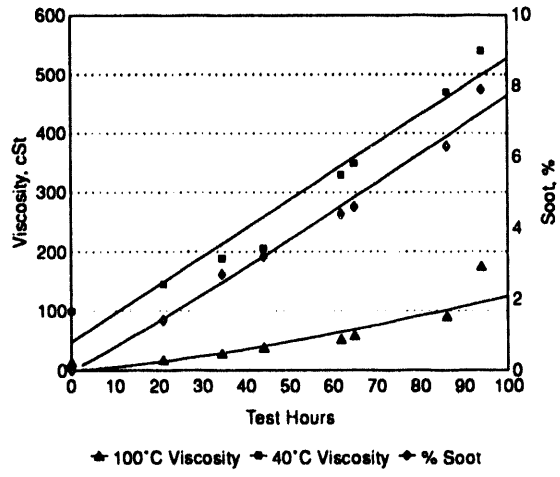


Figure 5.3

HTL-2
Viscosity and Soot Increase

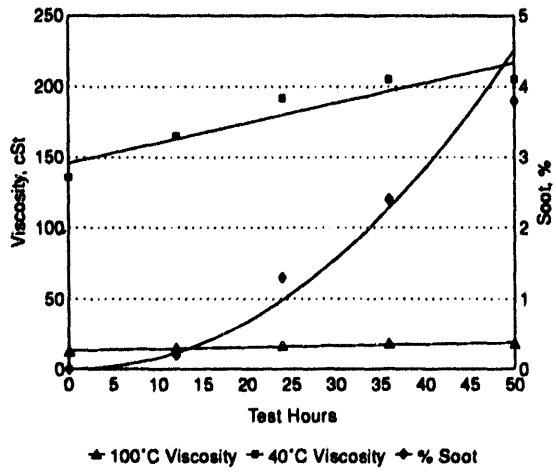


Figure 5.4

HTL-3
Viscosity and Soot Increase

Total base number (TBN) is shown in Figure 5.5. Lubricants HTL-2 and HTL-3 show a decrease in TBN. While a depletion of the overbase is expected, these lubricants exhibited a very rapid decrease at the beginning of the test. However, lubricant HTL-1 did not show a consistent decrease in TBN. This is most likely due to the relatively high oil consumption rate of HTL-1. The high consumption rate and corresponding high replacement rate of HTL-1 resulted in relatively fresh lubricant being present in the engine throughout the test. This is substantiated by the measured TBN values remaining very near the fresh lubricant value.

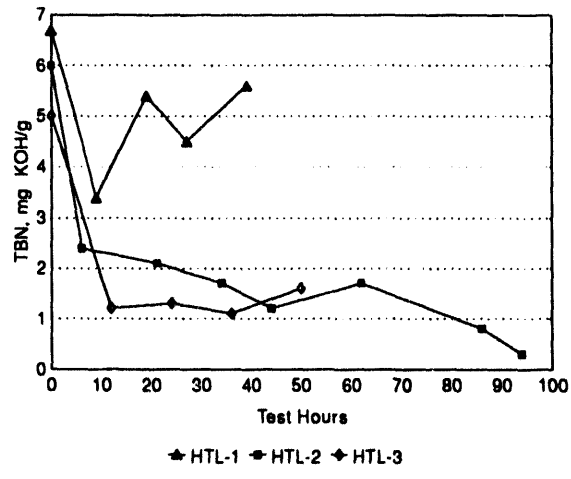


Figure 5.5

TBN Decrease
(Initial Engine Operating Conditions)

Ring wear is shown in Figure 5.6. Lubricant HTL-2 shows the best overall performance in terms of ring wear. The straight-grade synthetic, HTL-3, shows the worst performance.

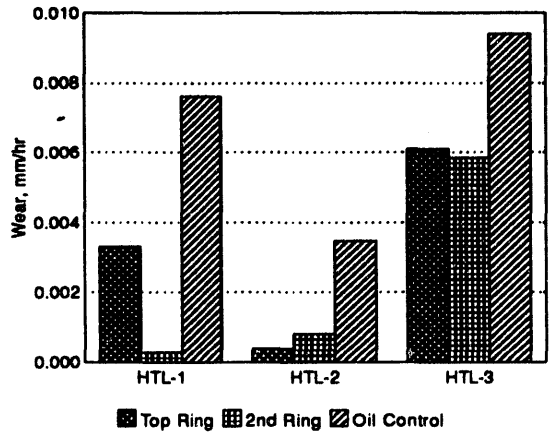


Figure 5.6

Ring Wear
(Initial Engine Operating Conditions)

Figures 5.7 and 5.8 show the iron concentration and lead concentration increase for each of the oils. Note that each graph shows a marked increase at 35 test hours for lubricant HTL-3. As discussed previously, the turbocharger failed at 50 test hours with this lubricant. However, Figures 5.7 and 5.8 indicate that the engine was failing bearings (lead increase) at least 15 hours earlier. This tends to explain the high levels of wear debris in the oil, the plugged oil drilling and the eventual failed turbocharger thrust bearings.

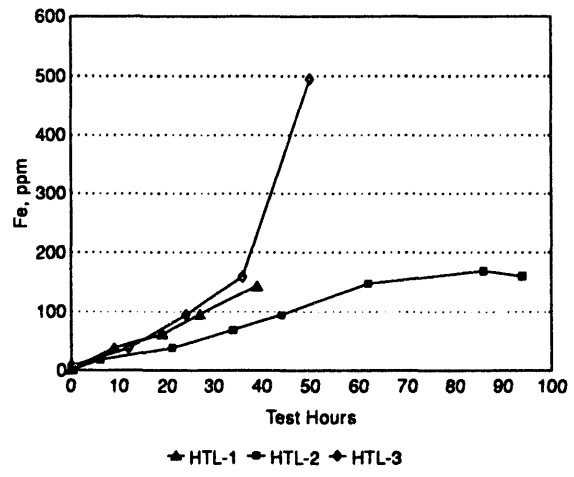


Figure 5.7

Iron Concentration Increase
(Initial Engine Operating Conditions)

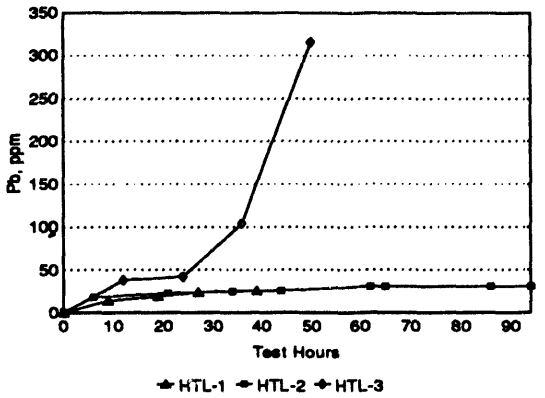


Figure 5. 8

Lead Concentration Increase
(Initial Engine Operating Conditions)

Piston ring groove deposit ratings are presented in Figure 5.9. The data show no clear performance advantage of one lubricant versus another. However, one must keep in mind that the evaluation of HTL-2 ran approximately twice as long as HTL-1 and HTL-3. With this understanding, HTL-2 appears to have performed better than either HTL-1 or HTL-3 with HTL-1 and HTL-3 performing similarly.

The data shows that for each lubricant the first groove deposits were less than the second groove deposits. Typically, first groove deposits are greater than second groove deposits and second groove deposits are greater than third groove deposits. The behavior of the data shown in Figure 5.9 can be attributed to the elevated piston temperatures. At 335°C (see Table 4), the first groove deposits have most probably been burned away. At a cooler 280°C (see Table 4), the second groove deposits have remained relatively stable.

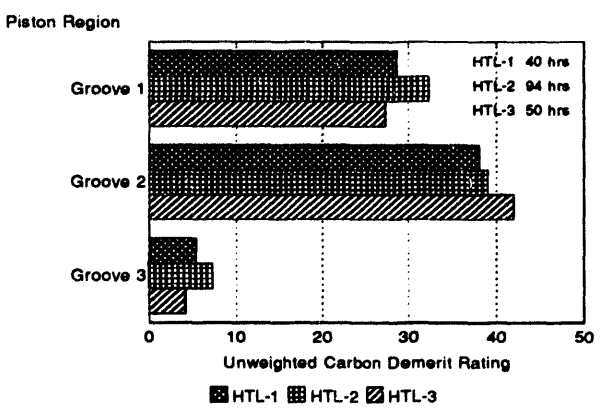


Figure 5.9

Piston Ring Groove Deposit Ratings
(Initial Engine Operation Conditions)

Piston land deposit ratings are presented in Figure 5.10. HTL-1 and HTL-3 performed better than HTL-2. However, as stated previously, the evaluation test for HTL-2 ran twice as long as both HTL-1 and HTL-3.

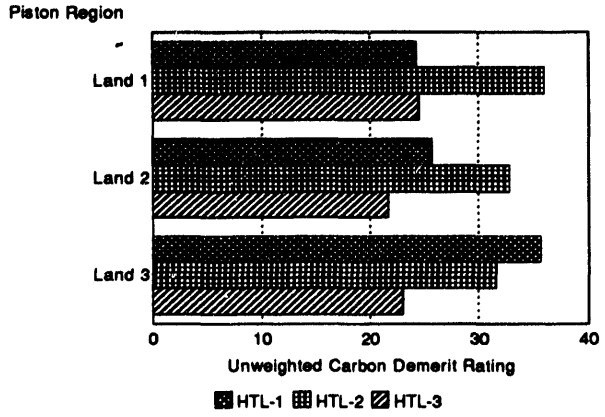


Figure 5.10

Piston Land Deposit Ratings
(Initial Engine Operating Conditions)

Figure 5.11 shows the steady-state particulate emissions measured for each of the lubricants. Lubricant HTL-3 is shown to have the same emissions characteristics as lubricant HTL-2 at 1300 rpm. However, lubricant HTL-3 is only marginally better than lubricant HTL-2 at 1800 rpm. Both synthetic lubricants performed better than the mineral oil (HTL-1) at 1300 and 1800 rpm.

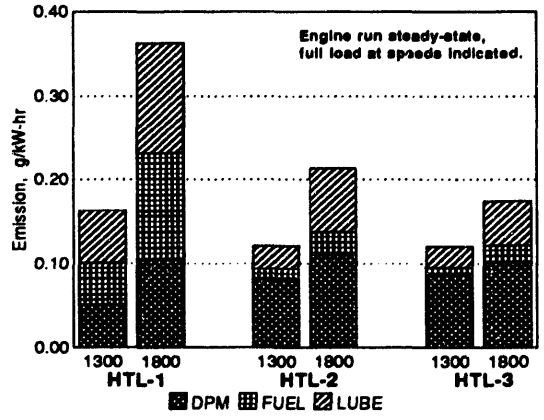


Figure 5.11

Steady State
Particulate Emissions
(Initial Engine Operating Conditions)

Based on the initial engine evaluation, it was determined that:

1. HTL-2 (15W-40) exhibited the best overall performance in terms of oil consumption, ring and bearing wear, piston deposits and soot dispersion.
2. HTL-3 (SG 40) provided inadequate bearing film at test conditions. However, HTL-3 had the lowest particulate emissions of the oils tested.

5.4 Final Lubricant Tests

The rate of soot increase in the engine oil was 0.8% soot/10 test hours. The high soot concentration in the oils made it difficult to differentiate whether or not the measured viscosity increases for HTL-1, HTL-2 and HTL-3 were due to oil degradation.

The final engine operating conditions achieved with the engine hardware modifications are shown in comparison to the initial engine operating conditions in Table 5.6. The engine torque for the final engine operating conditions was greater than the initial engine operating conditions. The torque increase was due to the increased fueling rate done to maintain elevated cylinder kit temperatures. As a result of the torque increase, it was necessary to reduce the oil sump temperature so that the connecting rod bearings would survive under the increased engine load. The piston nozzle cooling flow rate was reduced from 6.4 liters per minute (lpm) to 1.9 lpm as another means of maintaining elevated cylinder kit temperatures. Target test duration was maintained at 100 engine hours.

Table 5.6

Final Engine Operating Conditions

	Initial	Final
Engine Speed, rpm	1200	1200
Engine Torque, N-m	1630	1695
Sump Temperature, °C	150	120
Intake Manifold Temp., °C	66	66
Head Coolant Temp., °C	93	93
Piston Nozzle Flow, lpm	6.4	1.9

Representative cylinder kit temperatures achieved at the final test conditions are shown in Table 5.7 in comparison with the kit temperatures achieved at the initial test conditions. Note that despite the increased fueling rate (limited by an upper exhaust temperature of 730°C) the cylinder kit temperatures achieved at the final test conditions were lower than the cylinder kit temperatures achieved at the initial test conditions. The reduction in piston cooling nozzle flow did result in an increase in piston undercrown temperature, however. The final result was that in order to reduce soot formation it was necessary to allow a reduction of cylinder kit temperatures.

Table 5.7

Cylinder Kit Temperatures at Final Test Conditions

Position	Initial* (°C)	Final** (°C)	Δ (°C)
Top Land	400	360	-40
1st Groove	335	285	-50
2nd Groove	280	250	-30
Undercrown	305	330	+25
Top Ring	330	265	-65

* Data from Cylinder #6.
** Average of Cylinder #3 and #4 data.

The lubricants tested under the final test conditions were HTL-4 and HTL-5. Both of these lubricants were multi-grade 15W-40 synthetics blended from the base stock used in lubricant HTL-2. In HTL-4, the base stock was blended with a low ash additive package and a supplemental anti-oxidant (2000 ppm Cu). For HTL-5, the base stock was blended with an alternative low ash additive package. The final test lubricant properties are shown in Table 5.8 and are defined in Table 4.0.

Table 5.8

Final Test Lubricant Properties

	HTL-4	HTL-5
Viscosity @ 40°C (cSt)	99.8	110.9
Viscosity @ 100°C (cSt)	12.6	13.4
Viscosity Index	122	120
TAN (mg KOH/g)	0.93	0.46
TBN (mg HOK/g)	5.9	8.8

The engine evaluation tests for lubricants HTL-4 and HTL-5 completed the target 100 hours. As shown in Figure 5.12, the rate of soot increase in the oils was significantly reduced by the engine hardware modifications.

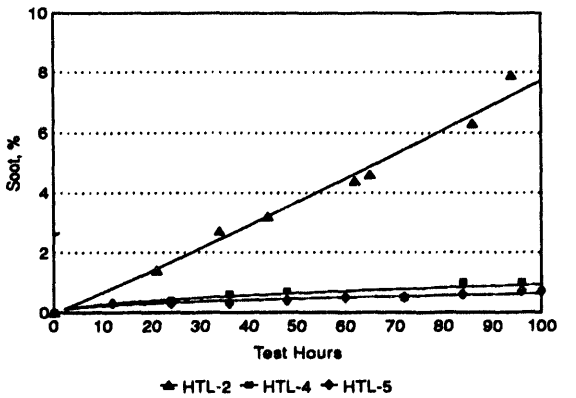


Figure 5.12

Soot Increase
(Final Engine Operating Conditions)

The average oil consumption for lubricants HTL-4 and HTL-5 is shown in comparison to lubricant HTL-2 in Figure 5.13. The average oil consumption for HTL-4 and HTL-5 is essentially the same. The fact that the average oil consumption for HTL-4 and HTL-5 is lower than for HTL-2 is most probably due to the lesser severity (lower cylinder kit temperatures) of the final test conditions.

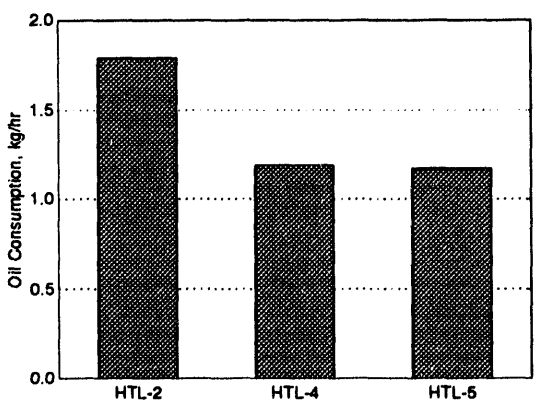


Figure 5.13

Average Oil Consumption
(Final Engine Operating Conditions)

The increases in 40°C viscosity and 100°C viscosity for lubricants HTL-4 and HTL-5 are shown in Figures 5.14 and 5.15, respectively. Viscosity increases observed for the test lubricants are significantly lower than observed for HTL-2. The reduced severity of these test conditions and

the lower level of soot in the lubricants were major factors contributing to lower viscosity increase. However, added stability from the alternate additive packages may also be a factor in improving the performance of HTL-4 and HTL-5. While the performance of the two lubricants was similar, HTL-4 had a stability advantage over HTL-5.

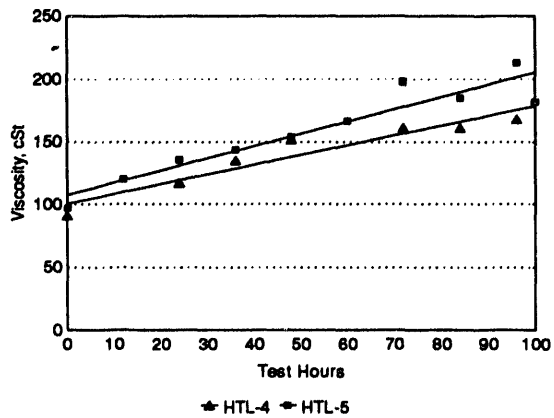


Figure 5.14

40°C Viscosity Increase
(Final Engine Operating Conditions)

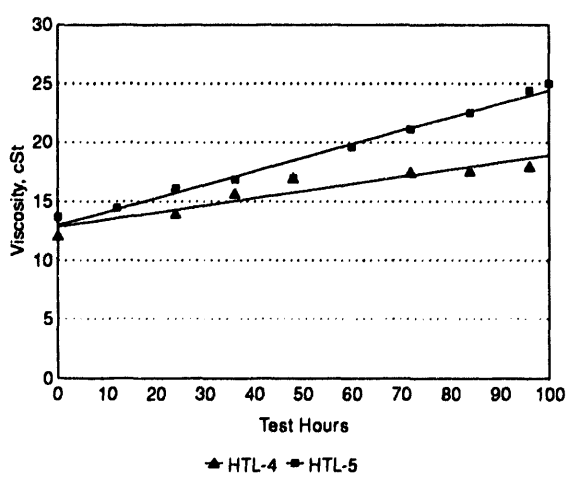


Figure 5.15

100°C Viscosity Increase
(Final Engine Operating Conditions)

The TBN decrease for lubricant HTL-4 is presented in Figure 5.16. While the formulations are not identical, these lubricants incorporate the same overbased detergent at the same level. The rapid depletion of the overbase with HTL-2 in spite of the higher oil consumption rate is probably

related to the thermal breakdown of the additive. The less severe final operating conditions used for HTL-4 allow the additive to survive and function as intended.

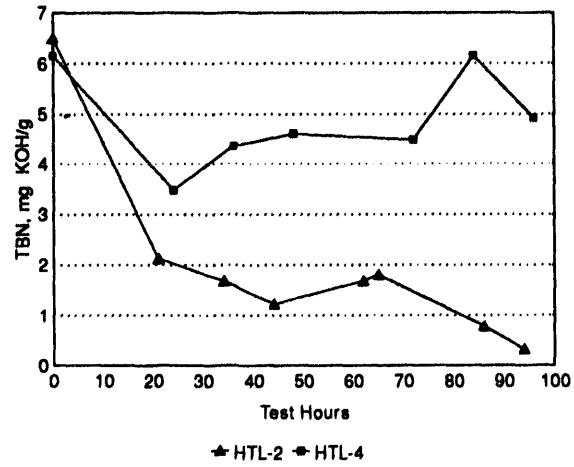


Figure 5.16

TBN Decrease
(Final Engine Operating Conditions)

Ring wear for lubricants HTL-4 and HTL-5 are shown in comparison to HTL-2 in Figure 5.17. Both HTL-4 and HTL-5 performed very well. However, HTL-4 did show poorer ring wear performance with regard to the oil control ring than HTL-5.

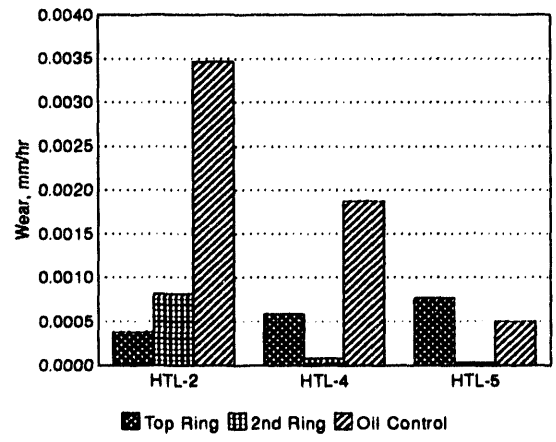


Figure 5.17

Ring Wear
(Final Engine Operating Conditions)

Figures 5.18 and 5.19 present the iron and lead concentration increases in the test oils. Both graphs show a satisfactory low level of wear metal increase for HTL-4 and HTL-5.

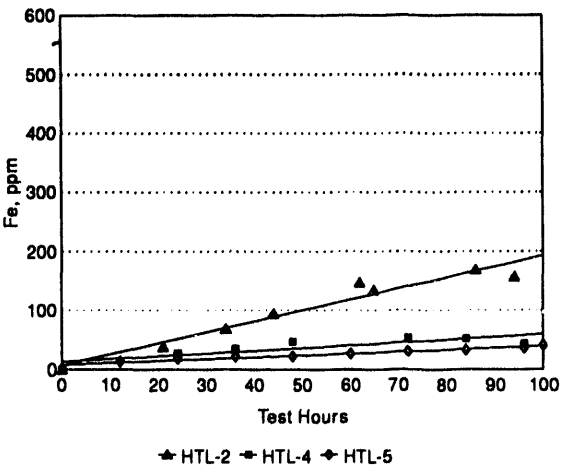


Figure 5.18

Iron Concentration Increase
(Final Engine Operating Conditions)

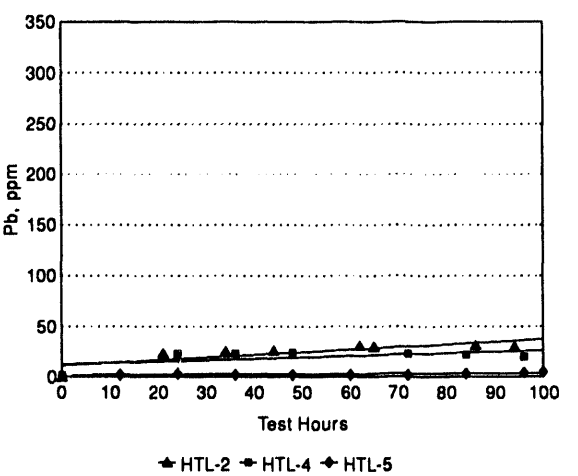


Figure 5.19

Lead Concentration Increase
(Final Engine Operating Conditions)

Piston ring groove deposit ratings are presented in Figure 5.20. There is no significant difference in ratings for each of the lubricants. The deposit ratings for each of the grooves is similar to those presented previously for lubricants HTL-1, HTL-2 and HTL-3 (see Figure 5.9).

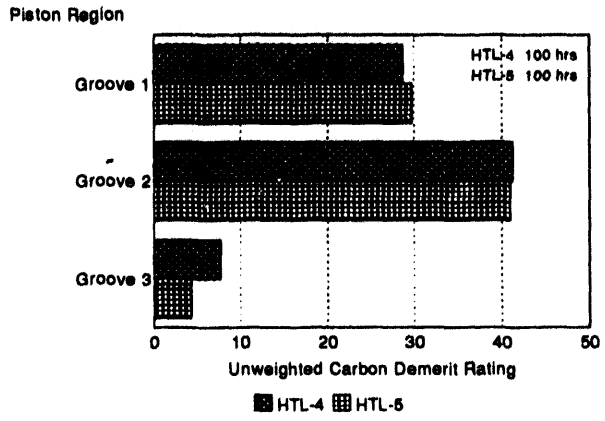


Figure 5.20

Piston Ring Groove Deposit Ratings
(Final Engine Operating Conditions)

Piston land deposit ratings are presented in Figure 5.21. Again, there was no significant difference in ratings for each of the lubricants. However, HTL-4 and HTL-5 performed better than HTL-2 (same base stock). This may be another indication of the lesser severity of the final test conditions and/or benefits from the improved formulations.

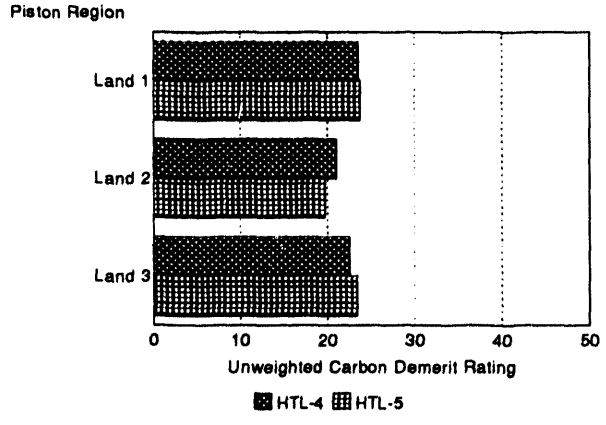


Figure 5.21

Piston Land Deposit Ratings
(Final Engine Operating Conditions)

Figure 5.22 shows the steady state particulate emissions measured for HTL-4 and HTL-5. The lubricants have similar emissions characteristics at 1300 rpm and 1800 rpm. Both lubricants showed an increase in total particulate matter in comparison to HTL-1, HTL-2 and HTL-3 (see Figure 5.11). The increase is due mainly to the increase in dry particulate matter (DPM). The

increase in DPM is directly attributable to changing the combustion process from the production design optimum in order to reduce soot formation at the operating conditions for this evaluation.

The lubricant contribution to particulate for HTL-4 and HTL-5 was lower than HTL-1, HTL-2 and HTL-3. This is most likely due to the reduction of the overall oil consumption at the final test conditions. The fuel contribution to particulate was unchanged.

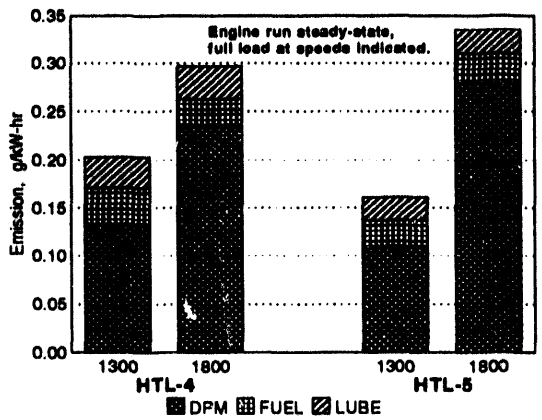


Figure 5.22

Steady State
Particulate Emissions
(Final Engine Operating Conditions)

6.0 CONCLUSIONS

1. Aromatic esters and diesters show the lowest level of deposits compared to other base stocks including polyol esters, poly-alpha-olefins, or super refined mineral oil of comparable viscosity. Within each class of base stock, the level of deposit seems to be viscosity (or possible volatility) dependent to some degree.

2. Changes made in the operating conditions of the Alcor deposition test were ineffective at separating lubricants for high temperature operation.

3. A process to use the Falex panel coker test was most effective for discriminating the deposit characteristics of SDL-1 and SDL-3. Using a 10 second on/60 second off cycle in the panel coker, SDL-1 exhibits dramatically higher deposits than SDL-3. The information available from this test procedure was further amplified by performing one hour deposit tests for each lubricant at a series of temperatures, thus generating a temperature/deposit profile for each lubricant.

4. Conventional diesel detergent inhibitor additive packages were found to significantly contribute to the deposits formed in a lubricant formulated with the aromatic ester. This is evident from the amount of deposits formed from the aromatic ester lubricant SDL-3 at 371°C versus the amount of deposits formed at the same temperature of the aromatic ester itself.

5. The Alcor deposition test results at antioxidant treat levels of 3% and 1.5% illustrate the importance of selecting the proper additive concentration. The antioxidant can be deleterious at the higher levels. The higher antioxidant level offers no advantage in viscosity control and contributes to deposits.

6. The deleterious effect of the overbased detergent on the high temperature stability of the fluid was indicated by a significant viscosity change. However, the incorporation of a small amount of overbase was considered to be necessary to prevent corrosion and control the build up of acid byproducts in the lubricant. A low level of zinc diaryldithiophosphate was also found to be necessary as a coantioxidant.

7. The investigation showed that classical aryl and alkyl ZDP antiwear additives are ineffective in reducing wear with aromatic esters. The phosphate ester was a much better antiwear additive for the aromatic ester. Polyol esters are also found to be much more amenable to treatment with ZDP than the aromatic esters.

8. A range of zeolites and clays was investigated as potential media for a chemically active (acid removal) filtration system.

9. The interaction of Zeolite H with various additive components of EXP-4 was evaluated by selectively deleting individual components from the total formulation and measuring the acid number and base number following treatment with 10 wt% of Zeolite H at 75°C for 72

hours. The results of these experiments demonstrate that Zeolite H does not remove the acidic additives such as the arylamine antioxidants or the aryl ZDP antiwear additive. Some depletion of the overbase is observed. These studies demonstrate that Zeolite H is a viable candidate for the active media of an external acid absorbing filter.

10. HTL-4 (15W-40, Synthetic) is the best overall lubricant in terms of stability, wear control, deposit control, dispersancy and particulate emissions.

7.0 RECOMMENDATIONS

1. The premium mineral oil was not evaluated at the final engine operating conditions. If additional development of HTL-4 is undertaken it is recommended that baseline testing with thepremium mineral oil be performed.

2. The performance of the lubricants in this evaluation was directly affected by the engine hardware performance at non-production, elevated operating temperatures. The high oil consumption data is vivid testimony to the previous statement. Therefore, as future engine designs increase the temperature environment of the lubricant it will be necessary to design the lubricant as another "component" of the engine rather than a generic variable to be dealt with by the customer.

8.0 ACKNOWLEDGEMENTS

This program is sponsored by the U.S. Department of Energy Heavy Duty Transport Technology program under the direction of John Fairbanks through DOE/NASA Contract DEN3-373. The support of NASA project managers, Jack McFadden and Jim Wood are greatly appreciated.

We would also like to acknowledge the contributions of the following researchers at Akzo, Penn State, NIST, ETS, Paru Engineering, and Cummins without whom the program would not have been possible:

Akzo

Dagmar Machan	-	Formulation
Linda Wiles	-	Testing
Dr. M. Kanakia	-	Principle Investigator

Penn State

Prof. E. Klaus	-	Project Manager

NIST

Dr. S. Hsu	-	Project Manager
Dr. J. Perez	-	Principle Investigator

ETS

R. Victor Moore	-	Test Operations
Nancy M. Strete	-	Test Engineering
J. Terry Gray	-	Test Engineering
Russell E. Davies	-	Operations Management

Paru Engineering

Paul A. Bennett	-	Project, Lubricant Engineering

Cummins

Henry B. Stott	-	Test Operations
Irene M. Sorrells	-	Report Compilation
Tom M. Yonushonis	-	Report Compilation
Ed C. Owens	-	Project Management
Jim W. Patten	-	Report Review/Project Overview

9.0 APPENDICES

9.1 Cummins L10 Engine Cylinder Head Cooling Modifications

To achieve elevated cylinder kit temperatures the engine was operated without cylinder liner cooling. The liner coolant cavities were separated from the cooling system by threading pipe plugs into the coolant supply and return ports on each of the six cavities.

Engine oil was used as the cylinder head coolant. Previous single-cylinder L10 engine tests with elevated cylinder kit temperatures resulted in repeated head gasket failures which led to mixing propylene glycol (high temperature engine coolant) with the test oil. The results were ruined tests. To avoid mixing with propylene glycol, the multi-cylinder L10 was operated with test oil as the coolant. If the multi-cylinder head gasket failed, test oil would mix with test oil. The gasket would be replaced and the test continued.

The cylinder head was cooled with a reversed flow of oil. This was accomplished by supplying oil to what would normally be the coolant return manifold. The oil was pumped across the cylinder head and drained through pipes fabricated into the cam box. The drain pipes were located one each under the rocker lever supports so as not to interfere with the valve and injector push rods. These pipes traversed the cam box and exited through the side of the head. To avoid stagnating the coolant at the front and rear of the head two drain ports were fabricated into the head at those locations.

Since the specific heat of oil is lower than that of more typical engine coolants (aqueous ethylene glycol - AEG) it was necessary to nearly double the oil flow to achieve equivalent cylinder head cooling. The engine oil pump could not provide the required pumping capacity. Therefore, a separate pumping and cooling loop was fabricated external to the engine. A schematic of the cylinder head cooling system is shown in Figure 9.1.

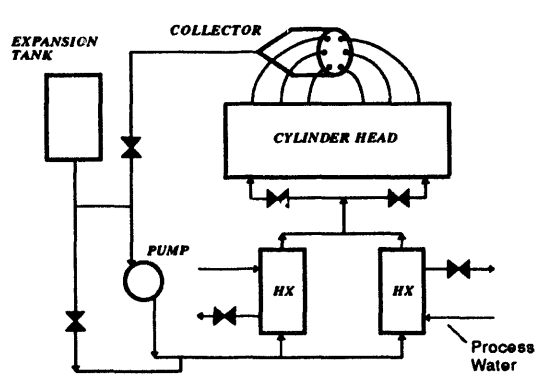


Figure 9.1

Cylinder Head Cooling Schematic

9.2 Lubricant Engine Test Data Summary

The engine testing for this contract was performed at Engineering Test Services in Charleston, South Carolina. Five engine tests were conducted: one test for each of the lubricants. The report summary from each of the engine tests is provided in the following sections.

9.2.1 Engine Test HTL-1

Purpose

To establish a baseline for comparison with experimental oils to be run under NASA Contract #DEN3-373. The tests are to be run in a modified 88LTA10-350 Cummins engine, ESN 34556509, with limited engine cooling and at a 300°F oil sump temperature.

Results

This report summarizes the results obtained from reference tests using HTL-1 engine oil. A period of 14 hours was first run using a special steel top compression ring. The engine was then rebuilt using production top rings and the engine ran an additional 40 hours.

The results at the end of the 40 hours of operation were -

Average Oil Consumption	2.02 lb/hr
Unweighted Average Piston Deposits	174
Crownland Carbon	0
Top Groove Fill	13%
Viscosity Increase, 40°C (at 40 oil test hours)	78%
Used Oil Soot Content (at 40 oil test hours)	4.2%
Liner Condition	Severe Score
Particulate Emissions, 20 oil hours	
1800 rpm, 710 lb-ft	0.27 g/hp-hr
1300 rpm, 880 lb-ft	0.13 g/hp-hr

Conclusions

Engine operation is sufficiently severe that when using HTL-1, an oil with good field performance in commercial engines, the modified L10 engines showed considerable deposits, viscosity increase, and wear. It should thus be possible to demonstrate improved performance using this test to evaluate superior high temperature lubricants.

9.2.2 Engine Test HTL-2

Purpose

To document the performance of an experimental oil, E-92013, run in a modified 88LTA10-350 Cummins engine, ESN 34556509, with limited engine cooling and at a 300°F oil sump temperature. The test was run under NASA Contract #DEN3-373.

Results

Summary test results for oil E-92013 at the end of 94 hours of operation were -

Average Oil Consumption	0.81 lb/hr
Unweighted Average Piston Deposits	208
Crownland Carbon	0
Top Groove Fill	25%
Oil Ring Plugging	Severe
Sludge	Heavy Black
Viscosity Increase, 40°C	455%
Used Oil Soot Content	7.9%
Liner Condition	Moderate Scores, Light Scratches

Particulate Emissions, 94 oil hours

1800 rpm, 710 lb-ft	0.15 g/hp-hr
1300 rpm, 880 lb-ft	0.09 g/hp-hr

Conclusions

The liner scoring observed with oil E-92013 was much reduced over that observed in tests HTL-1/1A. In addition, considering the hours on test, piston deposits were also much reduced. However, oil ring plugging was severe, and the entire engine was coated with a heavy layer of tenacious black sludge. The used oil soot content was very high as was the oil viscosity increase.

9.2.3 Engine Test HTL-3

Purpose

To document the performance of an experimental oil, E-92013, run in a modified 88LTA10-350 Cummins engine, ESN 34556509, with limited engine cooling and at a 300°F oil sump temperature. The test was run under NASA Contract #DEN3-373.

Results

Summary test results for oil E-91043 at the end of 50 hours of operation were -

Average Oil Consumption	1.64 lb/hr
Unweighted Average Piston Deposits	159
Crownland Carbon	0
Top Groove Fill	14%
Oil Ring Plugging	15%
Viscosity Increase, 40°C	51%
Used Oil Soot Content	3.8%
Liner Condition	Light Score
Turbo	Failed
Rod Bearings	Failed

Particulate Emissions, 20 oil hours

1800 rpm, 710 lb-ft	0.12 g/hp-hr
1300 rpm, 880 lb-ft	0.09 g/hp-hr

Conclusions

The relatively low piston deposits probably partially reflects the short test hours. Liner distress was relatively light although again some scratches continue to the top of top ring travel. Others stop at the top of second land travel. There was no appreciable sludge observed. Oil viscosity increase was moderate as was the used oil soot content. The used oil wear metals started to increase as early as 12 test hours. The generation of this used oil debris probably was responsible for the turbo and bearing failures observed.

9.2.4 Engine Test HTL-4

Purpose

To document the performance of an experimental oil, E-92013, run in a modified 88LTA10-350 Cummins engine, ESN 34556509, with limited engine cooling and at a 250°F oil sump temperature. The test was run under NASA Contract #DEN3-373.

Results

Summary results for test HTL-4 after 100 test hours were -

Operational	
Oil Consumption, avg	0.54 lb/hr
Used Oil	
Viscosity Increase (40°C)	85%
Soot Content	1.0%
Wear	
Top Compression Ring Gap Increase	0.00230"
Liner	Very Low Wear
Wear Step, Avg	0.00015"
Connecting Rod	
Small End Bushing Condition	Poor
Bearing Inserts	Very Low Wear, Polished
Deposits	
Unweighted Average Piston Demerits	174
Crownland Carbon	0
TGF	12%
Undercrown Demerits	24%
Oil Ring Plugging	Low
Pan Sludge	9.7
Piston Pins	Discolored
Particulate Emissions	
1800 rpm, 710 lb-ft	0.20 g/hp-hr
1300 rpm, 880 lb-ft	0.10 g/hp-hr

Conclusions

This test was run after making several changes in engine setup and operating conditions intended to reduce used oil soot levels and connecting rod bearing problems. The test modifications were successful from the standpoint that soot levels were reduced, piston deposit levels were reduced, and connecting rod bearing wear was essentially eliminated, including liner scuffing.

The condition of the piston pin bushings in the piston were much poorer than in previous tests, and significant deposit was observed on the piston pins. The extent of the deposit on the piston pins was much greater than that observed before.

It is not known if any of the above observations are the result of the use of an engine oil which was different than the previously used oils.

9.2.5 Engine Test HTL-5

Purpose

To document the performance of an experimental oil, E-92013, run in a modified 88LTA10-350 Cummins engine, ESN 34556509, with limited engine cooling and at a 250°F oil sump temperature. The test was run under NASA Contract #DEN3-373.

Results

Summary results for test HTL-5 after 100 test hours were -

Operational	
Oil Consumption, avg	0.53 lb/hr
Used Oil	
Viscosity Increase (40°C)	82%
Soot Content	0.7%
Wear	
Top Compression Ring Gap Increase	0.00302"
Liner	Very Low
Wear Step, Avg	0.00017"
Connecting Rod	
Small End Bushing Condition	Poor
Bearing Inserts	Good, Some Polish
Piston, Pin Bushing	Poor

Deposits

Unweighted Average Piston Demerits	171
Crownland Carbon	0
TGF	7%
Undercrown Demerits	25%
Oil Ring Plugging	11%
Pan Sludge	9.6
Piston Pins	Discolored

Particulate Emissions

1800 rpm, 710 lb-ft	0.24 g/hp-hr
1300 rpm, 880 lb-ft	0.12 g/hp-hr

Conclusions

Oil consumption and the used oil condition was similar at that observed in test HTL-4. Engine wear was generally comparable except for the piston pin bushing in the piston. These were in worse shape than in HTL-4. Piston pin deposits (discoloration) were also different. General engine deposits were similar to those in HTL-4.

DATE
FILMED
8/16/94

END