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LUBRICANTS QUALITY ANALYSIS SYSTEM DEVELOPMENT

INTERIM REPORT
BFLRF No. 293

Volume I

Performance Measurements by Potential Portable Test Kits

By

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13. ABSTRACT (Maximum 200 words)

In the field or depot, the equipment user needs an answer, usually within a few minutes or hours, as to the condition of his mechanical components and of the lubricant or fluid in those components. Present petroleum testing operations, while adequate in controlled peacetime environments, are inappropriate for use in a faster, more mobile future battlefield.

Personnel from Mobility Technology Center-Belvoir and Belvoir Fuels and Lubricants Research Facility selected the criteria, techniques, equipment devices, and test methodology required to accurately evaluate the condition of in-service lubricant and fluids. A midproject redirection of this work effort to assess the quality of new and unused lubricants and fluids did not change the basic approach. During this evaluation, the techniques, technologies, and kit devices required some modification and refinement decisions.

Eighteen test devices were evaluated, some of which were used for more than one test technique. Approximately ten devices, with minor modifications, could be hardened for use with the Lubricant Quality Analysis (LQA) System. Four of the devices were man-portable and could be used for maximum forward tactical testing, while six were transportable and could be used for operational and control theater testing.

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EXECUTIVE SUMMARY

Problems: In the field or depot, the equipment user needs an answer, usually within a few minutes or hours, to the following questions:

(1) What is the condition of the mechanical component (i.e., engine, transmission, final drive, hydraulics)?

(2) What is the condition of the lubricant in the mechanical component, the lubricant shelf-life quality, and the condition of the possible replacement lubricant/fluid products?

(3) How can premature lubricant/fluid changes be reduced or eliminated along with misapplication of the lubricant/fluid?

(4) How can equipment failures be reduced or eliminated?

The Army's Oil Analysis Program (AOAP) has aided in answering the first and fourth questions. However, answering questions two and three is more difficult in that no acceptable method of rapidly establishing the lubricant condition for inservice, stored, captured or host nation lubricants has been found by which operators and maintenance personnel can assess lubricant quality quickly.

Objective: The objective of this project was to identify the criteria, techniques, equipment, and test methodology required to accurately evaluate the condition of inservice lubricants and fluids and to assess the quality of new and unused lubricants and fluids. The results of these assessments will then be used to develop a Lubricant Quality Analysis (LQA) System.

Importance of Project: Modern military combat and tactical equipment have complex and expensive components that require exacting specifications. A major requirement for successful operation and maintenance of such equipment is an adequate supply of proper lubricants and fluids. Present petroleum testing operations, while adequate in controlled peacetime environments, are inappropriate for use in a faster, more mobile future battlefield. The ability to use captured enemy, unknown, or host nation lubricant products will be important to the future commander. There is a strong need to develop performance-measuring equipment capable of on-site determination of the condition and quality of the lubricants and fluids as far forward as possible in battlefield conditions. This equipment should be state-of-the-art, transportable, and some tests man-portable; all equipment should be integrated toward computer compatibility.

Technical Approach: This effort was initiated with a literature search to identify currently available portable kits or devices and to review currently developing technologies in lubricant monitoring that should be transportable or man-portable. In conjunction, Mobility Technology Center-Belvoir (MTCB) and Belvoir Fuels and Lubricants Research Facility (BFLRF) personnel selected the criteria, kit devices, and techniques or technologies to evaluate the condition of inservice lubricants. The devices and technologies that demonstrated the most promise would be developed for use as a Portable Lubricant Quality Monitor (PLQM). The selected techniques or technologies, kit devices, and condemning criteria performance would be evaluated and correlated where possible to ASTM standards using selected lubricant samples from various reference standards, engine tests, and AOAP. A midproject redirection of this work effort to verify new lubricants in the field did not change the basic approach. During this evaluation, the techniques, technologies, and kit devices required substantial modification and refinement decisions.

Accomplishments: Eighteen test devices were evaluated, some of which were used for more than one test technique. Approximately ten devices, with minor modifications, could be hardened for use with the LQA System. Four of the devices were man-portable and could be used for maximum forward tactical testing, whereas six were transportable and could be used for operational and control theater testing.

Military Impact: The establishment of the LQA System would increase vehicle and equipment readiness and meet requirements in the following areas:

- Logistics – Assess the useful life of new lubricants and functional fluids and ease the logistics burden.
- Environmental – Reduce the impact of used drained lubricants and functional fluids disposal.
- Maintenance – Extend the useful life of stored, used, or new lubricants and functional fluids, helping to eliminate the erroneous use of lubricants and fluids and reduce maintenance costs.

FOREWORD/ACKNOWLEDGMENTS

This work was performed at the U.S. Army Belvoir Fuels and Lubricants Research Facility (BFLRF) located at Southwest Research Institute (SwRI), San Antonio, TX, during the period October 1988 to September 1993 under Contract Nos. DAAK70-87-C-0043 and DAAK70-92-C-0059. Work was funded by the U.S. Army TARDEC, Mobility Technology Center-Belvoir (MTCB), Fort Belvoir, VA. Mr. T.C. Bowen (AMSTA-RBFF) served as the contracting officer's representative, and Mr. M.E. LePera (AMSTA-RBF) served as the technical monitor.

The author acknowledges the assistance provided by the staff of his organization and that of MTCB, with special recognition to the following: Dr. P.I. Lacey for his consultation in the wet-friction/clutch wear test development, Mr. T.C. Bowen for his technical guidance, Mr. E.A. Frame for his technical counsel and friendship, and Mr. J.W. Pryor, Ms. L.A. Pierce, and Ms. M.M. Clark for editing and report preparation.

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I. INTRODUCTION AND BACKGROUND

Oil analysis of internal combustion engines and other power-train systems has been a widely used maintenance tool within both the industry and the military services to assess or predict component failure and to establish oil drain intervals. Although the two functions reflect differing analytical technologies, they are complementary but not necessarily dependent upon each other. The Army Oil Analysis Program (AOAP) has aided greatly in defining the condition of the engine or component. However, the AOAP tests primarily determine the condition of the components rather than the lubricant or fluid. There is concern that oil analyses conducted in the current AOAP may not adequately detect degradation of used engine oil when the oil degradation is not accompanied by obvious engine problems such as a high wear, fuel, or coolant contamination. Similar concerns exist for power transmission oils. This situation is particularly important as oil drain intervals are becoming longer and are based on oil condition rather than established time or mileage intervals.

The current AOAP used oil analysis protocol includes the following:

- Viscosity density product;
- Elemental analyses (i.e., wear metals, lubricant or coolant additives, airborne contaminants);
- Crackle test for water content; and
- Blotter test for total contamination, coolant, alkalinity, and dispersancy.

Qualitative laboratory analysis guidelines (normal, marginal, high) are available for elemental analyses. While these tests appear to provide adequate information concerning **equipment** condition, they may not be adequate to define **used oil** condition. Additive depletion of a used oil must be carefully monitored. For example, a minimum total base number (TBN) of 1.0 to 2.0 is often used as an indication of reserve alkalinity, additive depletion, and needed oil drain.

Also, a total acid number (TAN) increase to 5.0 is often used as an oil drain indicator. Insoluble contents of greater than 1.0 percent is another quantitative oil drain guideline. The current AOAP procedures do not provide **quantitative** data concerning these important oil degradation properties.

Recent developments in additive technology and oil formulations have impacted the monitoring of used oil elemental analyses as oil drain criteria. Current engine oil formulations may now contain substantial quantities of added copper, boron, and silicon, which had been previously associated with engine wear or contamination. This added material obviously confounds oil drain recommendations. As a result, there is concern that AOAP data interpretation may need revision. Currently, wear metals limits are set based on an absolute maximum level. It may be better to use a combination of criteria that includes a maximum level and a trend indication such as an increase from the last sample.

As a result of these concerns, a cooperative program was proposed for Ft. Knox, KY, to initiate a resolution of this "oil condition" aspect of AOAP. Due to a lack of funding, the work was stopped, and on 22 March 1989, a summary letter report, "Improved Used Oil Analysis," was issued covering this effort.(1)*

Even though the AOAP has aided greatly in defining the condition of the components, it takes three to ten days for the test results to reach the user of these systems. In the field, the user wants an answer, usually within a few minutes or hours, to the following questions: (1) What is the condition of the mechanical component (i.e., engine, transmission, final drive or steering pump)? (2) What is the condition of the lubricant in the mechanical component? (3) How can premature oil changes be reduced or eliminated? and (4) How can equipment failures and removals be reduced or eliminated? The AOAP has aided in answering the first and fourth questions. However, answering questions two and three is more difficult in that no acceptable method of rapidly establishing in-service lubricant condition has been found by which maintenance personnel can schedule oil changes based on immediate on-site determinations of lubricant condition.

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* Underscored numbers in parentheses refer to references at the end of this report.

The concept of a test kit to rapidly establish in-service or used lubricant condition is not new to the Army. Interim Report AFLRL No. 117, entitled "Feasibility of Field Test Kits for Assessing In-Service Condition of Army Engine Oils," AD A081112, October 1979.(2), presented work performed in this area. Articles in the open literature recounting technological developments since the 1979 study have shown the potential for developing a portable device that can rapidly determine oil condition qualities using physical and chemical tests. Additionally, the proposed effort to develop a device for on-site oil analysis was made by the Deputy Commanding General for Material Readiness in 1987, following an Inspector General review of the U.S. Army Oil Analysis Program (AOAP).(3)

Present petroleum testing operations, while adequate in controlled peacetime environments, are insufficient in a faster, more mobile future battlefield. The ability to use captured enemy, unknown, or host nation petroleum products will be important to the future commander. The type and quality of petroleum products must be verified rapidly and on-site if the products are to be of use to U.S. forces during a conflict. The system should operate worldwide and be able to support any level of conflict.

The need to make military combat and tactical equipment more proficient has resulted in the use of more complex and expensive components with more exacting requirements. A major requirement for the successful operation and maintenance of such equipment is an adequate supply of the proper fuels, lubricant, and other fluid petroleum products. To assure a reliable and rapid response to problems related to quality of mobility petroleum products, the use of emerging state-of-the-art instrumentation, providing multifunctional test capabilities, is essential. Such equipment will provide field commanders with necessary data about the usability of petroleum products and will result in reasoned recommendations in a short response time.

Thus, there is a strong need to develop analytical chemical/physical property and performance-measuring equipment capable of ensuring quality control of petroleum products as far forward as possible in battlefield conditions. This equipment should be transportable, and some tests possibly man-portable, to conduct the quality assessment of fluids and lubricants at forward Army facilities. The use of this equipment will identify specific petroleum products and determine the compliance of the lubricants with the respective specifications. Such systems should also identify products of host nations and products of a commercial or unknown source that may be used instead of fully acceptable products [listed on the Qualified Products List (QPL)] either as an alternative or as emergency petroleum commodity for short-, medium-, or long-term usage.

Many state-of-the-art analytical tools require a relatively high degree of technical expertise, both in the ability to use the instrument and to interpret the resultant data. Expert systems that draw well-reasoned and expedient conclusions from results obtained from a battery of analytical methodologies need to be developed into a coherent computer program, and this program must become an integral part of the Petroleum, Oil, Lubrication (POL) instrumental package.

Additionally, extensive research will be required to correlate the data resulting from modern instruments to standard American Society of Testing and Materials (ASTM) inspection-type tests. This work is essential in bridging the possible technology gap between modern Army technologies and traditional ASTM methodologies, as industry will continue to use ASTM tests as its reference benchmarks. It should also be noted that ASTM Committee D-2 moves slowly in adopting new methodologies.

The initial intent of this program was to develop a one-person portable, hand-held, on-the-spot analysis device(s) capable of determining the quality of in-service used oils. This Portable Lubricant Quality Monitor (PLQM) was to be used to assess oil change intervals in combat and tactical ground vehicles and equipment in motor pools or other direct support/general support (DS/GS) maintenance locations. Primary emphasis was placed on MIL-L-2104 diesel engine lubricants.

The determination of used oil quality is a maintenance function. However, in FY89, a new oil quality problem surfaced, and the capability of evaluating new oil quality became an urgent need. This requirement was then added to the PLQM specification, which increased the design requirements. Assessing new oil quality is a Quartermaster School (QMS) responsibility, and the QMS is not the proponent of the used-lubricants maintenance function. Therefore, the QMS could not support the PLQM as a hand-held maintenance device to be used by the troops.

Instead, the QMS needs a transportable or portable device to be used primarily for quality assurance of new lubricants and fluids in the field. Since the QMS has been the proponent of the Fuels and Lubricants Field of Endeavor, its guidance is important. In an effort to persuade QMS to support the PLQM, BFLRF (SwRI) developed comments and input tracing the chronology of events identifying/justifying the need for the then proposed PLQM. This is shown in Appendix A.

For the reasons noted above, BFLRF prepared the briefing package shown in Appendix B for the Lubricants/Fluids Quality Analysis System (LFQAS) dated 30 August 1990. This briefing package was developed in concert with QMS-MTCB-BFLRF as a result of visits, letters, telephone conversations, and other discussions related to MTCB's new Petroleum Quality Analysis (PQA) System. The proposed LFQAS was intended to be an integral part of the PQA System, which is being developed to provide petroleum support to the AirLand Operations. It appears that much of the initial PLQM used lubricant work would have been applicable for LFQAS use. The LFQAS addresses the need for assessing quality of lubricants and fluids in the field in order to fill an existing need for improved oil shelf-life retesting and to accommodate greater use of lubricants and fluids available from host nation support agreements and from commercial sources. However, it is essential that the developer of the requirements for lubricants and fluids continuously obtain feedback from used lubricant property results derived from limited sampling. These properties should be established neither in support of AOAP nor for equipment maintenance purposes but to help determine new oil property improvements and to define mission logistics requirements. A logical extension of this system is that it could be established as a system that meets requirements in the following areas:

- Logistics – Assess the quality of new lubricants and functional fluids and ease the logistics burden.

- Environmental – Reduce the impact of used drained lubricants and functional fluids disposal.

- Maintenance – Extend the useful life of stored, used, or new lubricants and functional fluids, helping to eliminate the erroneous use of lubricants and fluids and reduce maintenance costs.

The new effort is an integral part of the MTCB's PQA System that is being developed in support of the QMS Petroleum Field Testing Concept Statement.

Based upon the discussed FY91 redirection of future studies, this effort has focused on the quality assessment of new lubricants and fluids. Two parallel work efforts were established. Task I addresses performance acceptance measurements, and Task II includes chemical composition and physical property measurements. Task I is discussed in Volume I of this report, while Task II is discussed in Volume II.

II. OBJECTIVE

This program initially had two concurrent and interrelated tasks. The objective of Task I was to identify the criteria, the techniques, and the equipment required to accurately evaluate the condition of in-service lubricants in the field and to develop a PLQM. However, a new oil quality problem surfaced, and the evaluation of new lubricant quality was added. Task II was a cooperative effort between MTCB, TACOM, and the Material Readiness Support Activity (MRSA) to determine if the current AOAP testing protocol was providing adequate information regarding the condition of in-service oil (i.e., overall deterioration and serviceability). If deficiencies in the AOAP protocol were observed, revisions to the test protocol and on-condition change criteria would be recommended to alleviate the observed deficiencies. Subsequently, these findings will be employed in the development of a field PLQM that determines the condition of the lubricant in service.

In FY89, a new oil quality problem surfaced, and the capacity for evaluating new oil quality became an urgent need and was added to the work program. However, the objective of the program was redirected in FY91. The redirected objective was to identify the criteria, technologies, equipment, and test methodology protocol required in assessing the quality of new and unused lubricants and fluids and to employ these findings in the development of a Lubricant Quality Analysis (LQA) System. The LQA System would be an integral component of the MTCB's PQA System, which would support the Petroleum Field Testing Concept Statement of the QMS. This effort focused primarily on the assessment of the quality of new lubricants and, to some extent, used lubricants. Two parallel efforts were made. Task I addressed performance acceptance requirements, while Task II included state-of-the-art chemical composition and physical property measurements and development of correlation models.

A summary of program redirections is provided in the following listing:

Redirection	Date
1. Portable Lubricant Quality Monitor • Condition of in-service lubricant	1988
2. Improved Used Oil Analysis • AOAP procedures versus ASTM/manufacture	1988
3. Lubricant/Fluid Quality Analysis System – New and Used Lubes • In-service condition plus new lubricant quality	1990
4. Petroleum Quality Analysis System • Assessing quality of new or unused lubricants and fluids in field • Oil shelf-life retesting • Host nation and commercial lube testing • Unknown source (captured lube)	1991

III. APPROACH

When this effort was initiated in FY 1988, a comprehensive literature review was performed to identify the then available portable kits or devices from tests that could be conducted in the field. From this review, it was apparent that the only real changes in field test kits since 1979 were in degree of sophistication, rather than in technological developments. Due to limited funding, the field test kits on hand at Belvoir Fuels and Lubricants Research Facility (BFLRF) were assembled and evaluated. However, more recent information available from technical literature, industry personnel, and Condition Monitoring and Preventive Maintenance meetings indicated that new laboratory technologies are being developed for in-service lubricant monitoring. Therefore, another literature search and personal contact with developers were conducted in FY 1989 to identify currently available laboratory technologies employed in lubricant monitoring.

A meeting was held between MTCB and BFLRF personnel to discuss the criteria and techniques to evaluate the condition of in-service or the remaining life of used lubricants for the PLQM. At the meeting, it was decided that initial emphasis should be on Army diesel engines but that the program should include some transmissions and hydraulic work. The major obstacle was to evaluate in-service lubricants without the background knowledge of those lubricants. No technologies readily adaptable for use as a PLQM would meet the performance requirements established by the MANPRINT Management Plan. These requirements were that the test kit

1. Be lightweight, approximately 5 pounds, and capable of withstanding rough handling with only nominal protection;
2. Be portable and operable by one soldier;
3. Require no sample preparation;
4. Be simple to calibrate and operate with no special tools;
5. Provide on-site immediate analysis; and
6. Require no more than 10 mL of product sample.

These requirements were not immediately obtainable because the assessment of oil quality is a complex process due to the additive package technologies used to meet the wide range of lubricant performance requirements by Army combat and tactical equipment. It was decided to select the used lubricants critical criteria first. The critical areas of concern selected for assessing the used lubricant were as follows:

- Viscosity
- Oxidation
- Insoluble contamination
- Coolant/water contamination

- Dispersancy
- Acidity
- Wet-friction performance
- Fuel dilution
- Use of wrong type or grade
- Wear debris.

The various technologies obtained as a result of the literature survey and personal organization contact were discussed, and the following technologies were selected for further evaluation:

- Coolant/Water Contamination
  - Gly-Tek
  - Dielectric Constant
- Viscosity Technique Test
  - Electromagnetic Viscosity
- Electrochemical Reaction Tests
  - Cyclic Voltammetry
  - AC Impedance
  - RULLER Device
  - COBRA
  - RULLER TAN and TBN
  - Dexsil TBN
  - pH
- Fourier Transform Infrared Spectroscopy (FTIR)
- Laser Oil Spot Scanning Test
- Conductivity/Dielectric Tests
- Wet-Friction Tester
  - Ball-on-Cylinder
  - Cameron-Plint
- Deposition and Oxidation Tests
  - Microoxidation
  - LUBTOT
  - TFOUT
  - TDN.

After the technology areas were identified, the techniques and test devices were evaluated as they were developed and could be obtained and procurred, which covered a three-year time frame.

The ultimate goal is to develop for use as a PLQM those techniques that demonstrate the most promise in conjunction with critical criteria. In the interim, it may be necessary to develop small hand-held units capable of identifying severely contaminated lubricants or fluids of improper viscosity range, etc. These selected techniques, along with portable test kits that have been proven to be effective, will be used to analyze new, used, stressed, and blended lubricants, and the results will be compared to ASTM tests, if possible. This work, along with the present understanding of mechanisms of lubricant degradation, will be used to establish the condemning criteria and limits. The condemning criteria will be focused on 1) rise in oil acidity, 2) decrease in oil alkalinity, 3) increase or decrease in viscosity, 4) a rise in contaminants, and 5) wet-friction performance. However, the criteria will not be limited to these factors. Published used oil limits by various engine manufacturers will be considered. Best judgment by experienced scientists will be utilized to provide a basis for defining the oil condemnation limits. This work should predominantly include the diesel engine lubricants from the MIL-L-2104 tactical engine oil specification, along with some manufacture reference specification lubricants. However, a new oil quality problem surfaced in the field, and the capability of evaluating new oil quality was added. The selected techniques, portable kits, and condemning criteria performance will be correlated and evaluated with oil samples from engine tests being conducted at BFLRF, other Southwest Research Institute (SwRI) facilities, and those samples obtained in Task II of this project. These samples include tanks, artillery, etc., and samples from the Evaluation of Lubricant/By-Pass Filter (4) project being conducted at BFLRF. Prudent acquisition and utilization of these test samples will provide a basis for allowing further development of a prototype.

The FY91 redirection of this work effort to evaluate new lubricants in the field resulted in a modification of the goals. The revised goals were to test and design performance acceptance tests for LFQAS use that would be transportable or man-portable, be of the go or no-go type, use small quantities of lubricant, give fast results, and operate in conjunction and be compatible with composition or physical property tests discussed in Volume II. A technology-based effort is required to develop a fully functional performance testing system for use in the three PFT areas: 1) control theater testing (product usage quality for 40 days and beyond), 2) contact operational testing (product quality for 10 to 40 days), and 3) forward tactical testing (product quality for 7 to 10 days).

The most promising test techniques were to be developed as bench lubricant performance and correlated with standard ASTM tests or other engine/power transmission performance tests. The lubricants used for this performance development and correlation included engine and power transmission specification lubricants, and selected lubricants with various additive packages. The lubricants were then expanded to include several lubricants with well-known base stocks and additive packages. It was important to obtain a diverse sample set in order to generate as universal an application as possible. Where possible, the results of the performance measurement tests were compared to those from the results of the instrumental analysis from Volume II to help predict engine or power transmission performance parameters. During this evaluation, these tests required substantial modifications and refinement decisions.

IV. DISCUSSION OF RESULTS

A literature search was conducted to identify any test that could be run in the field and be incorporated into a portable test kit for use as a lubricant analyzer. The search encompassed the years since 1979, when BFLRF had previously investigated the tests and test kits available. The focus of the current search was for a portable field kit that contained one or more of the following tests or measurements: viscosity, total acid number, total base number, dispersancy, and presence of glycol, insolubles, or contamination.

The databases searched were Chemical Abstracts, National Technical Information Service, and Compendex. Although 98 documents meeting the search criteria were obtained, only four references were pertinent. In addition to this computerized search, a manual search of Chemical Abstracts was made for specific tests. Several potentially valuable references were found, and additional information was requested from the authors of the abstracts. The 1987 *Thomas Register* was also examined for companies manufacturing or selling oil testers. Those companies offering portable tests or test equipment of interest were contacted, and additional information was requested.

A. Initial Field Kit Testing

The kits and field tests already available at BFLRF from prior work were assembled and inspected. These test kits included the Gly-tek, pH, and the Lubri-Sensor (dielectric) kits. Arrangements were made to obtain a large quantity of used oil samples from laboratory engine tests in order to experiment with new procedures being identified. Also included with these used oil samples were analyses of the oil before and after the engine test, plus samples of the unused oils. Selected used oil samples being generated in the AOAP-Ft. Stewart and TACOM-Ft. Lewis programs were evaluated in the portable lube quality monitor project.

New and used oil samples were prepared containing 0.25, 0.50, and 1.00 wt% ethylene glycol antifreeze. These prepared samples, along with a blank, were evaluated with the Gly-tek test kit for the presence of ethylene glycol. As expected, all samples containing glycol indicated positive when tested, while the blank (without glycol) sample did not. The test is relatively simple to conduct and should be a good candidate for inclusion in the PLQM.

BFLRF laboratory analyses on samples received from the AOAP included determinations for TAN and TBN by ASTM D 664 procedure. From the AOAP samples available, nine were arbitrarily selected, and the pH of each sample was determined. This procedure involved blending 0.5 gram of the oil sample with a solvent mixture composed of 50 vol% toluene, 49.5 vol% isopropanol, and 0.5 vol% water. TABLE 1 compares the TAN and TBN of each sample to its pH. Also included in TABLE 1 are dielectric measurements made on these samples and the AOAP results from the Alkalinity Blotter Spot Tests and the ASTM E-1131 Thermal Gravimetric Analysis (TGA) Soot Test (5) conducted at BFLRF.

The dielectric measurements were made with a commercially available oil quality analyzer (Lubri-Sensor). This kit is also discussed in Section IV, Part I with the Complete Oil Breakdown

TABLE 1. Comparison of Laboratory-Determined Properties of AOAP Used Oil Samples

Sample No.	Engine Model	ASTM D 664 TBN	ASTM D 664 TAN	Relative Dielectric Measurement	TGA Soot	pH	AOAP Alkalinity Blotter Spot Test
16262	LDS 465-1	4.8	4.3	4.8	1.9	5.2	Bad
16263	NHC-250	5.5	2.2	2.3	0.6	6.7	Good
16264	LDS 465-1	4.6	1.8	2.0	0.5	6.0	Good
16265	LDS 465-1	2.8	3.9	4.5	1.7	4.5	Bad
16266	NHC-250	6.4	2.5	1.8	0.5	6.7	Good
16267	LDS 465-1	5.5	2.5	3.0	0.9	5.9	Good
16268	LDS 465-1	6.3	2.2	1.8	0.5	6.7	Good
16269	LDS 465-1	3.6	2.9	4.9	1.9	5.2	Good

Analyzer (COBRA). This portable instrument has been previously used at BFLRF with some success. The analyzer detects changes in an oil due to various types of contaminants such as acids, oxidation, water, antifreeze, and fuel. Its sensor system, based on thin-film technology, measures the dielectric property of the used oil as compared to a sample of the same unused oil. Its main disadvantage is that new oil samples are not always readily available to calibrate the instrument. Since the new unused oils were not available with the eight AOAP used oil samples, a qualified MIL-L-2104, SAE grade 15W-40 oil from a previously unopened container was used as a basis for this comparison. Figs. 1 and 2 show that the dielectric measurements correlate well with the TGA soot measurements and indicate a general trend with TBN, TAN, and pH. Samples with high dielectric values (16262, 16265, 16269) also have relatively low pH values. These three samples also have the highest TAN and TGA soot values, with two of the samples having a bad rating using the blotter test. It might be suspected that the third sample with the good rating (16269) might be borderline since the contaminants were rated medium and the dispersancy was rated fair by the same AOAP blotter test. A follow-up sample taken about 2 months later from the same vehicle with the same oil indicated heavy contaminants and poor dispersancy by the blotter test, although the alkalinity rating was still given as good.

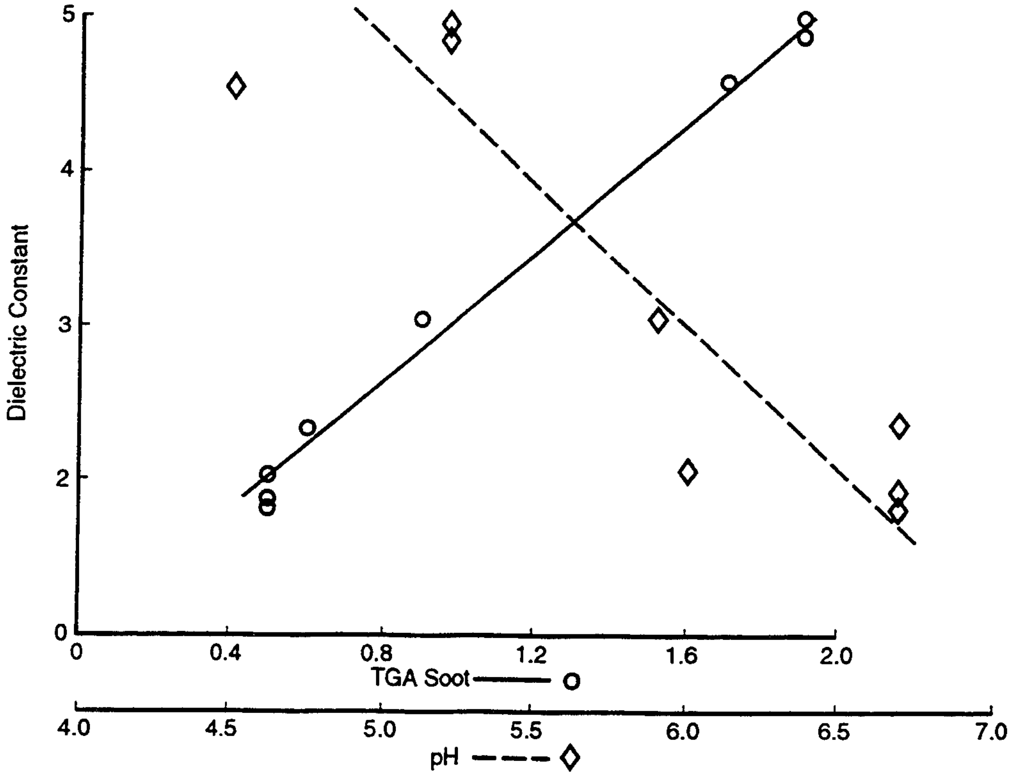

Figure 1. Dielectric constant versus TGA soot and pH

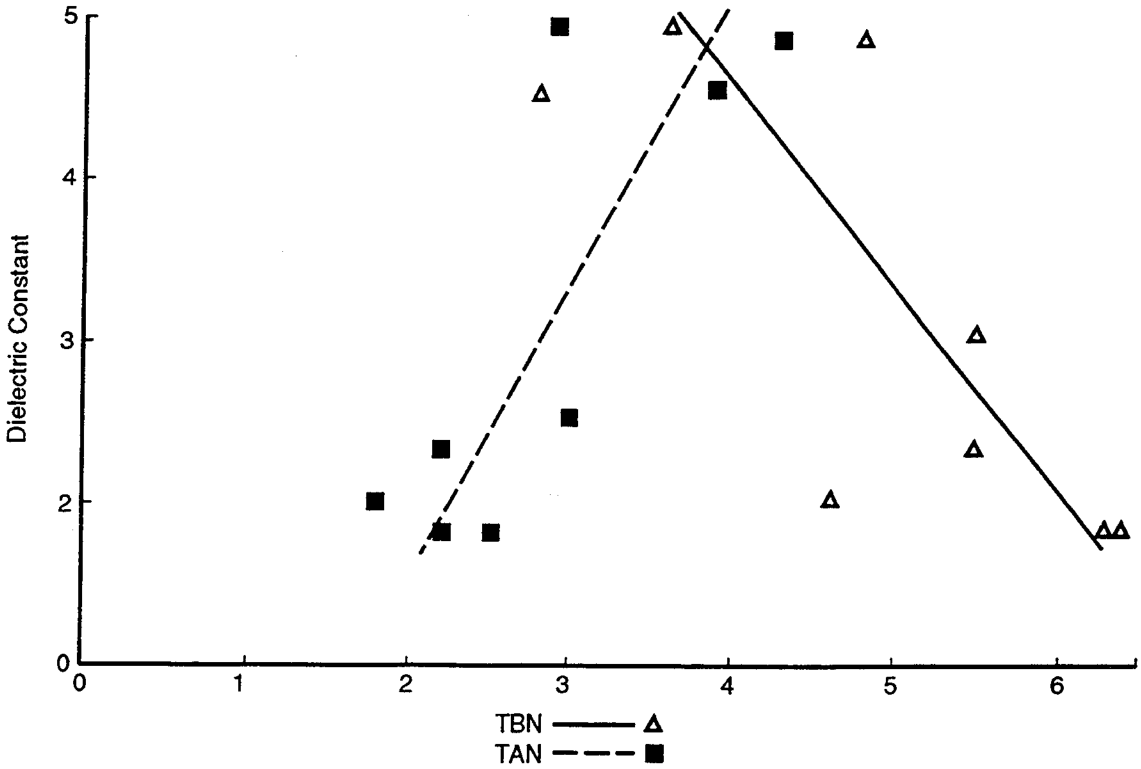

Figure 2. Dielectric constant versus ASTM D 664 TAN and TBN

B. Fourier Transform Infrared Spectrophotometer

A preliminary study was conducted to evaluate a commercially available used oil analyzer by Nicolet Instruments. The Nicolet Model 8210 used oil analyzer is a special-purpose Fourier transform infrared (FTIR) spectrophotometer that compares the spectrum of the used oil with the spectrum of the new oil from which the used oil originated. The computer then calculates the results from the differences between these spectra. The FTIR spectrophotometer uses an integral horizontal attenuated total reflectance (ATR) sample cell. It operates with software that performs automatic quantitative procedures especially designed for analysis of gasoline or diesel engine lubricants. New oil reference spectra are stored in the memory and may be called out individually for comparison to used oils or can be automatically selected by the software for a best fit to the used lubricant. Up to 40 reference lubricants may be stored on a single floppy disk, which also contains the operating software.

Sample preparation consists of spreading a layer of new or used lubricant in a sample trough. Cleanup between test samples is accomplished by wiping the trough with clean tissues and a solvent. The instrument checks for sample cell cleanliness between runs.

Time required for the analysis is under 30 seconds, including sample changeover. The information printout obtained from this analysis is shown in TABLE 2.

TABLE 2. FTIR Analytical Information Printout

Sample ID:
New Oil Reference:

Oxidation	Carbonyl group level in absorbance
Sulfation	Sulfate group level in absorbance for diesel oils
Fuel Dilution	Level in wt%
Glycol	Level in percent
Water	Level in percent
Soot	Percent transmittance value
Nitration	Nitro group level in absorbance for gas engine oils

"Antifreeze interferes with water values"

Samples were blended by weight using a diesel lubricant and a 20-percent bottoms portion of diesel fuel. The diesel fuel 20-percent bottoms were prepared by distilling off 80 percent of the fuel in the ASTM D 86 apparatus to approximate fuel that might be collected in the exhaust particulate fractions.(6) The results of analyses of these blends and other samples are shown in TABLE 3. The lubricant used for blending was selected as the reference lubricant for the standard samples. When the new lubricant is known, the FTIR measurement is very accurate down to approximately 2-percent fuel dilution. However, if new lubricant is not available, the accuracy is not as good. Agreement with the AOAP method for measuring fuel dilution (based on viscosity change) is fairly good when auto references are chosen. The 30-second analysis time translates into a good savings when compared with 15 minutes for a viscosity measurement and 1 hour for gas chromatography (GC) fuel dilution.

TABLE 3. Fuel Dilution Evaluation

Sample	Test Sample Reference	Nicolet Fuel Dilution % Level
1.0% fuel dilution (bottoms) in Oil A*	Oil A – 0 fuel dilution	Not detected
2.8% fuel dilution (bottoms) in Oil A	Oil A – 0 fuel dilution	2.8000
5.1% fuel dilution (bottoms) in Oil A	Oil A – 0 fuel dilution	5.0900
10.3% fuel dilution (bottoms) in Oil A	Oil A – 0 fuel dilution	10.340
2.0% fuel bottoms in fuel	Oil A – 0 fuel dilution	98.190
25.6% fuel in different oil	Oil A – 0 fuel dilution	45.760
Actual used oil, 16% fuel dilution by GC	New oil	22.110
16311 Actual oil, 10% fuel dilution, AOAP method	Auto reference	10.30
16285 Actual oil, 5% fuel dilution, AOAP method	Auto reference	7.33
16364 Actual oil, 0% fuel dilution, AOAP method	Auto reference	0.00
16311 Actual oil, 10% fuel dilution, AOAP method	Multigrade	15.92

* Oil A = Mineral oil

When oxidation is measured by FTIR and compared to differential infrared analysis (DIR), the correlation is good. These data results can be seen in TABLE 4.

TABLE 4. Oxidation

Sample	IR Absorbance/ cm Values	Nicolet Absorbance Values
Used oil, 50-hr	78.9	0.7000
Used oil, 100-hr	172.8	1.5900

The FTIR soot measurement is given in percent transmittance (% T), and acceptance levels have not been determined. When these measurements were compared to pentane and toluene insolubles and TGA, the results seemed reasonable. These data are reported in TABLE 5.

TABLE 5. Soot Content

Sample	B Pentane Insols %	B Toluene Insols %	TGA Soot %	Nicolet % T
0-hr	0	0	0	93.520
50-hr	2.88	2.56	3.53	7.1800
100-hr	6.45	5.12	6.21	0.8000

Sulfate levels were not known in any of the samples analyzed. The Nicolet FTIR reported values ranged from not detected to 1.46 absorbance units on the used oils, with values up to 1.72 for oxidation (carbonyl levels). Glycol levels also were not known on these samples, but the Nicolet FTIR results ranged from not detected to 0.25 percent. When both glycol and water were detected, a note was included in the printout report that read "antifreeze interferes with water values." The highest water value reported was 2.24 percent water and 0.93 percent glycol. When the sample was analyzed with a Karl Fisher titrator, a value of 3.3 percent was found. The sum of water and glycol reported agreed with Karl Fisher water results, but more tests would have to be done to evaluate the water and glycol measurements.

The Nicolet FTIR instrument was evaluated for use in a motor-pool type of environment where a variety of different oils are present. Lubricants and blends of varying chemical composition were analyzed against various reference oils. These data are shown in TABLE 6.

TABLE 6. Reference Oil Selection Data

				Fuel Dilution		
Sample	Reference	% T	Ox	Nicolet %	AOAP %	GC %
New Oils						
Oil A*	Oil B**	98.27	0.33	14.69	--†	--
Oil C‡	Oil B	92.15	0.21	10.44	--	--
Oil D§	Oil E♦	88.32	15.44	0	--	--
Oil B	Oil A	97.51	0	0	--	--
Oil C	Oil A	95.27	0	0	--	--
Oil C	Oil C	93.52	0	0	--	--
Oil A	Oil C	95.27	0.32	3.76	--	--
Oil B	Oil C	95.03	0.03	0	--	--
Oil A/B	Auto	93.84	0.16	0	--	--
Oil A/C	Auto	88.42	0.11	6.14	--	--
Used Oils						
16285	15689	88.27	0.05	7.33	5.0	5.0
16262	14180	15.53	0.30	11.67	0	--
16311	14180	67.98	0.14	15.92	10.0	5.1
16311	Auto (Nicolet)	85.53	0.13	10.30	10.0	5.1
16364	Auto (Nicolet)	76.72	0.15	0	0	--
16367	Auto (Nicolet)	70.71	0.14	0	0	--
15689 (50-hr)	15689 (30)	7.18	0.70	0	--	--
15689 (50-hr)	14180 (multigrade)	7.07	0.81	0	--	--
15689 (100-hr)	15689 (30)	0.80	1.59	0	--	--
15689 (100-hr)	14180 (multigrade)	1.17	1.72	0	--	--

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* Oil A = SAE 15W-40, PMA (AL-14712-L)
** Oil B = SAE 15W-40, OCP (AL-17122-L)
† Test not performed
‡ Oil C = SAE 30 (AL-15689-L)
§ Oil D = Polyolester (AL-8925-L)
♦ Oil E = 10.3% fuel dilution in mineral oil

Several potential problems of reference selection error were noted in this brief evaluation. Note that when new SAE 15W-40 polymethacrylate (PMA) VI improver oil or SAE 30 oil were analyzed using an olefin copolymer (OCP) oil as reference oil, false fuel dilution values were obtained, as well as small false oxidation values. When a new synthetic lubricant that contained polyolester was analyzed versus a mineral oil, high false oxidation results were produced. In some cases, reference oil choice can be significant. An unknown used lubricant, 16311, gave fuel dilution values differing by 5 percent when two different reference oils were selected. If a reference is available for a used oil, all results seem very acceptable. From these limited data, the FTIR appeared to have great potential in motor-pool trend analysis environments.

A more detailed evaluation of the Nicolet 8210 software package is summarized in BFLRF Interim Report No. 293, Volume II, under Section A entitled "Used Oil Analysis."

C. Recent Findings

The initial literature search was conducted to identify currently available portable kits from tests that could be conducted in the field. From this early survey, it became apparent that the only real changes in field test kits since 1979 were in the degree of sophistication, rather than new technological developments. However, recent technical literature and technical symposia indicated that new laboratory technologies were being developed for in-service used lubricant monitoring. Therefore, a new literature search was performed to identify currently available laboratory technologies employed in used lubricant quality monitoring. Four databases were searched: 1) National Technical Information Service (NTIS), 2) Compendex Plus, 3) Chemical Abstracts Service Search, and 4) Federal Research in Progress (FEDRIP) using the DIALOG Information Retrieval Service. From this literature, a total of 314 items were identified.

The literature search and personal visits identified several laboratory technologies that could possibly be used in the development of the PLQM. Other literature, technologies, and devices were also noted, but these would require a great amount of development for PLQM use. The technologies and devices that showed the most promise were evaluated as they could be obtained or procured.

The selected techniques/technologies and portable test devices were used to evaluate a wide range of new, used, stressed, and blended lubricants ranging from predominantly MIL-L-2104 and standard reference lubricant/fluids.

D. Automatic Engine Oil-Change Indicator

The first technology evaluated was the automatic engine oil-change indicator, developed by General Motors Research Laboratories.(7) The oil-change indicator is based on oil temperature and vehicle mileage or engine revolutions applied to a mathematical model of oil aging. The indicator gave good correlation with oil analyses, assuming that conditions remain the same. The system does not, however, directly determine oil properties. Therefore, the oil-change indicator cannot detect engine oils with the wrong quality or viscosity, nor can it detect engine malfunctions, such as antifreeze leaks into the oil. The oil-change indicator could not account for unusual conditions, such as excessively dusty environments, the use of poor quality fuels, etc. As a result of these observations, it was determined that this system was not a prime candidate for Army use and development for the PLQM.

E. Assessment of Remaining Lubricant Life

The second technology evaluated was the assessment of remaining lubricant life (8-10) for aircraft turbine engine oils using reductive-cyclic voltammetry. This work, conducted by the University of Dayton Research Institute, was funded by the U.S. Air Force Wright Aeronautical Laboratories. The following techniques were evaluated: oxidative and reductive cyclic voltammetry, thermal stressing, chemical stressing, titration, electrochemistry, and spectrophotometry. Of the remaining lubricant life assessment test (RLLAT) candidates noted during the investigation, the reductive-cyclic voltammetric (RCV) technique was the least expensive, easiest to operate, required the shortest analytical time, and produced the most accurate and precise remaining lubricant life assessments. A RLLAT based on RCV technique was developed that was capable of accurately assessing the remaining lubricant life of MIL-L-7808 oils and has potential for use by the Air Force. This technique determined the concentration of the generated antioxidant species (aromatic amines) and the potential of the oil to generate new antioxidant species in the scan from +1.0 to 0.0 V.

The primary antioxidant used in Army gasoline and diesel engine oils is zinc dithiophosphate (ZDP), which also serves as an antiwear additive. The cyclic voltammetry (CV) method enables a wide potential range to be rapidly scanned for reducible or oxidizable species. This capacity, together with its variable time scale and good sensitivity, makes the CV method the most versatile electrochemical technique thus far evaluated.

F. Electrochemical Reactions/Cyclic Voltammetry

The third technology, discussed in a paper entitled "The Nature of Electrochemical Reactions Between Several Zinc Organodithiophosphate Antiwear Additives and Cast Iron Surfaces,"(11) has produced results with CV that imply electrochemical reactions between the decomposition products of ZDP, and the electrode surfaces produce surface coatings on cast iron electrodes. The effects of oxygen, oleic acid concentration, and surface coating on the electrochemical reaction were measured. CV has become increasingly popular in all fields of chemistry as a means for studying redox states. Based upon this information, BFLRF pursued the use of cyclic voltammetry as a technology for possible PLQM development.

CV enables a wide potential range to be rapidly scanned for reducible or oxidizable species. This capability, together with its variable time scale and good sensitivity, makes CV the most versatile electrochemical technique thus far developed. It must, however, be emphasized that its strengths are still largely in the realm of qualitative or diagnostic experiments.

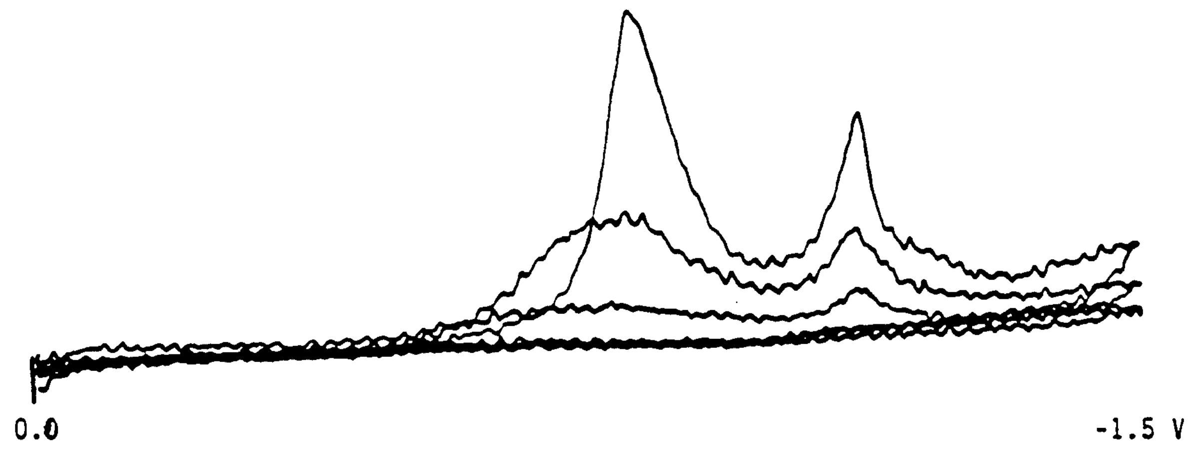
Initial efforts were made to determine if alkyl or aryl ZDP in new unused lubricating oils can be characterized by cyclic voltammetric measurements. The electrochemical reactivities of these types of antioxidant/antiwear additives in stressed mineral oil (oil subjected to thermal decomposition) have been studied; however, it was unknown if the ZDP additives would undergo redox reactions in fresh oil.

The electrochemical cell configurations used in the experiments consisted of either a glassy carbon or platinum working electrode against a Ag/AgCl reference electrode. The electrolyte was 0.05 M lithium per chlorate in an ether solution, and the sample was diluted with this electrolyte at a 1:50 ratio. The maximum and minimum scanned potentials applied were 4 to –4 V. The cyclic voltammagrams, which are plots of induced current versus applied potential, display no oxidative or reductive waves. The observations indicate that the ZDP itself does not undergo redox reactions at these conditions.

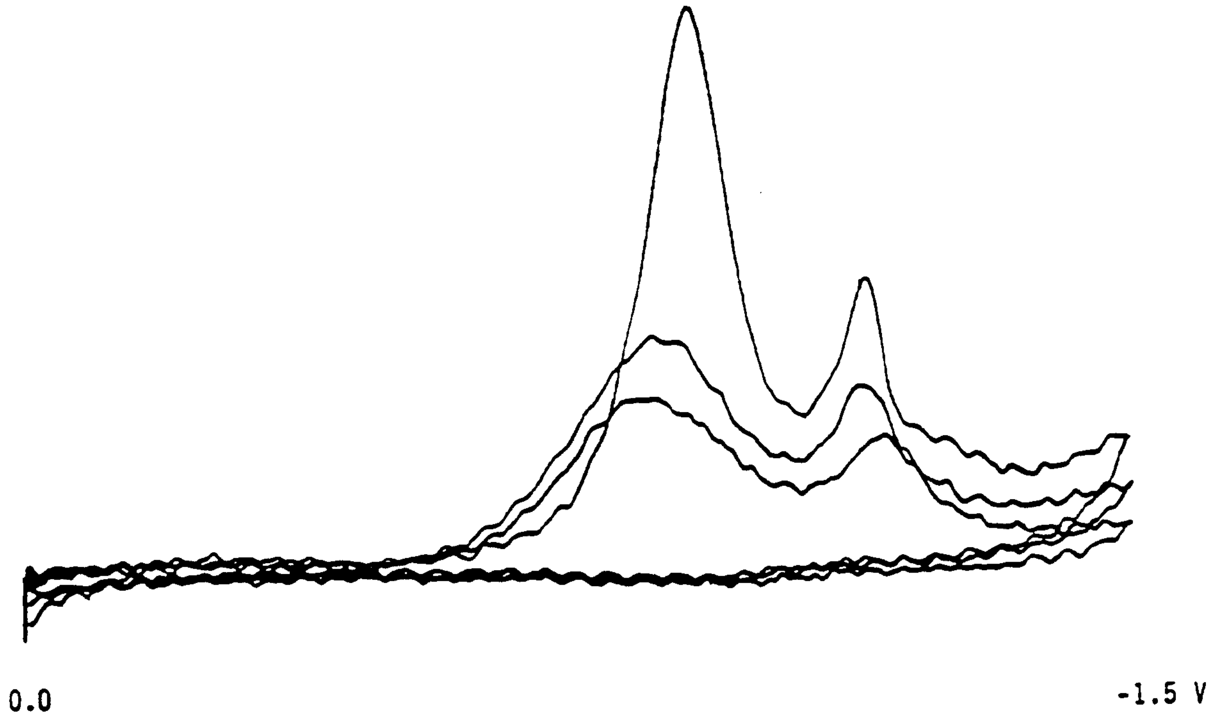
The induced current observed in previous studies could be from the decomposition products generated from ZDP and the iron metal electrode surface. BFLRF measurements of electrochemical impedance of the 0.1-percent ZDP sample indicated an increase in the cell capacitance when compared to the lubricant containing no ZDP. These data suggest the presence of a film plated on the surface of the electrode, or perhaps adsorption of ZDP.

The next cyclic voltammetric measurements were made using a mercury and gold electrode. This type of electrode is commonly used for the oxidation and reduction of sulfur-type compounds. CV measurements were performed with a Hg/Au working electrode, platinum counter electrode, and a Ag/AgCl reference electrode. Lubricant samples (0.20 mL aliquot) were combined with supporting electrolyte (9.8 mL of 0.1 M $LiClO_4$ in acetone), vigorously mixed, and filtered through 5 M Millex-LS filter media. The lubricant samples tested were thermally stressed to their breakpoint and half of the breakpoint time using the ASTM D 2272 Rotating Bomb Oxidation Stability Test. These tests were conducted in order to investigate characteristics of ZDP decomposition products, which should be responsible for the antioxidant and antiwear properties.

Continuous cathodic potential sweeps ranging from 0 to –1.5 V versus Ag/AgCl at 400 mV/s resulted in the appearance of adsorption waves for both stressed and unstressed samples (Fig. 3). Thus, it does not appear that differentiation was possible with respect to stress time under these conditions. However, continuous anodic sweeps from 0 to +1.5 V resulted in the formation of a diffusion-controlled wave at approximately 0.7 V (Fig. 4) for the two stressed samples tested

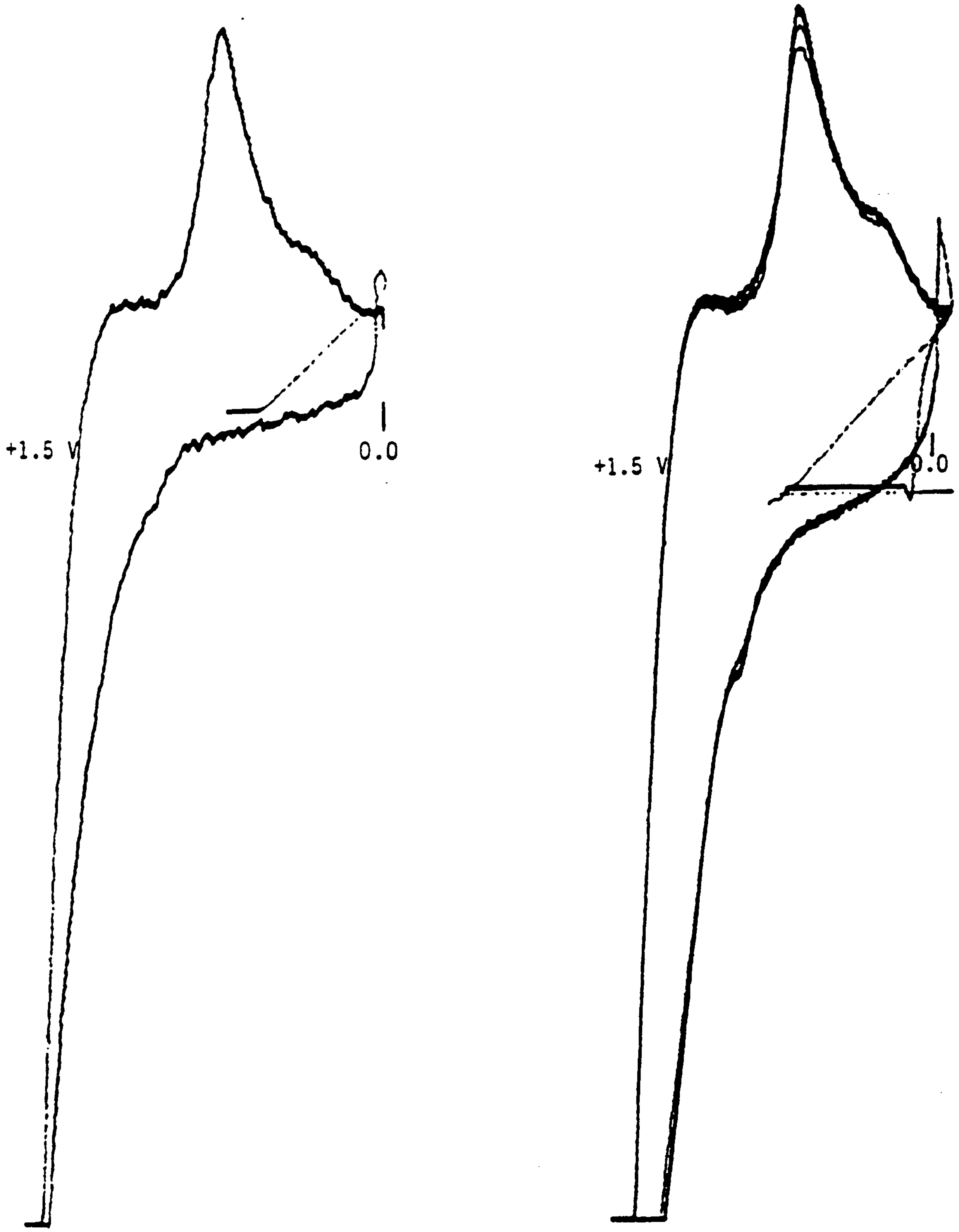


a. 38-minute sample



b. Unstressed sample

Figure 3. Cathodic potential adsorption waves



a. 38-minute sample

b. 105-minute sample

Figure 4. Anodic potential diffusion waves

(aryl 38 minutes and alkyl 105 minutes). A greater current output was observed with the alkyl 105-minute one-half stressed time sample than the aryl 38-minute one-half stressed time sample. As expected, these results suggest that the concentration of the ZDP decomposition products increases with thermal stress time. These initial results were confirmed by repeating these experiments for stressed and unstressed samples listed in TABLE 7.

TABLE 7. Stressed Lubricant Samples

Stress	Base Stock	Aryl, 0.1%	Alkyl, 1.0%
Breakpoint, min.	40	65	210
One-half breakpoint, min.	20	38	105

When conducting the CV measurements with samples containing aryl (AL-6184-A) ZDP, the current output did not increase with stress time, as one might expect if the concentration of ZDP-decomposition products increases with thermal stress time. Conversely, the current output increased with stress time for the samples containing alkyl (AL-6185-A) ZDP.

New 0.1-percent aryl-ZDP in a base lubricant was thermally stressed to its breakpoint and half the breakpoint time.

Stressed and unstressed lubricant samples (1 mL aliquots) were combined with 9 mL of the supporting electrolyte 0.1 M $LiClO_4$ in ethylacetate. The electrochemical cell consisted of an Hg/Au working electrode, platinum counter electrode, and an Ag/AgCl reference electrode.

Lubricant samples were analyzed by two CV techniques: 1) cathodic stripping voltammetry (CSV), and 2) continuous anodic scans. The results of these techniques are shown in Figs. 5 through 12. CSV (bottom scans of Figs. 6 through 10) involves preconcentration by oxidation with a subsequent cathodic scan to strip the material from the surface of the electrode. As indicated by the flatness of each scan (bottom) in Figs. 6 through 10, this technique did not afford a preconcentration effect of aryl- or alkyl-ZDP-decomposition products onto the surface of the electrode.

Thus, reduction waves were not observed for any of the stressed samples analyzed, since a negative potential scan (+0.5 → −1.5 V) was applied to the electrode, which otherwise may have indicated the deposition of reducible ZDP-decomposition products.

Continuous anodic scans (top scan of each figure) of samples containing either aryl- or alkyl-ZDP resulted in the appearance of a reduction wave at +0.6 V. This reduction wave was observed neither for the unstressed sample containing 0.1 percent aryl-ZDP AL-6184-A (Fig. 5) nor for the blank lubricant sample, which was stressed for 210 minutes (Fig. 11, top scan), suggesting that the wave is attributed to the reduction of a ZDP-decomposition product. For samples containing aryl-ZDP AL-6184-A, the current output does not continue to increase with stress time, as one might expect if the concentration of ZDP-decomposition products increases with thermal stress time. Conversely, the current output increases with stress time for samples containing alkyl-ZDP (AL-6185-A). This work produced current output increases with stress times with samples containing 0.1 percent aryl- and alkyl-ZDP, but there were problems with repeatability. This poor repeatability appeared to be caused by either of three factors.

The first factor is the **electrochemical cell**, which consists of the Hg/Au working electrode, platinum counter electrode, and the Ag/AgCl reference electrode. The cell has had problems with the Hg/Au plating of the electrode. Therefore, BFLRF is conducting new experiments with a cell using a glassy carbon working electrode, platinum counter electrode, and an Ag/AgCl reference electrode. The second factor is the use of a **solid substrate** that will be used when stressed or used lubricant samples are analyzed, since such samples also contain other oxidation products that could interface with the evaluation response unless removed from the solution by the substrate AC impedance. The third factor is use of a **slower scan rate** to allow for more complete decomposition.

For the next tests, a different electrochemical cell configuration was used while increasing the potential scanning range at slower rates.

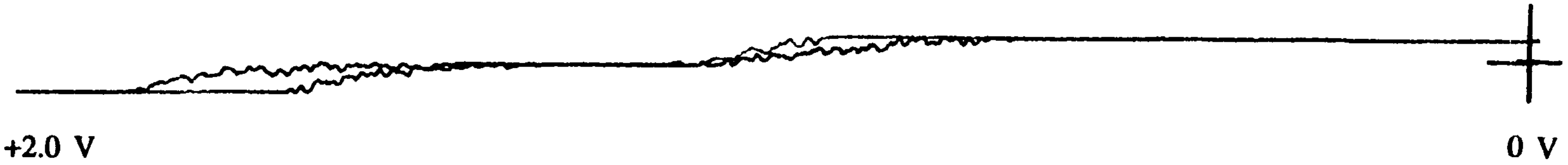
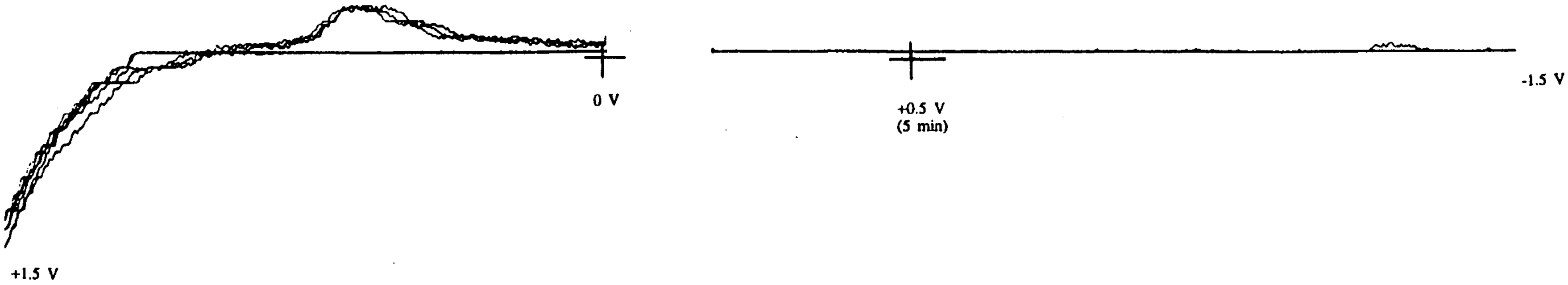


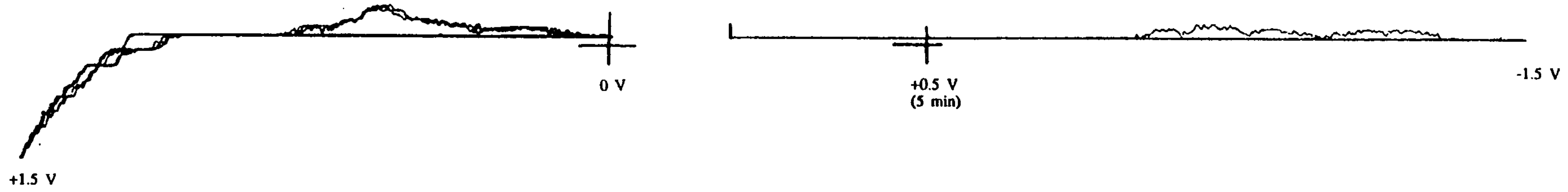
Figure 5. CV of unstressed AL-8881-L containing 0.1% AL-6184-A



a. Continuous anodic scans

b. Cathodic stripping

Figure 6. CV of AL-8881-L containing 0.1% AL-6184-A, stressed for 38 minutes



a. Continuous anodic scans

b. Cathodic stripping

Figure 7. <u>CV of AL-8881-L containing 0.1% AL-6184-A, stressed for 65 minutes</u>

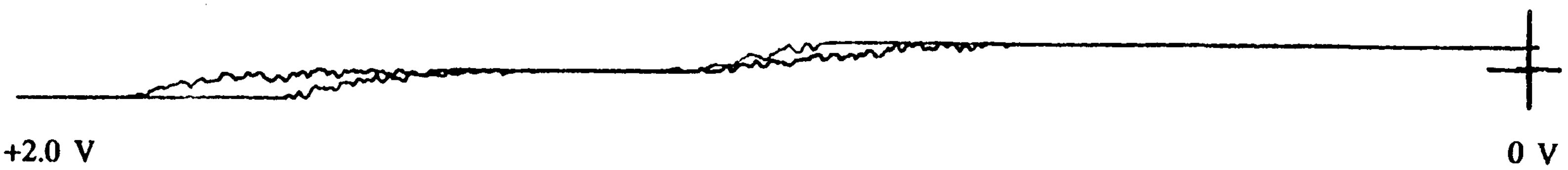
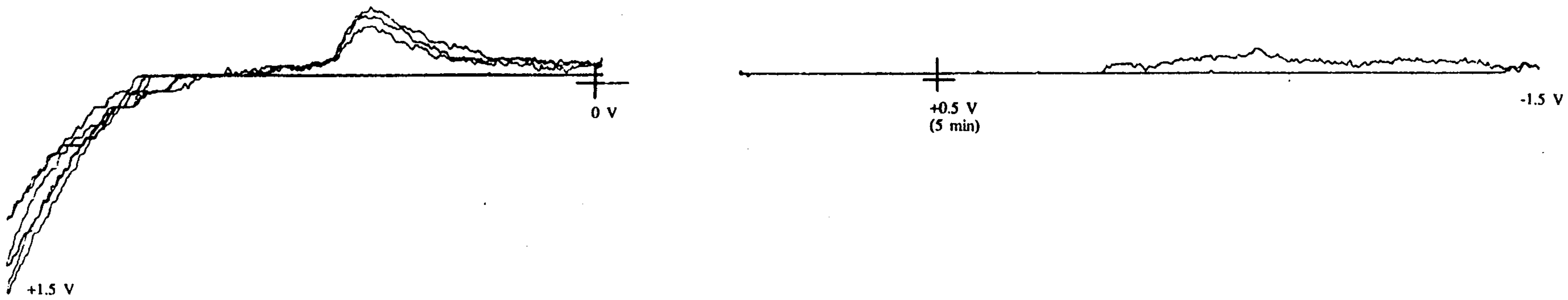


Figure 8. <u>CV of unstressed AL-8881-L containing 0.1% AL-6185-A</u>

a. Continuous anodic scans

b. Cathodic stripping

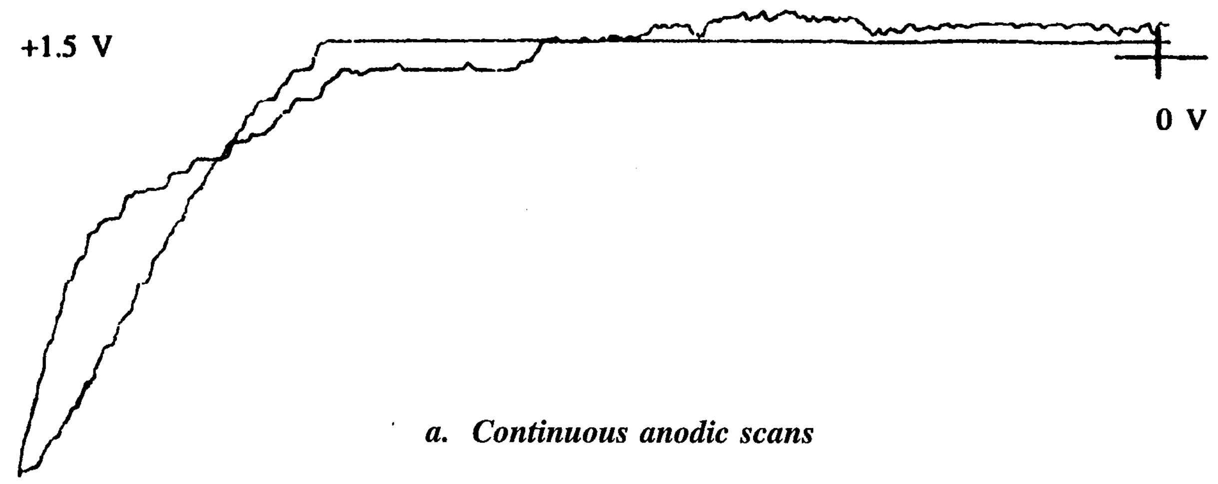
Figure 9. CV of AL-8881-L containing 0.1% AL-6185-A, stressed for 105 minutes



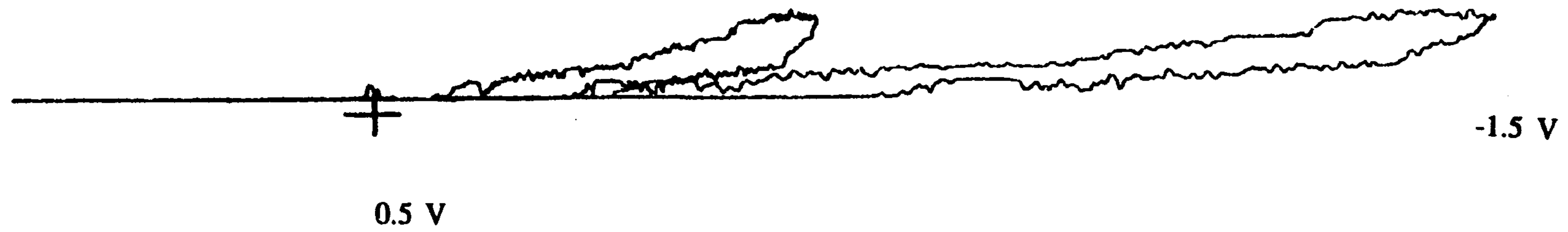
a. Continuous anodic scans

b. Cathodic stripping

Figure 10. CV of AL-8881-L containing 0.1% AL-6185-A, stressed for 210 minutes

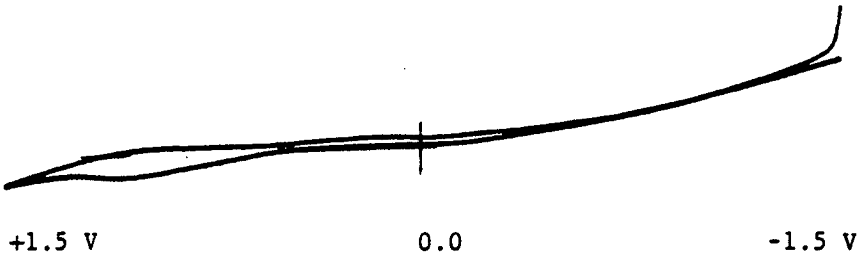


a. Continuous anodic scans

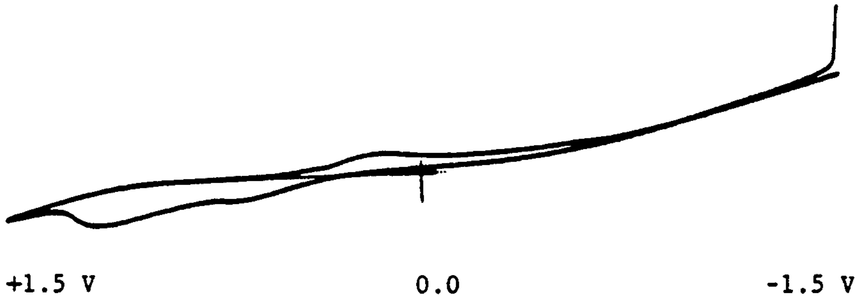


b. Cathodic stripping

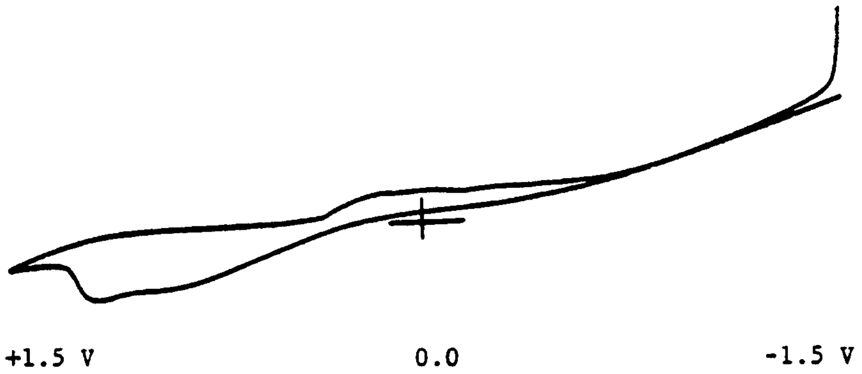
Figure 11. <u>CV of AL-8881-L, stressed for 210 minutes</u>



a. 28-minute stress



b. 36-minute stress



c. 58-minute stress

Figure 12. **<u>CV of AL-8881-L containing 0.1% AL-6184-A</u>**

Lubricant samples containing 0.1-percent aryl- and alkyl-ZDP were thermally stressed to their additive depletion breakpoint and one-fourth, one-half, and three-fourths of the breaktime. These stressed lubricant samples (1-mL aliquots) were combined with 9 mL of the supporting electrolyte 0.1 M $LiClO_4$ in ethylacetate and filtered through 0.5-μm filter media. The electromechanical cell was modified by removing the Hg/Au working electrode and replacing it with a glassy carbon working electrode (this electrode needs no plating). The platinum counter electrode and the Ag/AgCl reference electrode remained the same. Lubricant samples were analyzed by increasing the scanning potential range from –1.5 to +1.5 V versus Ag/AgCl at a slower rate of 10 mV/s. The results from these measurements were repeatable and quite good, and representative results can be seen in Fig. 12. As indicated in the figure, the anodic scans (–1.5 V → +1.5 V) resulted in two distinct oxidation waves at +0.7 and +0.9 V that increased in current with thermal stress. Additionally, small reduction waves occurred during the cathodic scans (+1.5 V → –1.5 V) at +0.05 V, whose current also increased with thermal stress time. These initial redox results are very promising because this technique appears to be measuring the additive depletion through the decomposition products.

G. AC Impedance Measurements

The principle of electrochemical impedance measurements relies on the fact that an electrochemical cell can be represented by a purely electronic model consisting of resistor and capacitor electronic elements. The instrumentation applies alternating excitation waveforms ranging from 100 KHz to 10 Hz to the electrochemical cell (electronic model) and analyzes the response. Vector analysis (impedance vector) of the resulting AC waveform provides a description of the electrochemical system in terms of its equivalent circuit.

The purpose of the impedance measurements presented in this report was to determine the value of the capacitance element in a simplified equivalent circuit of the stressed and unstressed lubricant sample using a steel working electrode. The capacitance values extracted from the response waveforms are graphically presented in Fig. 13.

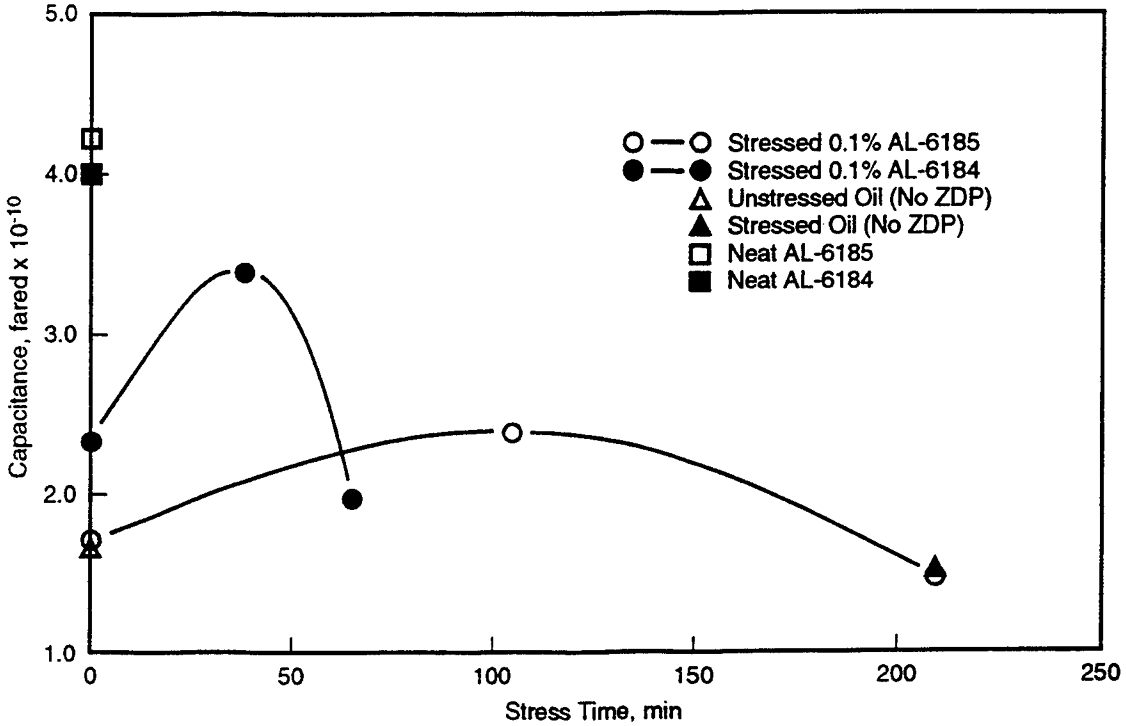


Figure 13. Capacitance values at a steel electrode versus thermal stress time

The results indicate that the capacitance at the steel electrode for samples containing alkyl-ZDP increases with thermal stress time up to 105 minutes. Beyond 105 minutes, the capacitance decreases to the control endpoint (i.e., the capacitance value at 210 minutes is equivalent to that of the base oil containing no ZDP). Similarly, the capacitance value at zero stress time is equivalent to the control startpoint (capacitance of unstressed oil containing no ZDP).

The capacitance values at the steel electrode for samples containing aryl-ZDP behaved in a similar fashion as that observed for alkyl-ZDP (i.e., the capacitance in response to thermal stress time reached a maximum value, then decreased to a value below that of the unstressed sample). Since a control endpoint was not obtained, it cannot be determined if the capacitance value of the aryl-ZDP sample at 65 minutes converges to the value of the control.

These data seem to suggest that 1) at zero stress time, there is virtually no association of alkyl- or aryl-ZDP to the steel electrode; 2) there is a maximum propensity for the association of alkyl- and aryl-ZDP decomposition products to the steel electrode with regard to thermal stress time;

and 3) the association of aryl-ZDP decomposition products to the steel electrode is considerably greater than that determined for samples containing alkyl-ZDP.

H. Blotter Spot Test by Laser Fluorescence Technique

The oil blotter spot test has been used to evaluate the condition of used engine oils. However, this method is quite visually subjective. Efforts to automate these readings included a laser to evaluate the blotter spots. Used oils from a 6.2-L engine high-temperature test were prepared for testing. The oil is AL-14180-L, grade SAE 15W-40. The selected used oil samples were taken at 0, 20, 50, and 182 hours. Then two portions were taken from the "as is" samples. One portion was blended with 1 wt% water and the other with 1 wt% antifreeze. Samples of the twelve portions were deposited on a blotter spot card and, after dispersion, were analyzed with a laser fluorescence technique. Initial work has not been promising because the results could not be correlated to ASTM tests.

I. Complete Oil Breakdown Rate Analyzer and Dielectric Constant

The complete oil breakdown rate analyzer (COBRA) was reported to measure the electrochemical activity increases due to breakdown of the ester base oil and antioxidant additive used in turbine engine oils. The USAF recommended the continued development of the COBRA since the instrument displays potential as a rate oil-change quality indicator for conventional MIL-L-7808 and MIL-L-23699 lubricants. The COBRA was used to evaluate MIL-L-2104 lubricants.

The COBRA and dielectric constant (operational discussion, page 12) devices were evaluated using lubricants stressed with a modified FTM-5307 Corrosiveness and Oxidation Stability of Engine Lubricants and the ASTM D 2272 Rotating Bomb Oxidation Tests (RBOT). Typical results are shown with a MIL-L-2104 grade SAE 40, AL-19092-L lubricant stressed with the modified FTM-5307 test for 96 hours. The test results from the collected used oil samples are shown in TABLE 8 and in Fig. 14. The dielectric constant device had good correlation by

TABLE 8. Results of Test Devices and ASTM Tests on Modified FTM-5307 Tested Lubricant

Test Hours	Viscosity at 100°C, cSt	Viscosity, % increase	TAN	DIR		Dielectric Constant Device	COBRA
				Oxidation	Nitration		
0	14.45	--	2.61	--	--	0	12.0
16	14.76	2.15	3.74	19.6	9.2	1.2	4.5
24	15.79	9.27	4.27	27.2	8.0	2.0	5.0
40	16.56	14.60	4.93	54.4	5.2	2.7	7.5
48	16.20	12.11	5.10	60.8	6.4	3.0	9.0
64	17.77	20.83	5.95	77.6	6.4	3.2	9.5
72	18.65	29.07	6.11	84.8	3.6	3.5	9.0
88	22.34	54.60	8.15	98.0	10.4	4.0	6.5
96	26.97	86.64	9.35	117.2	10.8	4.3	4.0

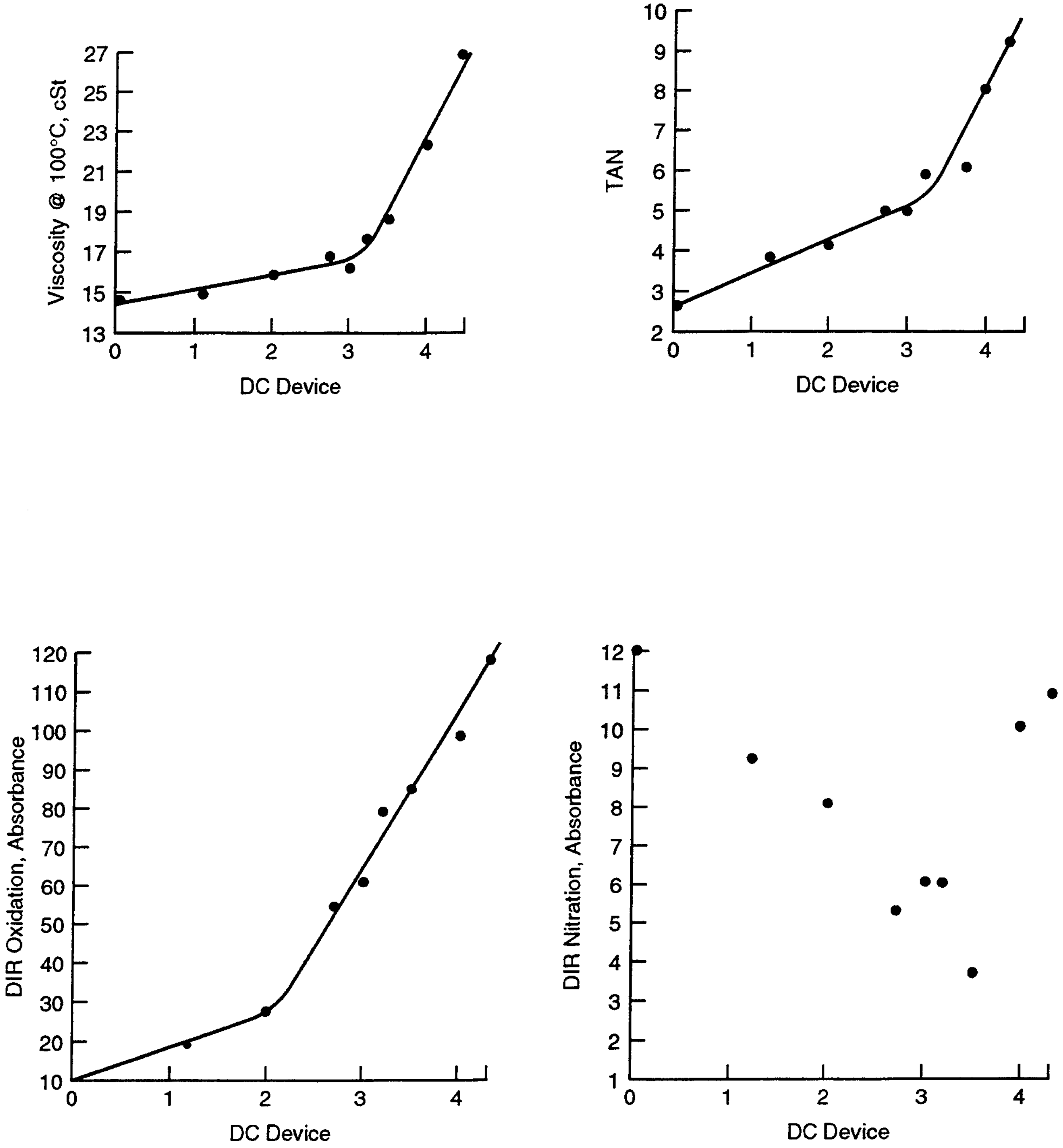


Figure 14. Dielectric constant device results

showing the break with the viscosity at 100°C, TAN, and DIR oxidation results. However, the DIR nitration results did not show a correlation. As Fig. 15 illustrates, the COBRA did not show good correlation.

Previous work with the cyclic voltammetry device produced promising results with two lubricants stressed with the RBOT. These two lubricants were laboratory blended using a virgin basestock with 0.1-percent AL-6184-A (aryl-zinc dithiophosphate) in one lubricant and 0.1-percent AL-6185-A (alkyl-zinc dithiophosphate) in the other. The COBRA and dielectric constant devices were also used to evaluate these two RBOT-stressed lubricants, and their results are shown in TABLE 9. Both test devices registered maximum plus on their meters, indicating lubricant failure. These readings would be acceptable for the 67- and 58-minute samples stressed to breakpoint, but the 36- and 28-minute samples were stressed to only one-half the breaktime, and these samples also indicated maximum plus meter readings. This high reading may have been caused by the approximately 9 percent water added to the lubricant during stressing. The COBRA did not have good correlations with the standard test methods, so no more evaluations were conducted. Additional work was conducted with the dielectric constant device to determine if the lubricant failure was caused by the water or by the other oxidation contaminants. BFLRF had a fully formulated SAE 10W-30, AL-18576-L, lubricant sample that had also been stressed with the RBOT test. This sample stabilized at 660 minutes and had not broken at 960 minutes. This lubricant had water content data on the used samples. The test results can be seen in TABLE 10. The 300-minute sample with 8.09 percent water pegged the dielectric constant meter at 12+, while the 960-minute sample with only 0.35 percent water content had a reading of 6.2. These samples were poured over a desiccant and left to absorb the moisture in the used samples. The dielectric constant readings from both samples were lowered. The 300-minute sample apparently contained too much water for it to be totally removed. Most of the water was removed from the 960-minute sample, which recorded a 2.7-meter reading. The 960-minute sample had not been stressed to the breakpoint. Therefore, with the water removed from the lubricant, insufficient oxidation contaminants were present in the lubricant for it to be failed. This method with the dielectric constant device appears to indicate that the failure of a lubricant can be determined regardless of its source of contamination.

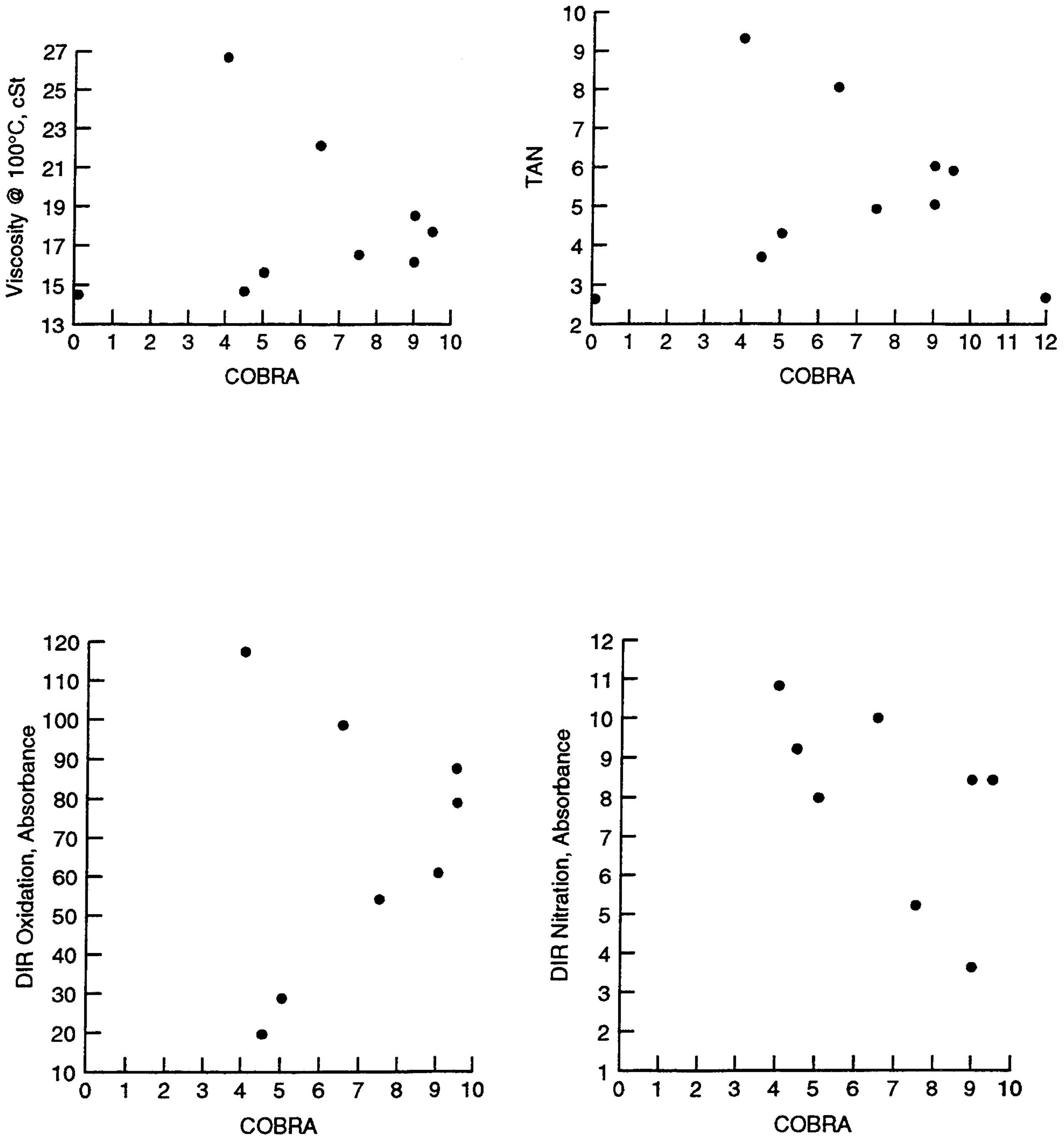


Figure 15. **COBRA device results**

TABLE 9. Lubricants Stressed in RBOT

Stress Time, min.	COBRA	Dielectric Constant Device
AL-8881-L + 0.1-Percent AL-6184-A (Aryl-Zinc Dithiophosphate)		
0	0	0
36 – 1/2 Breaktime	100 + (Pegged)	12 + (Pegged)
67 – Breakpoint	100 + (Pegged)	12 + (Pegged)
AL-8881-L + 0.1-Percent AL-6185-A (Alkyl-Zinc Dithiophosphate)		
0	0	0
28 – 1/2 Breaktime	100 + (Pegged)	12 + (Pegged)
58 – Breakpoint	100 + (Pegged)	12 + (Pegged)

TABLE 10. AL-18576-L, SAE Grade 10W-30, Stressed in RBOT

Stress Time, min.	% H_2O	Dielectric Constant Device	
		Untreated	Desiccant Treated
0	ND*	0	0
300	8.09	12 (Estimated 20)	12 (Estimated 15)
960 (Stabilized)	0.35	6.2	2.7

ND* = Not Determined

J. Wet-Friction Tests

The MIL-L-2104 lubricants include frictional requirements in the lube specification. In addition, problems existed in the field, with some commercial construction and combat/tactical equipment transmission frictional materials. It appeared a bench test device to measure wet-frictional characteristics was necessary for the PLQM.

In an effort to develop a bench test to evaluate lubricant effects on wet-friction brakes and clutches, initial tests were performed with the Ball-on-Cylinder wear test apparatus with reference oils. The Ball-on-Cylinder apparatus produces lightly loaded, unidirectional, high-speed sliding, similar to that in typical powershift transmissions. These early tests were performed using a brass ball on a textured steel ring with variations in applied load, sliding speed, temperature, and test duration. Each variable significantly affected the friction and wear characteristics of the lubricant. In each instance, however, material removal (wear) was characterized by an abrasive mechanism that precluded buildup of additive films and glazing of the surfaces.

Subsequently, the test ring was polished to a mirror finish, which prevented abrasion and significantly reduced wear. The temperature of the oil reservoir was set to 80°C, while the sliding speed was set to 600 rpm at an applied load of 1,900 grams. These conditions were set to simulate the Caterpillar TO-4 bronze friction retention test. The increased temperature reduces hydrodynamic lift, but promotes reaction of the lubricant additives. As shown in Fig. 16, the initial results for friction retention with the Caterpillar TO-4 pass and fail reference oils were encouraging and showed good separation. These test results reflect those results from the TO-4 test series, as shown in Fig. 17. Subsequent tests were conducted using the Allison C-4 friction test pass and fail lubricants. However, both lubricants produced unacceptably low friction when compared with TO-4 reference oil results. Additional brief tests were performed using the Cameron-Plint wear test apparatus with specimens machined from actual friction discs. However, in its present configuration, this apparatus produces a highly loaded reciprocating contact and requires further modification to effectively model wet-friction retention.

Simultaneously, work was also performed on stick-slip (wet-brake or clutch chatter) friction with multipurpose hydraulic or power transmission lubricants, using the Ball-on-Cylinder test apparatus. The initial work was conducted using the John Deere J20A reference oil, John Deere Hy-Gard factory fill oil, MIL-L-2104 SAE 15W-40, and the MIL-L-46167 SAE 0W-20 arctic lubricant.

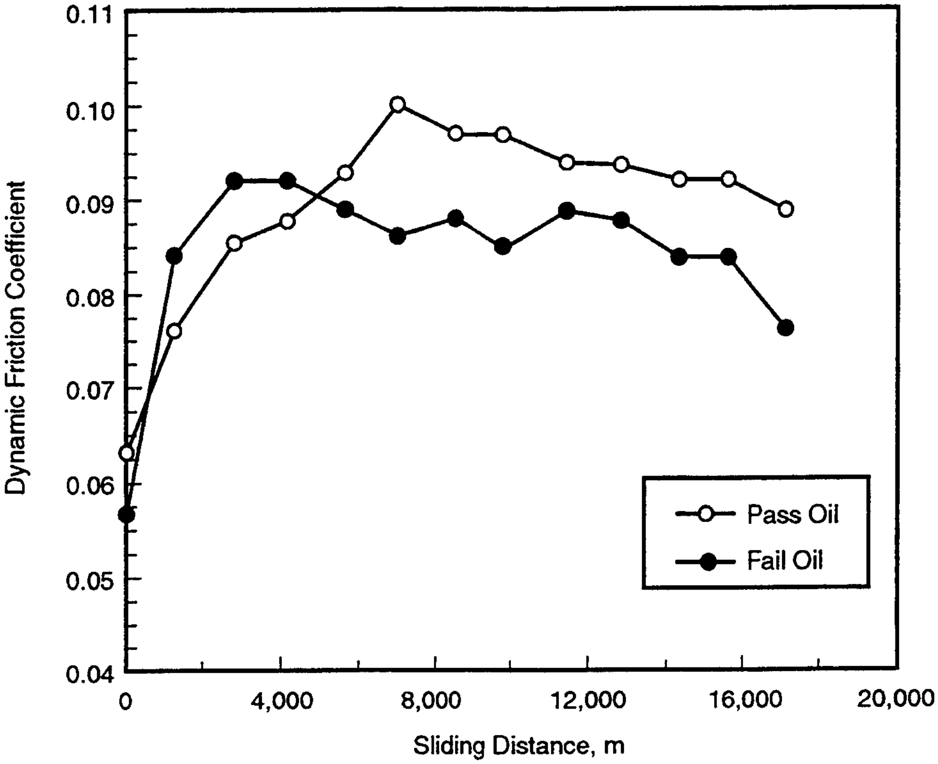


Figure 16. Initial results from BFLRF bench test for friction retention of Caterpillar TO-4 reference oils

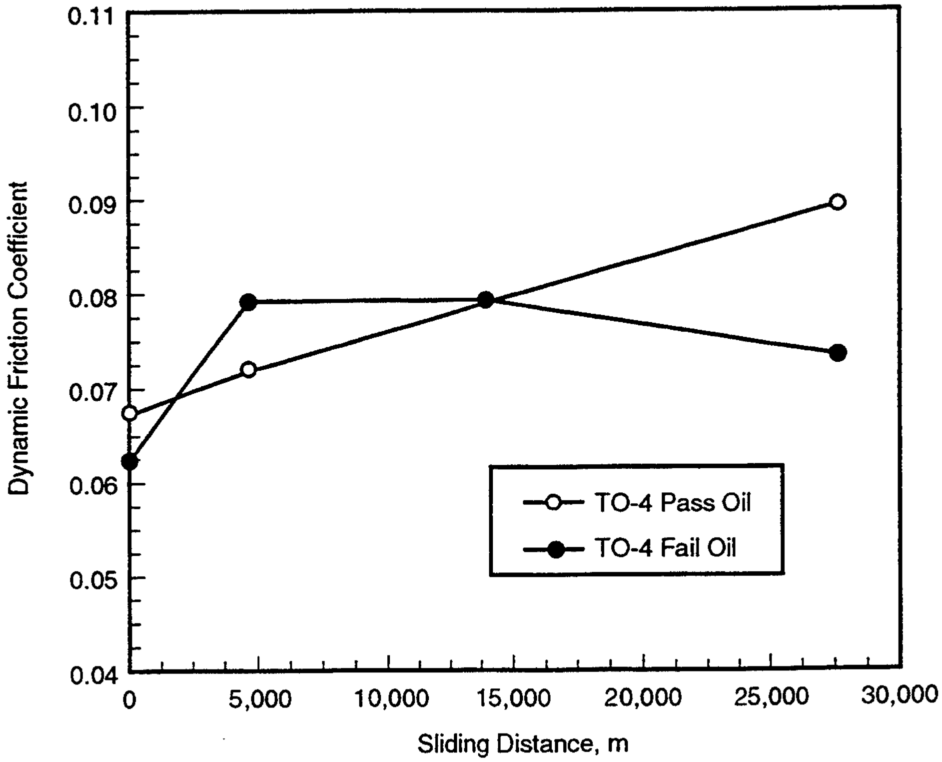


Figure 17. Results obtained in Caterpillar TO-4 test with pass and fail reference oils

A preliminary test series was performed to evaluate a range of operating conditions and metallurgies. These preliminary tests, performed using a segment of bronze clutch disc, showed some promise. However, most tests used a sintered bronze ball sliding on polished steel cylinders and a flexible drive system to simulate the potential energy buildup and dissipation during the stick-slip process. The friction force produced during both the stick and slip phases is electronically recorded. The maximum static friction force (F_S) is measured as the torsion on the flexible drive system increases. The minimum friction force (F_D) is that force remaining after the slip cycle is completed and reflects the dynamic coefficient of friction. The BFLRF stick-slip coefficient reflects the difference between F_S and F_D normalized by the applied load. An average value for both F_S and F_D is calculated over many stick-slip cycles, at applied loads of 500, 1,000, 1,500, and 2,000 grams at 10 rpm. The average normalized stick-slip coefficient for each oil is then calculated over the complete load range.

Results obtained at 100°C using this procedure are provided in Fig. 18. As shown in the figure, the J20A reference oil is ranked as the best lubricant, followed in order by Hy-Gard, MIL-L-2104 SAE 15W-40, and MIL-L-46167 SAE 0W-20. Test results at 50°C are also shown in Fig. 18. This lower temperature produces less chatter than does the 100°C temperature, since the test lubricant has an increased viscosity at lower temperatures.

The J20A and Hy-Gard friction-modified lubricants have less chatter than the MIL-L-2104D and MIL-L-46167 lubricants. However, the J20A lubricants have greater torque difference, a lower minimum torque in the Allison C-3 test, and a higher percent torque change in the TO-2 test than the military specification engine lubricants (TABLE 11).

The Caterpillar TO-4 pass and fail reference oils, the Allison C-4 pass and fail reference oils, the John Deere Quatrol™, and the ASTM TF-8 and J20A reference lubricants were also evaluated in the BFLRF stick-slip procedure, with the results shown in Fig. 19. As expected, the three friction-modified lubricants (J20A Reference, Quatrol™, and TF-8 Reference) had less stick-slip (chatter) than did the TO-4 and C-4 reference lubricants. Moreover, it seems logical that the TO-4 lubricants, which are commonly used with sintered bronze materials and designed primarily

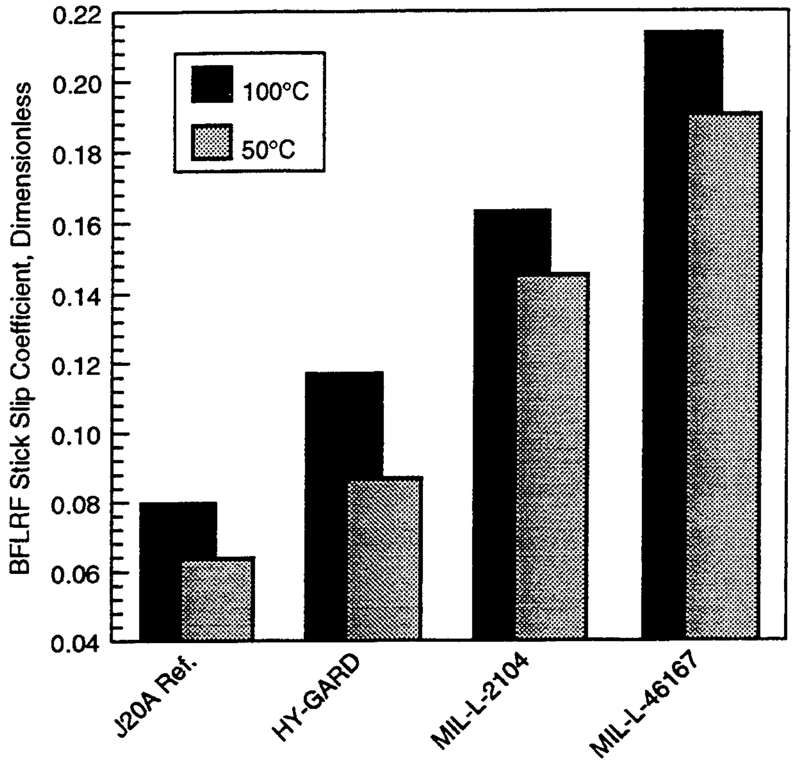


Figure 18. Initial results from BFLRF stick-slip test

TABLE 11. C-3 Friction Retention and TO-2 Friction Test Results

C-3 Friction Retention (Graphitic)	J20A Reference Oil	MIL-L-2104 SAE 15W-40	MIL-L-46167 SAE 0W-20
Maximum slip time at 5,500 cycles, 0.85 sec.	0.99	0.86	0.89
Minimum torque at 5,500 cycles, 75 ft-lb	55	83	73
Torque difference, 1,500 to 5,500 cycles, 30 ft-lb	49	12	25
TO-2 Friction Test (Bronze)			
Stopping time increase, % (15 max.)	25	19.2	23.0

for heavy-duty powershift transmissions, would be better than the C-4 lubricants in a bench test with bronze materials. Both C-4 oils are commonly used with graphite and paper clutch plate materials and are designed for both engine and powershift transmissions. In each instance, the fail lubricants were consistently worse than their respective pass lubricants.

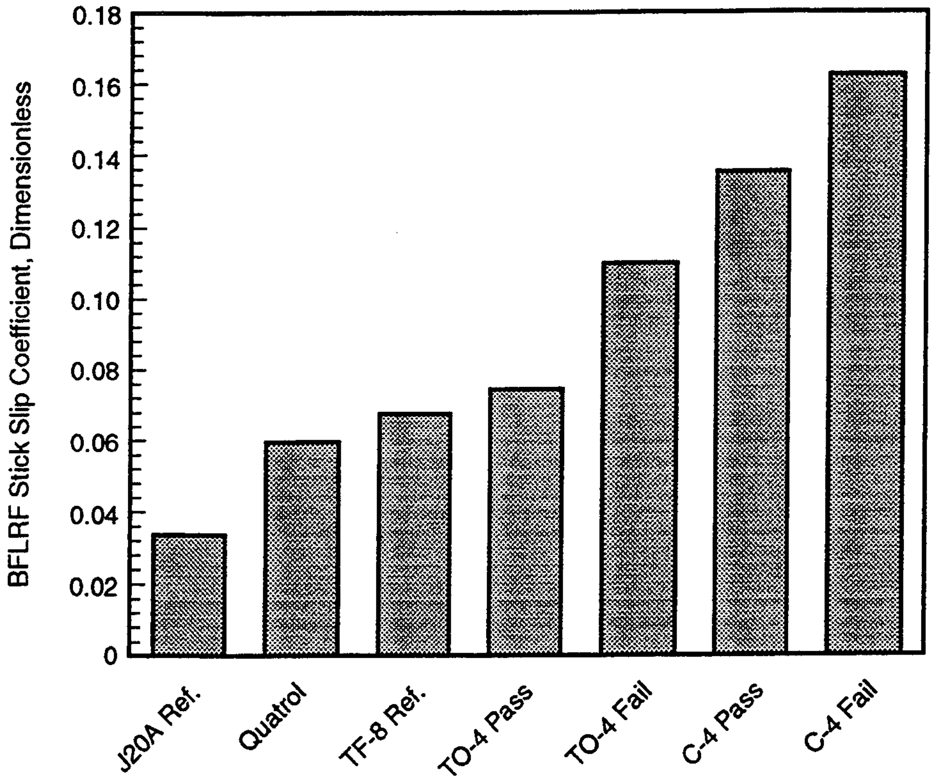


Figure 19. <u>**BFLRF stick-slip coefficient obtained with various oils**</u>

The ASTM/CEC Sintered Bronze Wet-Brake Fluid Friction test had been conducted on five military specification lubricants.(12) Since most of the brakes and clutch friction material used in combat or tactical power transmissions use bronze material, it was necessary to correlate the results of the BFLRF Bronze Stick-Slip tests to those reported in BFLRF No. 203.(12) Five lubricants, including SAE grade 10W, SAE grade 30, two SAE grade 15W-40, and one SAE grade 0W-20, along with the TF-8 reference oil were evaluated. Upon conducting the reference run with J20A reference fluid, it was learned that the Ball-on-Cylinder apparatus had a bad bearing, which amplified the chatter results. The bearing was replaced, and the tests were repeated. These repeat tests showed the lubricants relative ranking to be the same as the earlier tests, with less stick-slip (chatter) being noted. The five lubricants and the ASTM TF-8 reference oil were tested in duplicate, and the average results can be seen in TABLE 12. When compared with the ASTM Sintered Bronze Wet-Brake Chatter test, the results produced a good correlation, as shown in Fig. 20. These initial data indicate the BFLRF Bronze Stick-Slip test has good potential for the LQA System use.

TABLE 12. BFLRF Stick-Slip and ASTM Sintered Bronze Wet-Brake Chatter Test Results

Lubricant	BFLRF Stick-Slip Results, units		ASTM/CEC Wet-Brake Chatter, units
	Bad Bearings	Replaced Bearings	
J20A Reference	0.79	0.034	---*
Quatrol™	0.132	0.060	---
TO-4 Pass	0.122	0.075	---
TO-4 Fail	0.152	0.111	---
C-4 Pass	0.183	0.136	---
C-4 Fail	0.207	0.163	---
ASTM TF-8 Reference	---	0.068	64.5 ± 16.7
AL-15360-L, SAE Grade 30	---	0.100	99.7
AL-13525-L, SAE Grade 15W-40	0.163	0.118	127.6
AL-14081-L, SAE Grade 10W	---	0.120	127.7
AL-13523-L, SAE Grade 15W-40	---	0.138	157.2
AL-13632-L, SAE Grade 0W-20	0.213	0.150	139.7

---* = Tests not performed

Continued development of the Ball-on-Cylinder Stick-Slip and Friction Retention tests will be of value for use in the LQA System but also can serve industry as a screening device for both wet-brake chatter and friction retention using various lubricant additives and friction materials. This process would be more economical and would yield faster results than the currently used full-scale Wet-Brake Chatter and Friction Retention tests. Also, this apparatus is used to evaluate fuel lubricity, so it could play a multifunctional role in the PQA System.

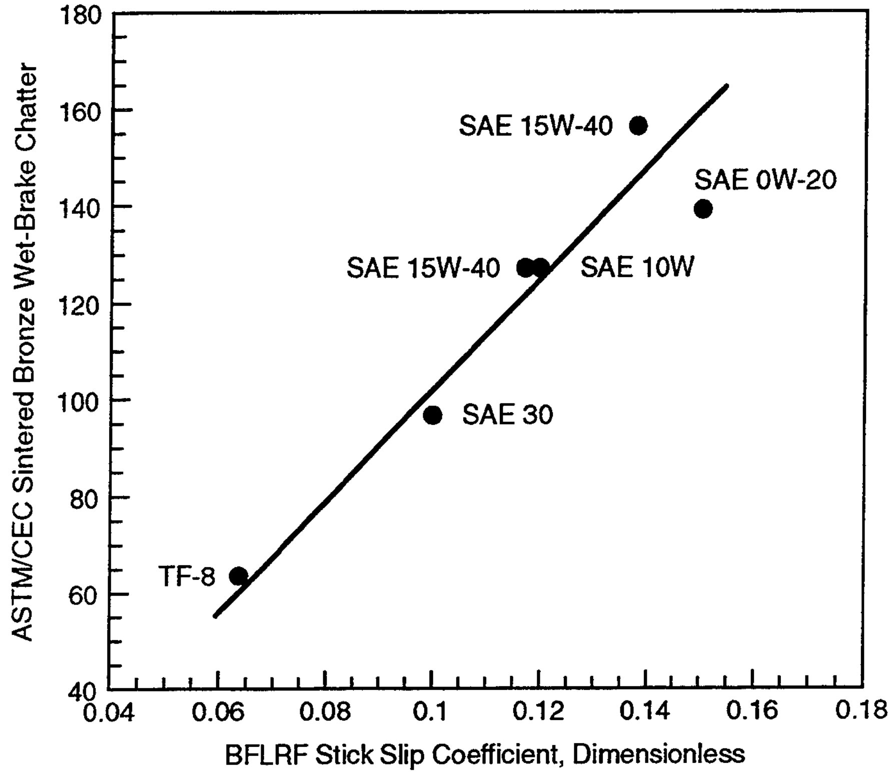


Figure 20. **Comparison between BFLRF stick-slip test and ASTM/CEC wet-brake chatter**

K. Electromagnetic Viscosity Apparatus

The Model TCV 300 Electromagnetic Viscosity Device (Cambridge Applied Systems) was designed for routine, repetitive viscosity measurements of engine lubricants in the viscosity range of 0.7 to 350 cSt, in four switch-selectable measurement ranges (0.7 to 3.5, 3.5 to 18, 18 to 70, and 70 to 350 cSt). To perform an evaluation, a 5-mL sample is decanted into the measurement chamber. After the **START** button is pressed, the apparatus brings the lubricant into the measuring chamber with a scrubbing action. The chamber and sample temperatures are stabilized at 40°C, and eight viscosity and temperature measurements are made. The device then averages the results, temperature compensates the output to adjust for differences between the actual measurement and the desired reference temperature of 40°C, computes the kinematic viscosity, and then displays the kinematic viscosity in centistokes. The excess sample is pumped from the chamber to ready it for the next sample. The use of the hydraulic scrubbing and pumping action to clean the chamber eliminates the need for environmentally harmful solvents or cleaners. The entire testing process requires less than 5 minutes. The displayed data can either be recorded manually or a computer can be connected to a terminal strip, which generates a 0- to 2-VDC signal that is proportional to the viscosity of the sample.

Initially, BFLRF conducted a series of tests using five viscosity reference fluids (K3, K6, K20, K60, and K200), along with several new oils. When tested in the ascending order of K3 through K200, the results were quite good. However, when testing the low viscosity K20 sample, immediately followed by the high viscosity K200 sample, several repeat tests with the same lubricant were required to stabilize the high viscosity results. This instability of the test sample was caused by the scrubbing action of the piston contaminating the new fluid in the measurement chamber with the residual fluid that remained in the drain tube (Fig. 21).

To minimize this contamination, a mix deflector was inserted into the drain tube flush with the bottom of the measurement chamber (see Fig. 22). With the mix deflector in place, tests were conducted with lubricants in the 20-cSt range and then in the 100-cSt range. When ascending from the lower to the higher viscosity oils, no more than two tests were required to stabilize the

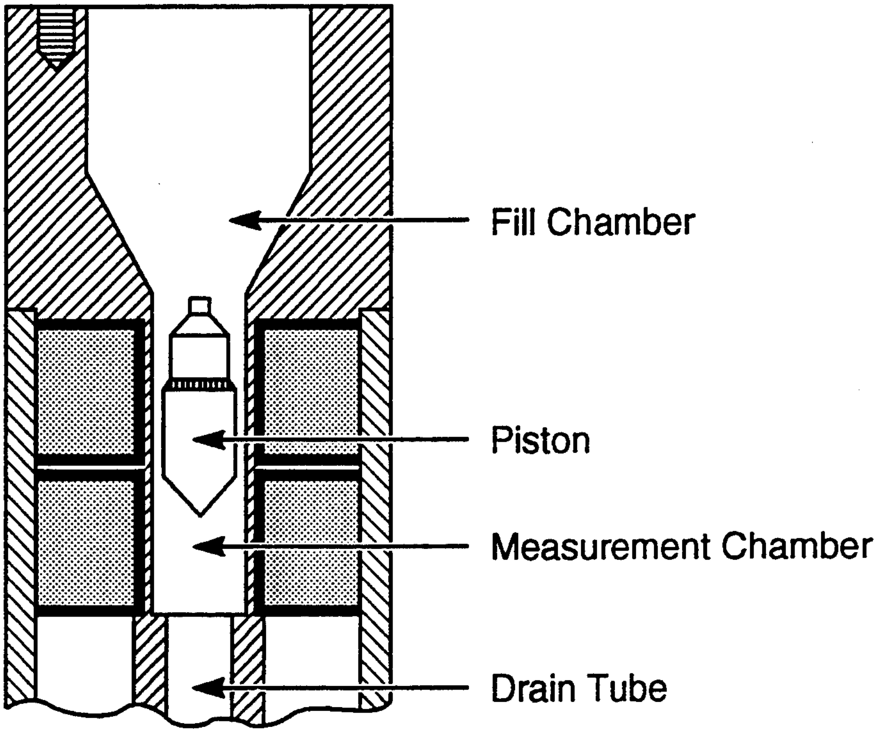


Figure 21. Details of viscosity sensor

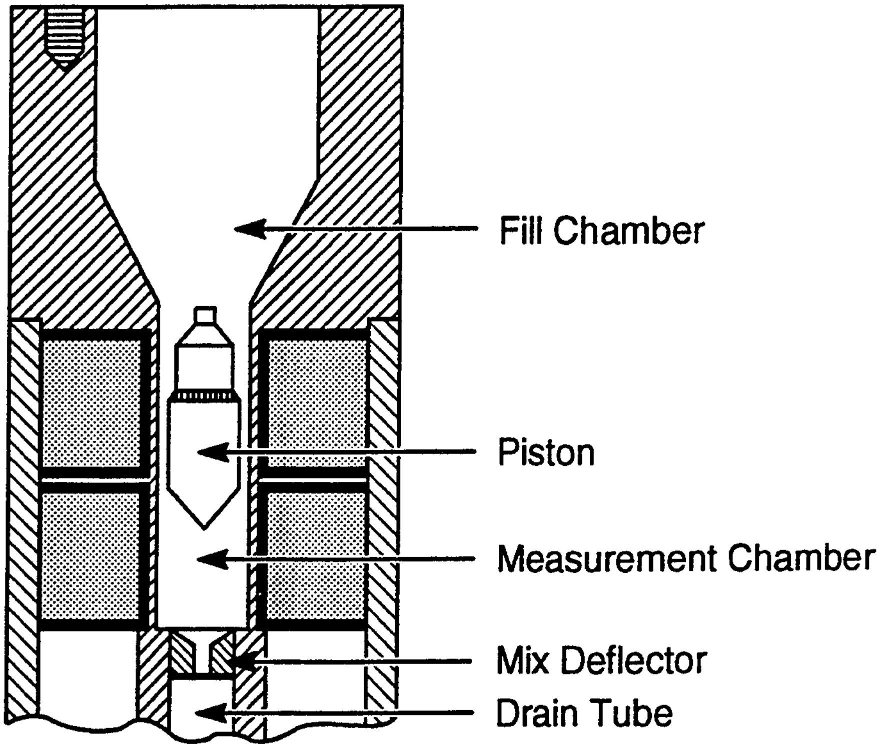


Figure 22. Mix deflector location

results. Then a series of tests was conducted with the modified device. These tests used 7 viscosity reference lubricants, 13 new lubricants, and 7 used lubricants. The results are listed in TABLE 13. These electromagnetic viscosity test results were then correlated to the ASTM D 445-measured viscosity at 40°C, producing the results shown in Fig. 23.

The electromagnetic viscosity apparatus using the 70- to 350-cSt range piston and the 70 to 350 calibration setting was used to evaluate the effect of viscosities outside that range.

The AL-19026-L viscosity was within the 70- to 350-cSt piston range and had the least difference, 2 percent (see TABLE 14). Lubricant AL-18677-L was outside the 70- to 350-cSt range and had a 10-percent difference. While the AL-19710-L lubricant was well beyond the 70- to 350-cSt range, it recorded a 50-percent difference. The manufacturer reported that a piston could be prepared that would cover the range of 15 to 200 cSt. In summary, this device shows great promise for being used in the LQA System.

TABLE 13. Viscosity Results Using ASTM D 445 Test Method and the Electromagnetic Device

Lubricant	Viscosity, cSt at 40°C	
	ASTM D 445	Electromagnetic Device
Reference Oils		
K3	3.0	3.0
K6	6.0	5.9
K20	20.0	19.6
K60	60.0	59.1
K176	176.0	174.2
K200	200.0	201.2
New Lubricants		
AL-15709-L	20.8	20.2
AL-18614-L	39.3	38.5
AL-18658-L	59.7	59.8
AL-18669-L	58.1	58.8
AL-18676-L	58.9	60.7
AL-18677-L	57.9	57.9
AL-18750-L	104.4	106.8
AL-18930-L	57.5	62.1
AL-18986-L	103.3	102.0
AL-19026-L	108.6	106.1
AL-19424-L	107.2	107.0
AL-19528-L	54.8	57.1
AL-19660-L	53.2	52.2
Used Lubricants		
AL-19746-L (Transmission)	69.2	70.1
AL-19747-L (Transmission)	110.0	102.8
AL-19750-L (Engine)	25.7	28.1
AL-19753-L (Engine)	82.4	83.4
AL-19758-L (Transmission)	85.2	81.8
AL-19765-L (Engine)	95.8	93.0
AL-19728-L (Generator)	151.5	151.0

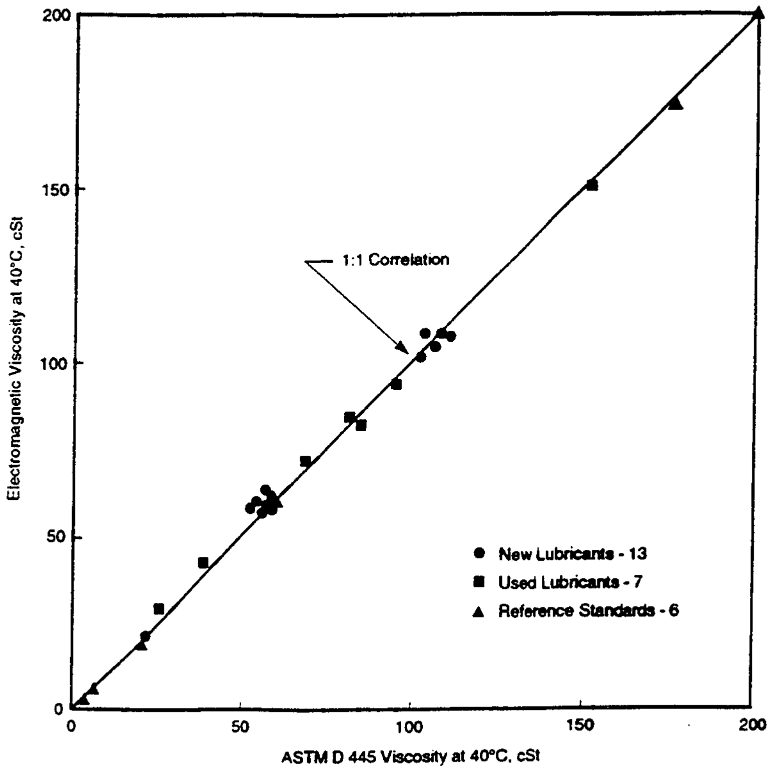


Figure 23. Electromagnetic viscosity versus ASTM D 445 viscosity at 40°C

TABLE 14. Effect on Viscosity Beyond the 70- to 350-cSt Piston Range

	Viscosity at 40°C, cSt		
Lube No.	Electromagnetic	ASTM D 445	% Difference
AL-19710-L	10.0	20.2	50
AL-18677-L	51.5	57.2	10
AL-19026-L	106.0	108.6	2

L. Dexsil Titra-Lube TBN

Twenty-two new lubricants were evaluated using the Dexsil Titra-Lube TBN kit. This disposable kit provides a colorimetric titration test for determining TBN values between 0 and 20 in approximately 5 to 7 minutes using 0.5-mL sample. These tests, which used MIL-L-2104 and MIL-L-21260 engine/transmission lubricants, OEA-30 candidates, MPTF, Caterpillar TO-4/TO-5, Caterpillar 1K reference and Sequence IIID/IIIE reference lubricants, were conducted in the laboratory at an approximate room temperature of 24°C (76°F). All tests were conducted using a minimum of two tests per sample.

This kit contained the following:

- One polyethylene tube with a fixspout cap containing two breakable ampules (one containing isooctane and the other containing a hydrochloric acid and isopropyl alcohol solution);

- One polyethylene tube with screw cap containing a sodium sulfate solution along with an ampule of methyl red in ethanol solution;

- A plastic syringe buret containing sodium hydroxide solution; and

- A plastic sample syringe.

The results from the Dexsil TBN kit were compared to the ASTM D 664 and ASTM D 2896 TBN test method results in TABLE 15. The results, when correlated to the D 664 TBN, were good, with good repeatability (Fig. 24). The figures show that the average results of the Dexsil TBN are slightly higher than the D 664 TBN results.

The Dexsil TBN results were compared to the D 2896 TBN. They produced a good correlation, as shown in Fig. 25. The Dexsil test kit had good repeatability.

Also, some old used AOAP oils were evaluated using the Dexsil TBN test. A sufficient quantity of fresh used engine oil samples were not available at this time. The old used AOAP samples only had D 664 TBN results. These results can be seen in Fig. 24. BFLRF is collecting used engine oil samples that will be tested and correlated with the ASTM D 664 and D 2896 TBN test methods.

TABLE 15. Results for Dexsil TBN Kit, ASTM D 664, and ASTM D 2896 TBN

AL-Code	Description	D 664 TBN	Dexsil TBN	D 2896 TBN
	New Oil Samples			
AL-18930-L	OEA-30 Candidate	6.6	9.5	9.9
AL-19424-L	SAE 15W-40 MIL-L-2104	7.3	9.5	8.6
AL-18750-L	SAE 15W-40 MIL-L-2104	8.0	9.0	8.1
AL-19026-L	SAE 15W-40 MIL-L-21260	8.5	7.4	7.4
AL-19528-L	OEA-30 Candidate	9.2	10.2	10.2
AL-12798-L	SAE 15W-40 MIL-L-2104	13.6	15.0	15.6
AL-15592-L	SAE 15W-40	14.0	15.0	--
AL-18614-L	MPTF	7.6	6.8	7.3
AL-18658-L	MPTF	11.5	12.0	10.6
AL-18669-L	MPTF	10.8	11.5	9.9
AL-18677-L	MPTF	13.2	15.0	15.2
AL-19660-L	OEA-30 Candidate	7.0	9.0	10.2
AL-18676-L	MPTF	11.2	10.7	9.8
AL-18986-L	Grade TO-4/TO-5 Service Fill	8.4	9.0	8.6
AL-19665-L	1-K Reference	13.1	14.0	14.5
AL-19666-L	1-K Reference	7.6	7.5	8.4
AL-19667-L	1-K Reference	6.8	7.5	8.1
AL-19636-L	IIID/IIIE Reference	4.5	9.5	8.9
AL-19637-L	IIID/IIIE Reference	0	7.0	8.2
AL-19638-L	IIID/IIIE Reference	5.3	5.0	5.7
AL-19639-L	IIID/IIIE Reference	0	7.5	7.0
AL-19640-L	IIID/IIIE Reference	7.5	9.5	9.2
	Old Used AOAP Samples			
AL-18695-L	MIL-L-2104	1.5	4.5	--
AL-18720-L	MIL-L-2104	2.1	4.0	--
AL-18723-L	MIL-L-2104	2.6	3.5	--
AL-18524-L	MIL-L-2104	3.1	5.5	--
AL-18388-L	MIL-L-2104	3.7	6.5	--
AL-17436-L	MIL-L-2104	4.4	6.0	--
AL-16872-L	MIL-L-2104	5.3	7.5	--
AL-18702-L	MIL-L-2104	6.9	7.5	--

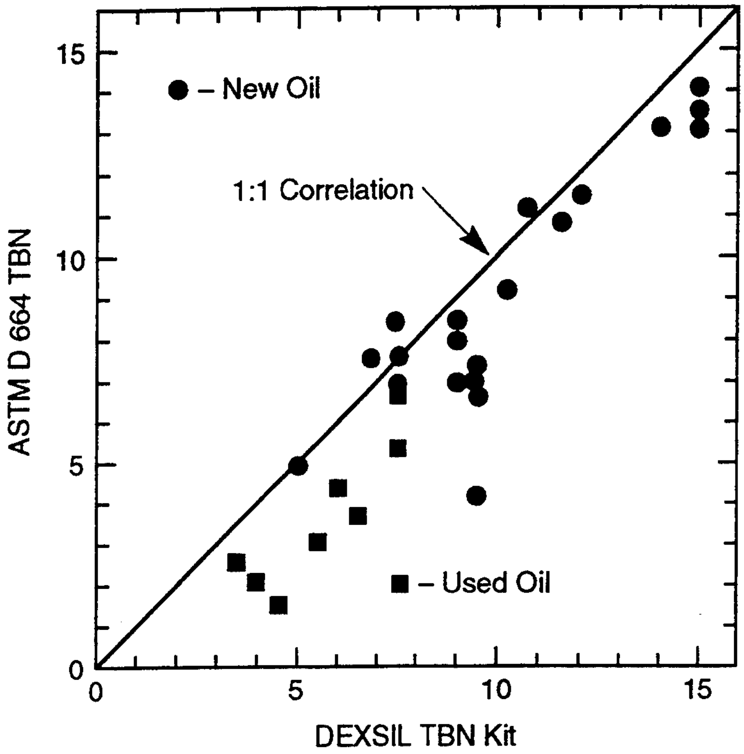


Figure 24. D 664 versus Dexsil TBN

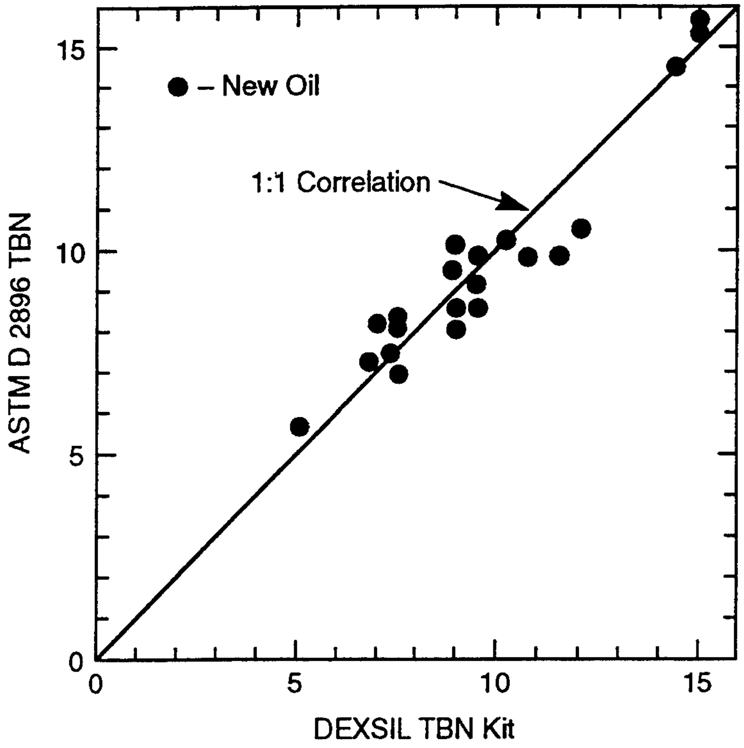


Figure 25. D 2896 versus Dexsil TBN

M. RULLER Test Device

The Remaining Used Lubricant Life Evaluation Rig (RULLER) test device was assembled from a commercially available, microcomputer-controlled voltammograph, which was equipped with a glassy carbon working electrode, a platinum wire reference electrode, and a platinum wire auxiliary electrode.

Oil samples from 100 to 300 μL were diluted with a water and acetone solution containing a neutral salt electrolyte with a suspended solid substrate. The sample was shaken, and the insoluble oil coated the solid substrate. After standing for 60 seconds, the agglomerated particulates quickly settled out to produce a clear solution for analysis. The voltage of the auxiliary electrode was scanned from 0.0 to 1.0 V at a rate of 0.5 V/second. The resulting peaks produced were then evaluated using a computer and were reported as percent of additive remaining. This test requires only 5 to 10 minutes, uses a small sample, and is conducted using inexpensive instrumentation. Lubricant samples with 1-percent alkyl- and 1-percent aryl-ZDP were thermally stressed to their breakpoint and half of the breakpoint time using the ASTM D 2272 RBOT. These samples were evaluated with the RULLER test device. Both the 1-percent alkyl- and 1-percent aryl-ZDP samples stressed to the breakpoint had no percent of remaining additive, while the one-half breakpoint recorded 61 and 42 percent of the alkyl and aryl remaining, respectively.

Used oil samples AL-18927-L, MIL-L-2104 SAE grade 30 from a recent engine fleet test were available for testing. These samples had been taken every 500 miles. They were evaluated, and the results depicted in Fig. 26. The additive, which appeared to be a multifunctional ZDP, was depleted at 4,000 miles and, at that point, the viscosity and TAN began increasing significantly until the lubricant was drained at 5,000 miles. These results showed great promise. Next, samples of a different lubricant from a Sequence IIID performance test were taken at each 8-hour sampling period. These results showed tremendous scatter (Fig. 26), with just a general decreasing additive trend. When the samples were evaluated simultaneously with one calibration, the results in Fig. 27 were produced. These results are representative of all the work conducted

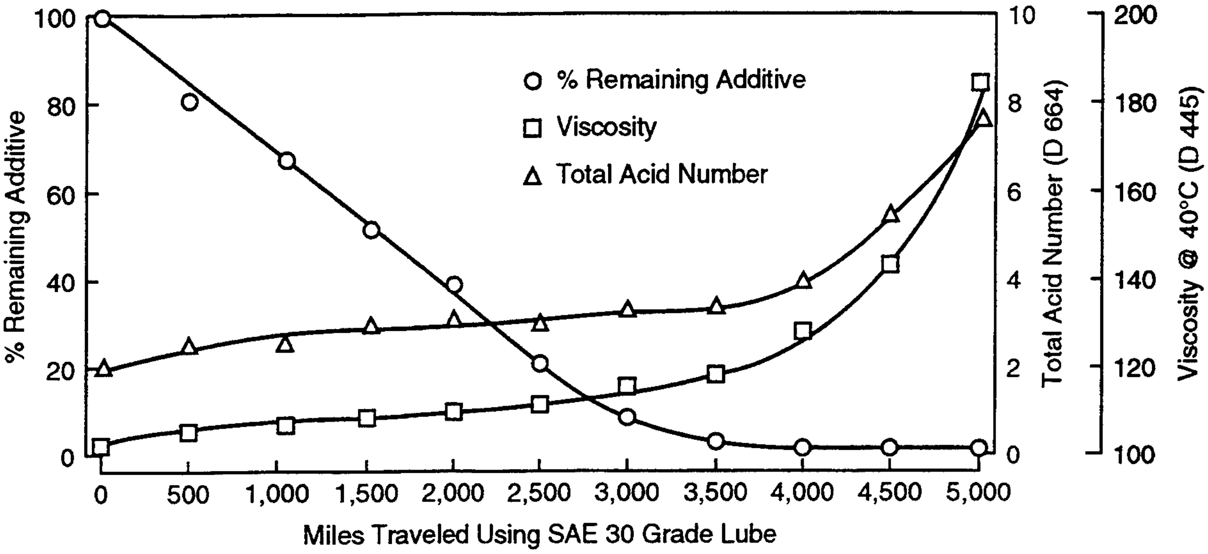


Figure 26. Lubricant samples from engine test fleet

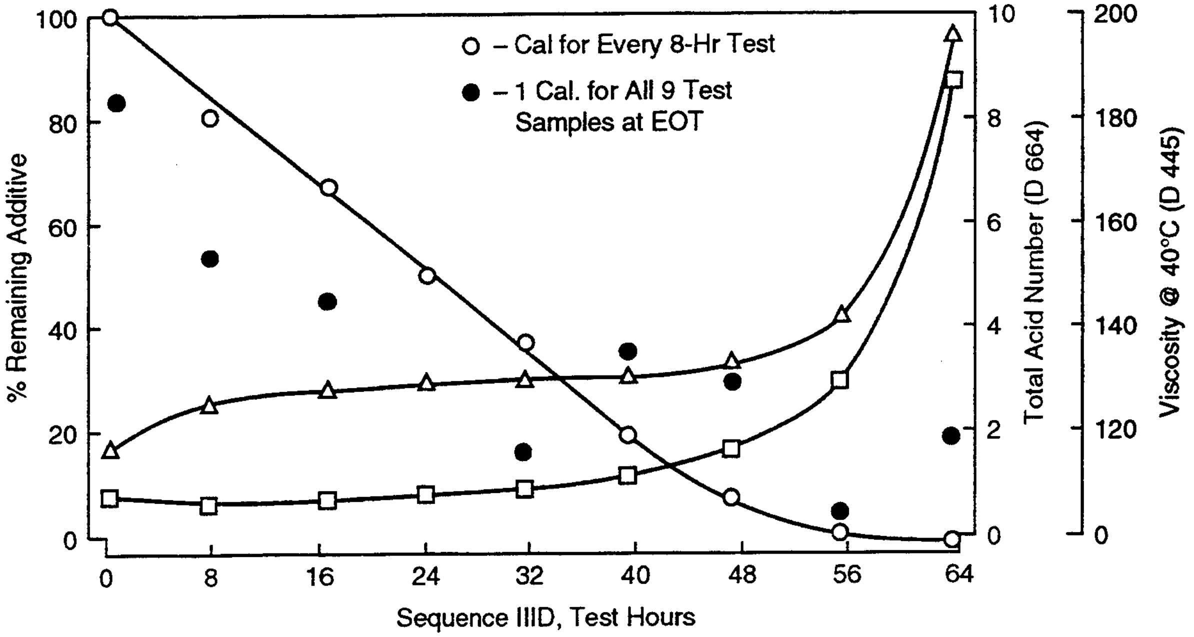


Figure 27. IIID engine test stand lubricant samples

with the RULLER test device. There was a problem of repeatability with test sequence baseline to test sequence baseline using the same reference oil. Therefore, the RULLER device did not do well with new lubricants due to data scatter. The RULLER device should be a good test device for both new and used lubricants when the repeatability bug, which appeared to be a technical problem, is solved.

N. RULLER TBN Test

Twenty-two new lubricants (MIL-L-2104 and MIL-L-21260 engine/used transmission lubricants, OEA-30 candidates, MPTF, TO-4/TO-5, 1-K reference and IIID/IIIE reference lubricants) were evaluated using the cyclic voltammetry (RULLER) TBN test method. The test uses only 100- to 200-μL sample size and leaves only 2- to 3-mL solution that must be disposed of (see TABLE 16). The RULLER test requires less than 5 minutes to conduct. The RULLER test is

TABLE 16. RULLER TBN Results Compared to ASTM D 664 and ASTM D 2896 TBN

AL-Code	Description	D 664 TBN	D 2896 TBN	Cyclic Voltammetry TBN
AL-18930-L	OEA-30 Candidate	6.6	9.9	13.9
AL-19424-L	SAE 15W-40 MIL-L-2104	7.3	8.6	15.3
AL-18750-L	SAE 15W-40 MIL-L-2104	8.0	8.1	15.5
AL-19026-L	SAE 15W-40 MIL-L-21260	8.5	7.4	15.4
AL-19528-L	OEA-30 Candidate	9.2	10.2	15.3
AL-12798-L	SAE 15W-40 MIL-L-2104	13.6	15.6	15.0
AL-15592-L	SAE 15W-40	14.0	--	15.7
AL-18614-L	MPTF	7.6	7.3	7.3
AL-18658-L	MPTF	11.5	10.5	9.6
AL-18669-L	MPTF	10.8	9.9	6.8
AL-18677-L	MPTF	13.2	15.2	7.7
AL-19660-L	OEA-30 Candidate	7.0	10.2	14.4
AL-18676-L	MPTF	11.2	9.8	6.5
AL-18986-L	TO-4/TO-5 Service Fill	8.4	8.6	5.0
AL-19665-L	1-K Reference	13.1	14.5	14.8
AL-19666-L	1-K Reference	7.6	8.4	15.8
AL-19667-L	1-K Reference	6.8	8.1	15.9
AL-19636-L	IIID/IIIE Reference	4.5	8.9	15.8
AL-19637-L	IIID/IIIE Reference	0	8.2	15.5
AL-19638-L	IIID/IIIE Reference	5.3	5.7	12.7
AL-19639-L	IIID/IIIE Reference	0	7.0	13.0
AL-19640-L	IIID/IIIE Reference	7.5	9.2	15.6

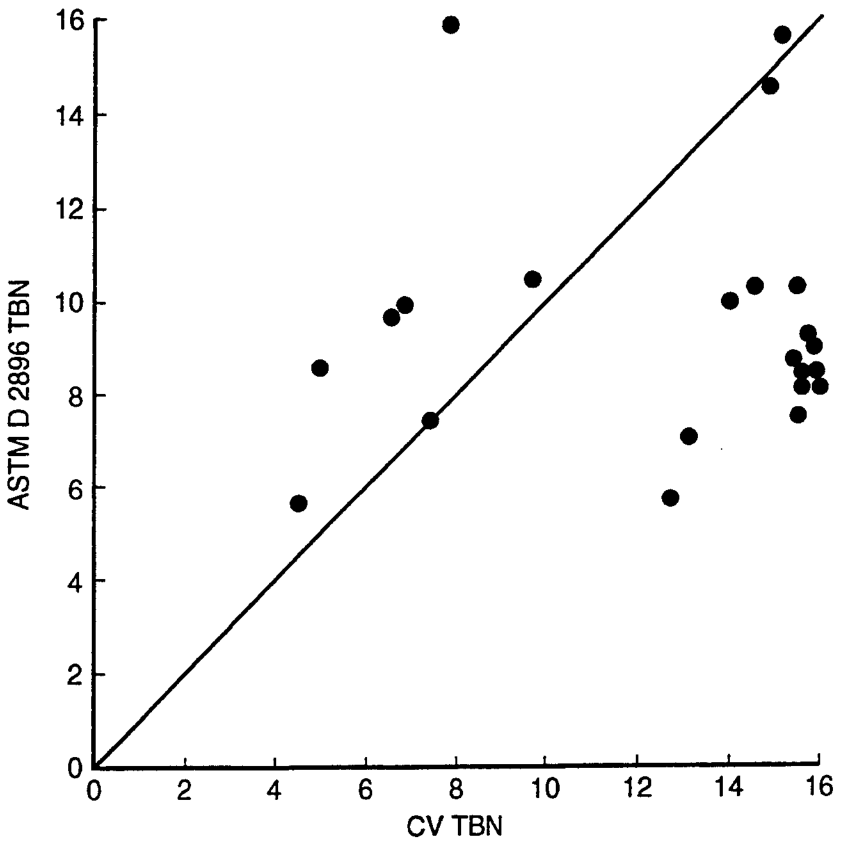
of interest because the system has been used to measure the remaining useful lubricant life (oxidative degradation) of used lubricants and could serve as a dual-function test kit. All lubricants were tested twice and averaged. The repeatability was quite good. These results were compared to the results from the ASTM D 664 and ASTM D 2896 TBN test methods. As seen in Fig. 28, there appears to be no correlation between these test methods. Due to the results from these correlations, no additional work is planned for the cyclic voltammetry TBN test method.

O. Caterpillar Microoxidation Test

The Caterpillar microoxidation test (CMOT) is an improved procedural refinement of the Penn State microoxidation test. Penn State also developed a method based on first-order kinetics deposit-forming reactions to rank the relative deposit-forming tendency of lubricants in the piston ring belt area and top land of heavy-duty diesel engines. The method uses a 20-µL sample and enough points to determine the induction time, which can be measured in 10 to 12 man-hours. These requirements make this microoxidation test quite inexpensive in comparison to a full-scale engine test as well as being much quicker to execute. This test was initially used to evaluate the Caterpillar 1-K pass, borderline, and fail reference lubricants. Tests were repeatable and correlated well with Caterpillar results (Fig. 29). The borderline lubricant had an induction time of 122 minutes, while BFLRF produced an induction time of 120 minutes on the same lubricant.

P. LUBTOT Device

In previous BFLRF work, a lubricant thermal oxidation tester (LUBTOT) device was developed (13) as a predictor of diesel engine deposits (Caterpillar 1G-2), and to better rate the deposits, the BFLRF Deposit Measuring Device (DMD) was developed (14) for use in measuring LUBTOT deposits. The LUBTOT uses an oil-air flow system. The tester consists of a stainless steel heater tube that serves as the test section. Oil and air are circulated directly around the outside of the regulated heater tube by a metering pump. An oil-in preheater maintains the oil-in temperature. Oil deposits formed on the outside of the heater tube. These deposits were then measured with the BFLRF DMD rating technique. These two methods were evaluated using



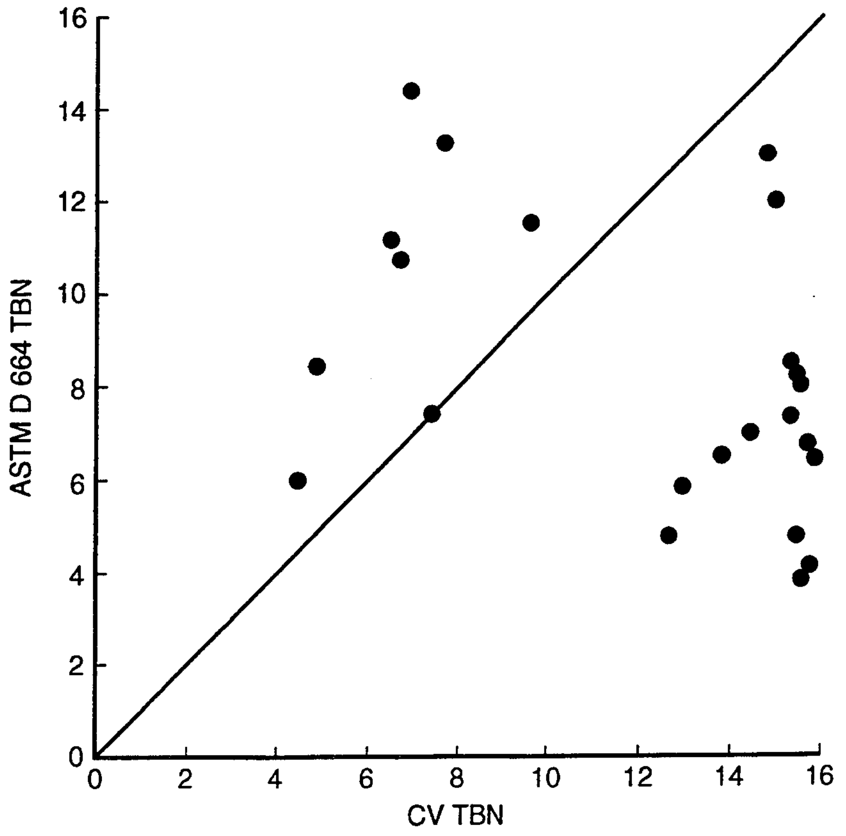


Figure 28. **ASTM TBN versus cyclic voltammetry TBN**

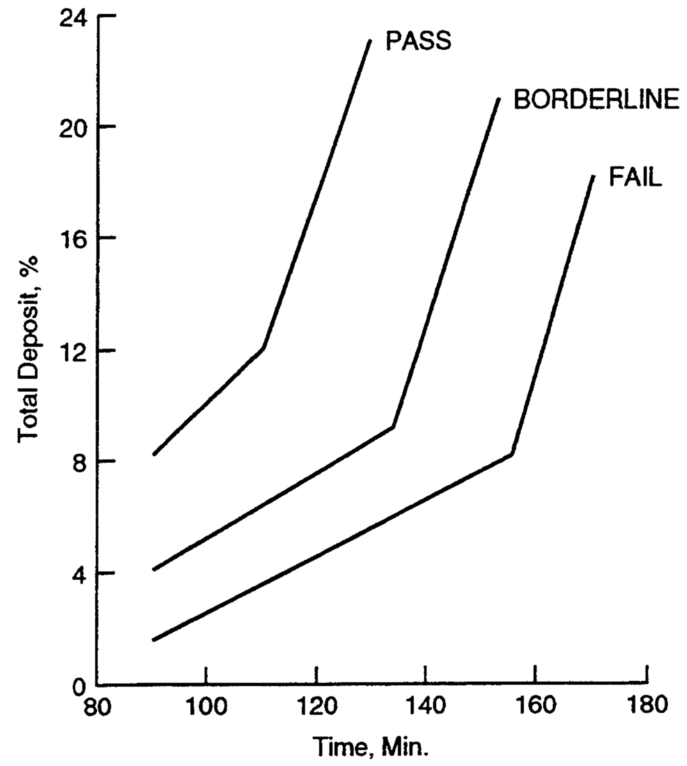


Figure 29. Caterpillar microoxidation test deposit formation curve at 230°C

three Caterpillar 1G-2 reference lubricants and nine MIL-L-2104E lubricants (four grade 30 and five grade 15W-40). These limited test results looked quite good when compared to the Caterpillar 1G-2 WTD Piston ratings (Fig. 30). All twelve lubricants fell within the maximum and minimum 1G-2 confidence range of industry reference runs. For the lubricants evaluated, these bench tests appear to show promise as a predictor of diesel piston engine deposits.

Q. TFOUT

This test was conducted at 160°C, utilizing a modified D 2272 RBOT bomb. The bomb was pressurized with oxygen, along with a lubricant mixed with a metal catalyst package, a fuel catalyst, and water. The bomb was rotated axially at 100 rpm until a rapid decrease of bomb pressure was observed. Five IIID reference oils were evaluated. The two fail oils had TFOUT breaktime averages of 31 and 53 minutes. The borderline pass oil at 85 minutes and the two pass oils at 134 and 143 minutes produced a good correlation when compared to the IIID engine test viscosity break (see Fig. 31 and TABLE 17). This work was not continued because this test

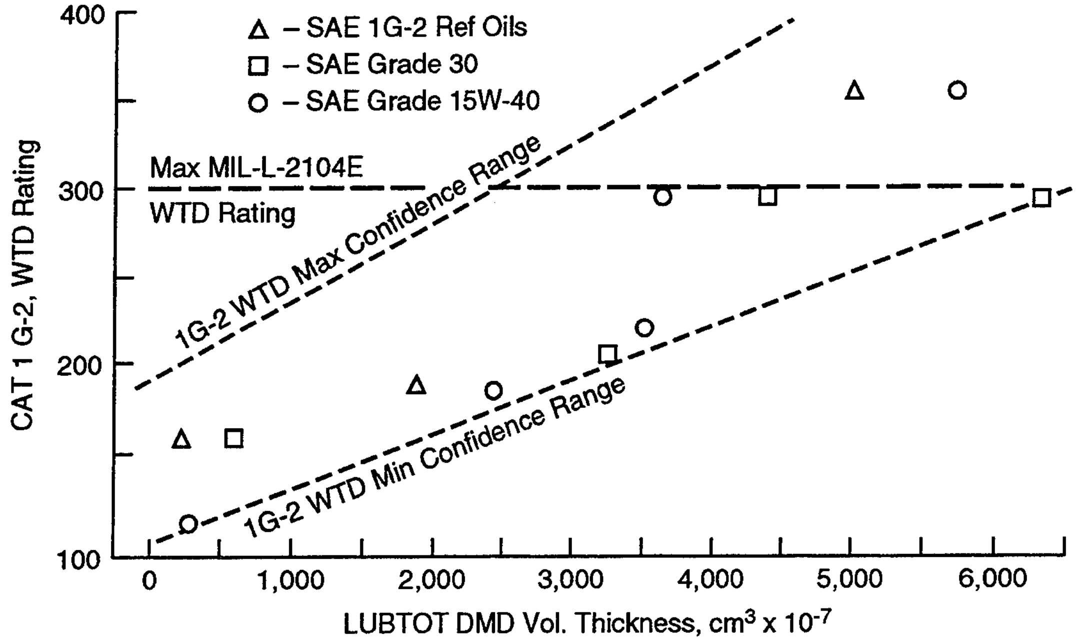


Figure 30. LUBTOT DMD ratings versus Caterpillar 1G-2 piston WTD ratings

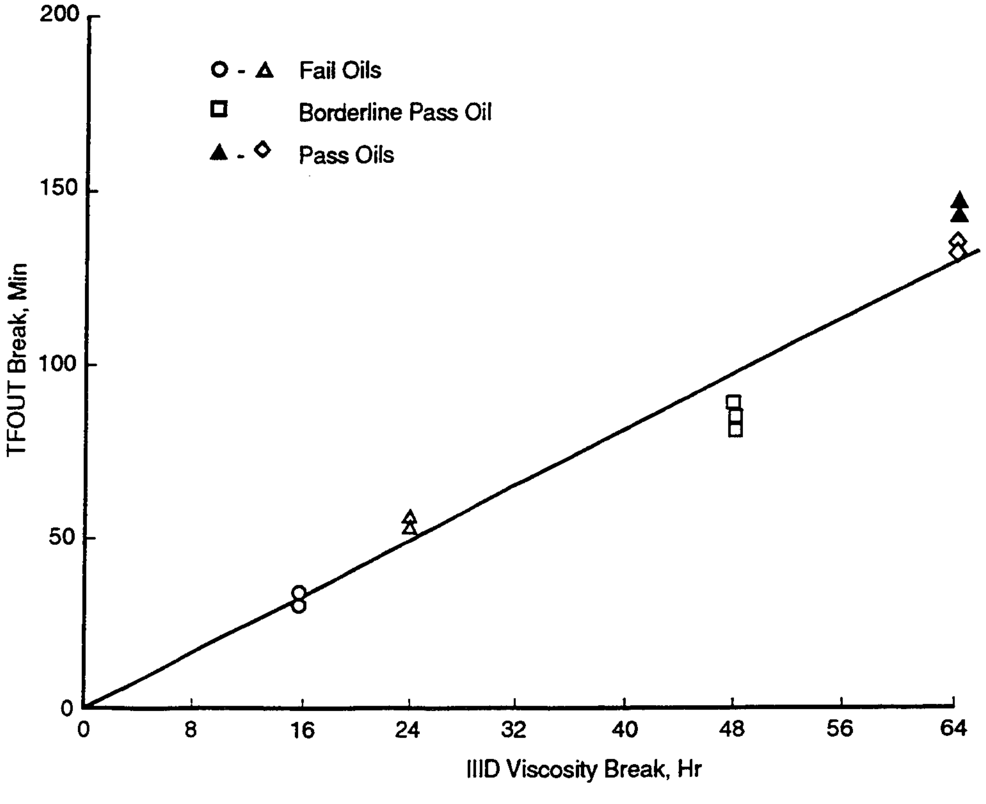


Figure 31. TFOUT versus IIID engine test using IIID reference oils

TABLE 17. IIID Viscosity Break Versus TFOUT Breaktime

Oil	IIID Engine Test Viscosity Break, hr	TFOUT Break, min.
76 A-3	64	143 (141, 145)
400	64	134 (131, 137)
73 B-1	48	85 (80, 85, 91)
77 B-3	24	53 (50, 56)
72 A-1	16	31 (29, 33)

pertains predominantly to gasoline engine service, and the U.S. Army has a very limited number of gasoline engines in service. It is possible that this test could be developed for diesel engine service, but funding constraints prohibited further development.

R. Total Dispersancy Number

MIL-L-2104 lubricants are formulated with dispersants which retain and suspend particles in the lubricant to minimize sludge deposits. Therefore, the development of a Total Dispersancy Number (TDN) is of interest.

The data from the 1988-89 detergent/dispersant TDN were reevaluated. Additionally, some work conducted with the GM 6.2L diesel engine in 1986-87 was reviewed. These data showed a good correlation between the ASTM D 893 insolubles test and the TGA soot tests. These data were encouraging enough to recommend work on the development of a TDN test method.

V. SUMMARY AND CONCLUSIONS

A. Specific Conclusions for Diesel Engine and Powershift Transmission Lubricants

1. Coolant/Water Contamination

The Gly-Tek test could detect ethylene glycol antifreeze containing as little as 0.25 wt% in both new and used lubricants. It appears that this test kit can be developed to detect ethylene glycol as part of the LQA System.

2. Viscosity Technique Test

The electromagnetic viscosity device results, when compared to the ASTM D 445 (derived viscosity at 40°C), produced an excellent correlation. This device shows good promise for being used in the LQA System.

3. Electrochemical Reaction Tests

The use of cyclic voltammetry (CV) appears very promising because measurements were made with both the oxidation and reduction waves. The waves increased in current with increases in thermal stress. These redox results are promising because this technique appears to be measuring the additive depletion through the decomposition products.

AC impedance data show a maximum tendency for the linking of alkyl- and aryl-ZDP decomposition products to the steel electrode with regard to thermal stress time. In addition, the linking of the aryl-ZDP decomposition products to the steel electrode is considerably greater than that determined for samples containing alkyl-ZDP.

When applied to used diesel engine oils, the RULLER test device results of percent remaining additive correlated very well with TAN and viscosity at 40°C. This test shows great possibility for both new and used lubricant application.

The RULLER TBN test results showed no correlation when compared to ASTM D 664 and ASTM D 2896 TBN results. In its present set-up, this test is not recommended for LQA System development.

The Complete Oil Breakdown Rate Analyzer (COBRA) was used to compare to viscosity, viscosity percent increase, TAN, oxidation, nitration, and water content. Since no good correlations were produced, no additional work with this device is being recommended.

The Dexsil TBN results correlated slightly higher than the ASTM D 664 TBN results and produced an excellent correlation with ASTM D 2896 TBN. The Dexsil TBN also had good correlations with ASTM D 664 and D 2896 when using used diesel engine lubricants. This test device shows great promise for use in the LQA System.

The pH test for those lubricants stressed with the modified FTM-5307 and ASTM D 2272 tests, along with several used lubricants, selected the highest TAN, lowest TBN, and highest TGA soot levels.

4. Fourier Transform Infrared Spectroscopy

The Fourier Transform Infrared (FTIR) Spectrophotometer has some valuable application potential. In a situation in which a limited number of new lubricants is used--so that the reference library is fairly complete--many common lubricant quality factors can be evaluated in less than a minute. The optimum applications appear to be in engine test support and motor pool environments in which new lubricant sources are somewhat controlled. The analysis would screen for fuel dilution, possibly replacing GC; for water, replacing Karl Fisher titration; oxidation and sulfation, replacing other infrared systems; and soot, replacing insoluble procedures. Aside from wear metals analysis and viscosity or TAN measurements, lubricant condition could be monitored. If the glycol and water and oxidation values could be accepted in lieu of viscosity and TAN values, only wear metals would need to be measured in addition to the FTIR. Earlier in the program, it was recommended that an FTIR be purchased for a more complete evaluation for Volume II work.

5. Laser Oil Spot Scanning Test

Initial limited work with the blotter spot by laser fluorescence was not promising, but the work showed promise in automating the blotter spot reading.

6. Conductivity/Dielectric Tests

The dielectric constant device for those lubricants stressed with the modified FTM-5307 and ASTM D 2272 tests, along with several used lubricants, selected the highest TAN, lowest TBN, corresponding DIR oxidation number, and highest TGA soot levels. This test device appears to indicate that failure of a lubricant can be detected regardless of its source of contamination, especially if the used lubricant characteristics are known.

7. Wet-Friction Tests

The friction retention and BFLRF stick-slip tests, using the Ball-on-Cylinder apparatus, produced very promising results for use in the LQA System. The Ball-on-Cylinder apparatus is also used to evaluate fuel lubricity, so it could play a multifunctional role in the PQA System.

8. Deposition and Oxidation

The Caterpillar microoxidation, LUBTOT, and TFOUT require some development in field hardening, but all three show good possibilities for LQA System use.

TDN shows good potential, but it would probably take many years to develop.

B. General

In conclusion, of the devices, techniques, and technologies evaluated, ten showed promise for being used in the LQA System. Of these, the Gly-Tek, dielectric, Dexsil TBN, and the RULLER

TABLE 18. Summary of Test Devices Evaluated – Potential Use

Test Devices	Long-Range Potential	Near Potential	
		Man-Portable	Transportable
Automatic Engine Oil Change	×		
Coolant/Water Contamination			
Gly-Tek		×	
Dielectric Constant		×	
Viscosity Technique Test			
Electromagnetic Viscosity			×
Electrochemical Reaction Tests			
Cyclic Voltammetry			×
AC Impedance	×		
RULLER Device		×	
RULLER TBN	×		
COBRA			
Dexsil TBN		×	
pH			×
Fourier Transform Infrared Spectroscopy (FTIR)			×
Laser Oil Spot Scanning Test	×		
Conductivity/Dielectric Tests		×	
Wet-Friction Tests			
Ball-on-Cylinder Tester			×
Cameron-Plint Tester			
Deposition and Oxidation			
Microoxidation Test	×		
LUBTOT			×
TFOUT	×		
TDN	×		

device show the best opportunity for development into man-portable devices to be used for maximum forward tactical testing (see TABLE 18). Six devices--the pH, FTIR, CV, Ball-on-Cylinder Wear, electromagnetic viscosity, and the LUBTOT--show the most promise as transportable devices used in operational and theater testing. When these devices are field hardened and integrated with a computer, they should meet the condemning criteria of

1) a rise in oil acidity;
2) a decrease in oil alkalinity;
3) an increase or decrease in viscosity;

4) a rise in contaminants; and
5) wet-friction performance.

They should give fast, on-site results, and be of the go or no-go type. They should also establish the usability of unused or new lubricants.

VI. RECOMMENDATIONS

Condition monitoring and instrumental chemical analysis is a rapidly advancing field, with new techniques and technologies being continually developed. The new techniques, such as Solid-State Microsensor devices (15, 16) and the Oil View Portable oil analyzer (17), should be continually monitored and evaluated for LQA System application and development. The ten test devices and techniques or technologies (both man-portable and transportable) that show the most promise should be further developed and correlated to lubricants from field vehicles and components and with stored and unknown lubricants. This development and correlation would provide the Army with an LQA System that could assess inservice, stored, and new lubricant quality.

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APPENDIX A

BFLRF (SwRI) Comments to Quartermaster School (QMS)

BFLRF (SwRI) Comments to Quartermaster School (ATSM-CDM) 20 February 1990 Letter, Subject: Portable Lubricant Quality Monitor (PLQM)

BFLRF (SwRI) Comments/Input traces the chronology of events identifying/justifying the NEED for the PLQM.

1. AFLRL Report No. 117, "Feasibility of Field Test Kits for Assessing In-Service Condition of Army Engine Oils," October 1979 (AD A081112) - "benefits of using field test device to establish in-service oil quality include increased equipment readiness, reduction in maintenance time and costs, reduce logistics volumes, and reduce drain-oil disposition problems (Army and environmental goals)."

2. TROSCOM response, October 1986, challenged the requirement to develop a PWMA for both aviation and ground equipment as quoted in 24 February 1987, Portable Physical Property Analyzer (MRSA), paragraph 4 (quotation from October 1986 letter from TROSCOM, MG Skeen).

> "This is a good effort, and the end result will provide even greater benefits to the Army. There is an area, however, that calls for further consideration. Currently, only those items of equipment enrolled in AOAP benefit from physical property analysis. Many other items that are exempt from program participation could benefit if procedures and instrumentation were made available to a degree that would allow their participation. An example of this concept is a portable physical property test device. Such a device would be required to provide a measurement of viscosity, a determination of the lubricant's resistance to acids, a measurement of water and coolant in the oil, and other basic indicators of lubricant condition. The development of this device could place an analysis capability closer to the user."

3. Memorandum through Chief of Staff for Commander, AMC, 9 January 1987. Subject: The Army Oil Analysis Program (AOAP).

> "The Army Materiel Command (AMC) Inspector General was recently requested to investigate the AOAP. This IG inspection was conducted during 1Q FY 1987. Their summary conclusions found the AOAP to be valid and

> well established in the field. The aeronautical portion of the AOAP was found to have no disconnects and was being used as a baseline. The non-aeronautical portion of the AOAP, however, was found to have significant systemic problems."

4. Excerpts from 5 February 1987, Deputy CG for AMC (LTG Burbules) Note to BG Donovan, BG Stanlcup, and COL Schneider:

> "Discussion on 3 February 1987 raised some continuing concerns about the utility of the Army Oil Analysis Program (AOAP); particularly for ground combat vehicles.
>
> It appears that if the program is going to be successful a requirement exists to simplify the procedures to analyze oil samples.
>
> What is needed is a simple device that allows the user to take an oil sample and analyze it immediately--on the spot. To this end, the following taskers are assigned:
>
> - AMCMI – immediately initiate a market survey of foreign sources to determine the availability of an on-the-spot oil analysis device. Provide 30-day status updates to DCGMR as of date of this note.
> - AMCDE – immediately start an aggressive logistics R&D effort to develop a device to do on-the-spot analysis. Provide 30-day status updates to DCGMR as of date of this note.
> - AMCSM – answer the following question: Should we suspend the oil analysis program for ground combat vehicles until an on-the-spot oil analysis device is available?"

5. Proposed Development of a Portable Lubricant Quality Monitor, Fuels and Lubricants Division, (STRBE-VF), Belvoir RD&E Center, 28 April 1987, (paragraph 10):

> "Recent technological developments have shown the potential for developing a portable device (i.e., a Portable Lubricant Quality Monitor) that <u>can</u> rapidly determine "oil condition" qualities utilizing the types of physical/chemical test technologies mentioned in paragraph 9. The technology exists for development of the subject device. This is evidenced in reviewing a sampling of recent articles published in the open literature. Enclosure 1 entitled "An Annotated Bibliography of Selected References Covering Lubricant Testers" supports this position. However, there is no currently marketed kit available at this time,

> which can define the prerequisite oil characteristics needed to assess in-service oil quality."

6. Memorandum for Commander, USAQMS (Ft. Lee) from Fuels and Lubricants Division, (STRBE-VF), Belvoir RD&E Center, 14 December 1987 (paragraphs 3 and 4):

> "To determine whether such an approach would be supported by the appropriate schools, Reference 1 solicited a statement of interest from eight TRADOC organizations.* Responses from each of the schools have been received and these are attached as enclosures 1-7 (Note: No response was received from the U.S. Army Logistics Center). Comments were also provided by the U.S. Army General Materiel and Petroleum Activity and these are attached as Enclosure 8.
>
> In reviewing these comments, all would support the development of the "Portable Lubricant Quality Monitor" as there appears to be a bona fide need for this capability. This positive responsiveness should therefore serve as a basis upon which a requirements document can evolve. We would like to offer our assistance at this time in developing such a document."

7. Memorandum dated 28 December 1987 with Mr. Ambrose's (Office of Under Secretary of Department of the Army) comments on it. Memorandum for Executive to CG, USAMC.

> Memorandum for Executive to CG, USAMC, Subject: Hand Held Oil Analyzer.
>
> Please see Mr. Ambrose's comments on the attached memo:
>
> > "We surely need <u>something</u>, especially in third world locations. The troops will use whatever they can get their hands on, unless there is some means <u>readily</u> <u>at</u> <u>hand</u> to tell them OK or not.
> >
> > "It seems to me the issue is not 'accurate, reliable correlation with AOAP laboratory analysis', but

---

* Copies sent to CACDA (Ft. Leavenworth); ATSF-CML (Ft. Sill); ATZA-CDM (Ft. Belvoir); ATSH-CD-MLS-M, Infantry School (Ft. Benning); HQ USAOC&S (APG, MD); ATSM-CDM, QMS (Ft. Lee); ATSB-CO-ML, Armor School (Ft. Knox); Army Logistics Center

a. Will the damned stuff function in my machine on a short-term basis?

b. Is it loaded with water, sand, sugar, etc.?

"It seems incredible that we are no further along on such an item."

8. The Battenfield substandard new engine, transmission and hydraulic lubricants in the Army inventory; this problem further identifies the need for capability to verify new-oil quality in the field.

Summary and Conclusions

In reviewing the above positive need responses and comments (Nos. 1-8) they show there is a bona fide need for the development of the PLQM capability. In addition, the question has never been answered whether AOAP quasiqualitative physical/chemical tests correlate with standard ASTM physical/chemical test. Also, the following observations are made concerning the PLQM:

- What is needed is a simple device that allows the user to take an oil sample and analyze it—on the spot.
- The PLQM would aid in eliminating the usage of substandard lubricant or the wrong fluid by the user.
- It would appear that **when** the PLQM is developed and deployed at the depot/unit/field motor pool level, it would greatly increase the number of testing capabilities.
- Thus, the wartime role would be greatly enhanced, especially when our mobile forces are operating in underdeveloped theaters.
- In addition, it would increase equipment readiness, reduce maintenance time and costs, reduce logistics volumes and reduce drain-oil disposition problems (these are Army and environmental goals).

- The needed effort for PLQM development is currently being funded by AMC (BRDEC).

  a. A marketing and literature survey was made for on-the-spot devices and technologies. However, there is no currently marketed kit available that can define the prerequisite oil characteristics needed to assess in-service oil quality.

  b. Twenty-two devices and technologies were selected for PLQM development.

  c. These devices and technologies are being evaluated and compared to ASTM laboratory and field lubricant test results for possible PLQM usage.

What appears to be missing, to continue moving this worthwhile effort forward, is an Army concept document or wartime requirement (from TRADOC or FORSCOM).

Acronyms Used in Appendix A

TROSCOM	Army Troop Support Command
PWMA	Portable Wear Metals Analyzer
USAQMS	U.S. Army Quartermaster School
USAMC	U.S. Army Materiel Command
AOAP	Army Oil Analysis Program
TRADOC	Army Training and Doctrine Command
FORSCOM	Army Forces Command

APPENDIX B

Briefing Package
"Lubricant Quality Analysis System"

LUBRICANT QUALITY ANALYSIS SYSTEM
(LUQAS)

---

for

Belvoir Research, Development and Engineering Center's
Petroleum Quality Analysis System
(PQAS)

in support of

U.S. Army Quartermaster School's
Petroleum Field Testing

PRESENT AND PREVIOUSLY PROPOSED OIL ANALYSIS SYSTEMS

- ARMY OIL ANALYSIS PROGRAM (AOAP)
- PORTABLE OIL SPECTROSCOPIC ANALYZER (POSA)
- PORTABLE LUBRICANT QUALITY MONITOR (PLQM)
- ACCELERATED FUELS LUBRICANTS QUALIFICATION PROCEDURES (AFLQP)

PRESENT AND PREVIOUSLY PROPOSED OIL ANALYSIS SYSTEMS (Cont'd)

- WHAT AOAP CURRENTLY DOES:

  DETERMINES EQUIPMENT CONDITION BY MEASUREMENT OF SPECTROMETRIC USED OIL WEAR METALS AND OTHER CONTAMINANTS BY QUASI-QUALITATIVE CHEMICAL/PHYSICAL TESTS AND IS DEPLOYED AT SELECTED BASE LABORATORIES

- WHAT THE PROPOSED POSA WOULD DO:

  A TRANSPORTABLE TEST INSTRUMENT FOR DETERMINING AVIATION EQUIPMENT CONDITION BY SPECTROGRAPHIC MEASUREMENT OF WEAR METALS AND BE DEPLOYED IN SELECTED DEPOT AND FIELD LOCATIONS

PRESENT AND PREVIOUSLY PROPOSED OIL ANALYSIS SYSTEMS (Cont'd)

- WHAT THE PROPOSED PLQM WOULD HAVE DONE:

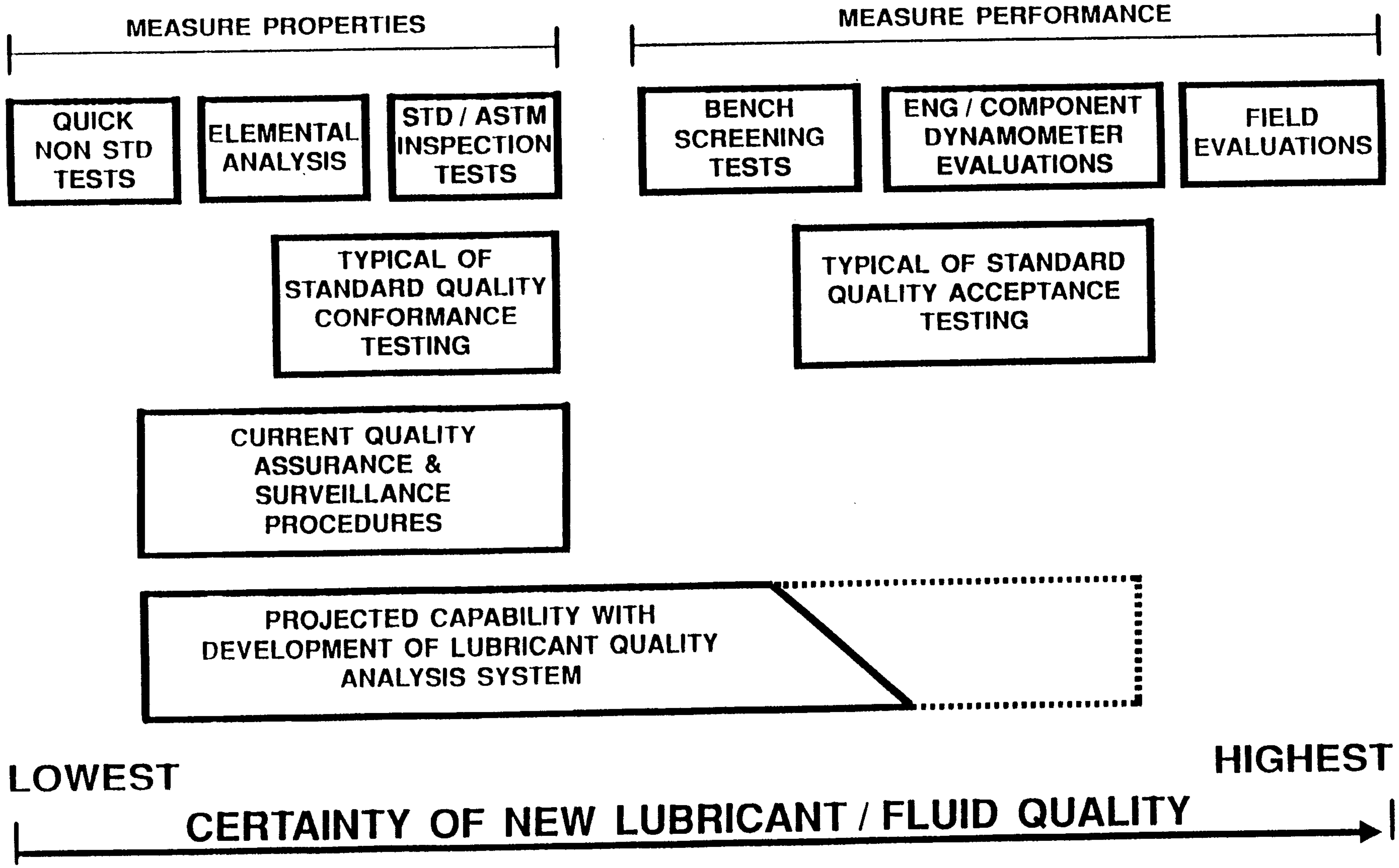
  DETERMINE IN-SERVICE CONDITION OF GROUND ENGINE VEHICLE/EQUIPMENT LUBRICANTS (ON-THE-SPOT) AND BE DEPLOYED AT END-USER LEVELS

- WHAT THE PROPOSED AFLQP WOULD DO:

  PROVIDE BENCH-SCALE TESTS AND MODELS TO ASSESS LUBRICANT PERFORMANCE QUALITY, MINIMIZING NEED FOR EXPENSIVE ENGINE DYNO TESTS

- NONE OF THE ABOVE PROVIDE ACCEPTABLE MEANS FOR ASSURING NEW OIL QUALITY

METHODS TO DETERMINE LUBRICANT / FLUID QUALITY



MEASURE PROPERTIES

MEASURE PERFORMANCE

QUICK NON STD TESTS

ELEMENTAL ANALYSIS

STD / ASTM INSPECTION TESTS

BENCH SCREENING TESTS

ENG / COMPONENT DYNAMOMETER EVALUATIONS

FIELD EVALUATIONS

TYPICAL OF STANDARD QUALITY CONFORMANCE TESTING

TYPICAL OF STANDARD QUALITY ACCEPTANCE TESTING

CURRENT QUALITY ASSURANCE & SURVEILLANCE PROCEDURES

PROJECTED CAPABILITY WITH DEVELOPMENT OF LUBRICANT QUALITY ANALYSIS SYSTEM

LOWEST

HIGHEST

CERTAINTY OF NEW LUBRICANT / FLUID QUALITY

LUBRICANT QUALITY ANALYSIS SYSTEM
(LUQAS)

- WHAT IS IT AND WHAT WILL IT DO?
- WHY IS IT NEEDED?
- WHERE AND HOW WILL IT BE DEPLOYED?
- DETAILED DISCUSSION
- BENEFITS

LUQAS

WHAT IS IT AND WHAT WILL IT DO?

- A SET OF OIL ANALYSIS DEVICES TO PROVIDE NEW LUBRICANT/FLUID QUALITY CONTROL THAT USE BOTH STANDARD LABORATORY TESTS AND BENCH-SCALE PERFORMANCE TESTS

- AN ANALYSIS SYSTEM THAT WILL BE FULLY RESPONSIVE TO FIELD AND BASE OPERATIONS

LUQAS

WHY IS IT NEEDED?

- TO FILL AN EXISTING NEED FOR ASSESSING LUBE OIL AND FLUID QUALITY IN THE FIELD
- TO FILL AN EXISTING NEED FOR IMPROVED SHELF LIFE RETESTING. CURRENT TESTS ARE ONLY: APPEARANCE, VISCOSITY AT 100°C, FLASH POINT, POUR POINT, AND FOAMING TENDENCIES
- WILL ENABLE A NEW OPPORTUNITY TO REDUCE UNNECESSARY DOWNGRADING OF DEPOT STOCKS DUE TO "NON-PRECISE" SHELF LIFE TESTING

LUQAS

WHERE AND HOW WILL IT BE DEPLOYED?

- DEPLOYMENT AT MOBILE PETROLEUM LABORATORIES
- OPERATION BY PERSONNEL IN POL MILITARY OCCUPATIONAL SPECIALTIES IN ADDITION TO THOSE TASKS ALREADY ASSIGNED
- THIS TECHNOLOGY COULD BE USED IN BASE LABORATORY IF DESIRED

LUQAS

WILL BE USED TO:

- VERIFY COMPOSITION/PROPERTIES OF KNOWN QUALIFIED PRODUCTS
- ACCOMMODATE GREATER USE OF LUBES/FLUIDS AVAILABLE FROM HOST NATION SUPPORT AGREEMENTS AND/OR COMMERCIAL SOURCES
- DETERMINE COMPOSITION/PROPERTIES OF UNKNOWN LUBES/FLUIDS
- PREDICT/VERIFY ACCEPTABLE PERFORMANCE OF UNKNOWN LUBES/FLUIDS TO BE USED IN ARMY VEHICLES/EQUIPMENT
- IMPROVE RELIABILITY OF RETESTING DEPOT STOCKS

L U Q A S
ENCOMPASSES OIL PROPERTIES & PERFORMANCE TESTS FOR NEW LUBRICANT/FLUID QUALITY ANALYSIS

PROPERTY TESTS	PROPOSED METHOD
VISCOSITY	MAGNETIC, VIBRATING BALL
TOTAL ACID NUMBER (TAN)	AUTOMATED TITRATION
TOTAL BASE NUMBER (TBN)	AUTOMATED TITRATION, CYCLIC VOLTAMMETRY (CV)
FLASH POINT	TBD
ADDITIVE ELEMENTS	EMISSION SPECTROMETER
INFRARED ANALYSIS (CATALOGING OR FINGER PRINTING)	FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR) NICOLET
LOW TEMPERATURE	TBD

PERFORMANCE TESTS	
OIL OXIDATION	TBD (I.e., DSC, TGA, TFOUT, MICROOX, CV, etc.)
DEPOSITION (HIGH & LOW TEMP)	TBD (LUBTOT, MOD. COKER, etc.)
WEAR	TBD (WEAR RIG, PROPERTY CORRELATION, etc.)
FOAM	TBD (RIG, PROPERTY CORRELATION, etc.)

TBD = TO BE DETERMINED

L U Q A S

ADDITIONAL BENEFITS DETERMINE THE FOLLOWING FUEL PROPERTIES:

FUEL PROPERTY TESTS	PROPOSED METHOD
VISCOSITY	MAGNETIC, VIBRATING BALL
TAN	AUTOMATED TITRATION
FLASH POINT	TBD
WATER	FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)
ADDITIVE DEPLETION	FTIR, CYCLIC VOLTAMMETRY (CV)
OXIDATION	DIFFERENTIAL SCANNING CALORIMETER (DSC) THERMAL GRAVIMETRIC ANALYSIS (TGA)

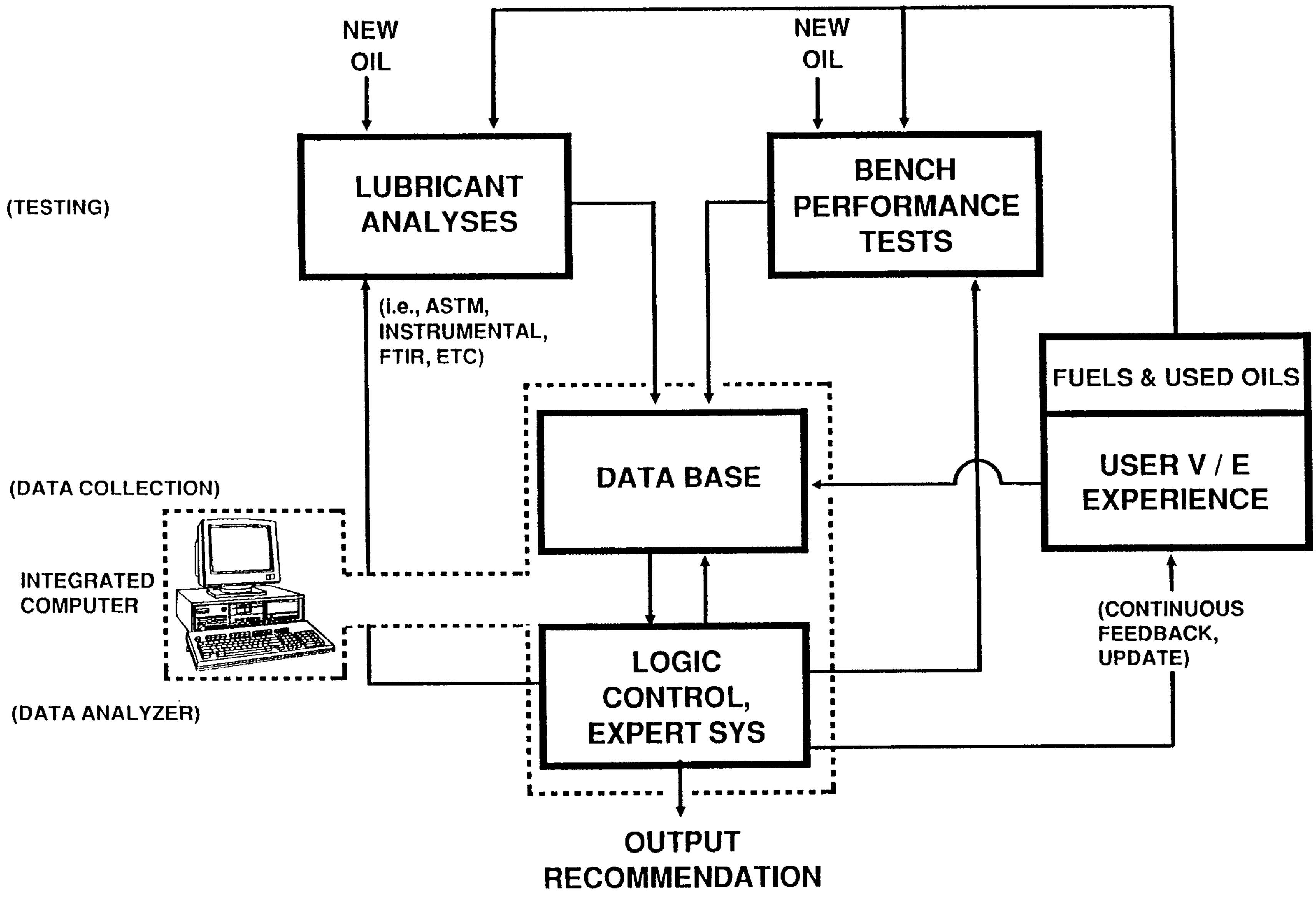
LUQAS CAN ALSO DETERMINE IN-SERVICE OIL CONDITION

LUQAS

PRODUCT QUALITY WILL BE ANALYZED BY:

- INTEGRATED COMPUTER SYSTEM
- LOGIC CONTROL ("EXPERT SYSTEMS")
- AUTOMATED ANALYSIS OF RESULTS
- MENU-DRIVEN AND USER-FRIENDLY, PROVIDING MAXIMUM FLEXIBILITY

LUQAS FLOW CHART



BENEFITS OF LUQAS

MAJOR BENEFITS

- PROVIDE A FIELD CAPABILITY THAT CURRENTLY DOES NOT EXIST
- QUALITY ASSURANCE OF FIELDED LUBRICANTS/FLUIDS:
    - QUALIFIED PRODUCTS
    - NONQUALIFIED/COMMERCIAL PRODUCTS
    - UNKNOWN PRODUCTS
- DEFINES OIL LOGISTICS REQUIREMENTS
- PROVIDES ADDITIONAL BATTLEFIELD POL ANALYSIS CAPABILITIES
- REDUCES POTENTIAL FOR OIL/FLUID-RELATED MAINTENANCE PROBLEMS DUE TO USE OF QUALITY-DEFICIENT PRODUCTS
- PROVIDES PRECISION TO SHELF-LIFE RETESTING PROCEDURES

BENEFITS OF LUQAS (Cont'd)

ASSOCIATED ARMY BENEFITS:

- ACCELERATES THEATRE-WIDE LOGISTICS DECISION MAKING
- IMPROVED VEHICLE/EQUIPMENT READINESS
- REDUCTION IN MAINTENANCE COST AND TIME
- SYSTEM IS VERSATILE—MEETS THE NEEDS OF MULTIPLE PROPONENTS PROVIDING ALTERNATE USES:
  - EXTENSION OF USEFUL LIFE OF LUBES/FLUIDS (MAINTENANCE)
  - REDUCE DRAIN-OIL DISPOSITION (ENVIRONMENTAL)

APPENDIX C

List of Abbreviations

AC	Alternating Current
Ag/AgCl	Silver/Silver Chloride
AOAP	Army Oil Analysis Program
ASTM	American Society of Testing and Materials
ATR	Attenuated Total Reflectance
BFLRF	Belvoir Fuels and Lubricants Research Facility
COBRA	Complete Oil Breakdown Rate Analyzer
CSV	Cathodic Stripping Voltammetry
CV	Cyclic Voltammetry
DIR	Differential Infrared Analysis
DMD	Deposit Measuring Device
FEDRIP	Federal Research in Progress
FTIR	Fourier Transform Infrared Spectroscopy
FTM	Federal Test Method
GC	Gas Chromatography
Hg/Au	Mercury/Gold
Hz	Hertz
LFQAS	Lubricant/Fluids Quality Analysis System
$LiClO_4$	Lithium Perchlorate
LQA	Lubricant Quality Analysis
LUBTOT	Lubricant Thermal Oxidation Tester
MPTF	Multipurpose Transmission Fluid
NTIS	National Technical Information Service
OEA	Oil Engine Arctic
PLQM	Portable Lubricant Quality Monitor
POL	Petroleum, Oil, Lubrication
PQA	Petroleum Quality Analysis
QMS	Quartermaster School
QPL	Qualified Product List
RBOT	Rotating Bomb Oxidation Test
RCV	Reductive-Cyclic Voltammetry
RLLAT	Remaining Lubricant Life Assessment Test
RULLER	Remaining Used Lubricant Life Evaluation Rig
SAE	Society of Automotive Engineers
SwRI	Southwest Research Institute
TACOM	Tank-Automotive Command
TAN	Total Acid Number
TARDEC	Tank-Automotive Research, Development and Engineering Center
TBN	Total Base Number
TDN	Total Dispersancy Number
TGA	Thermal Gravimetric Analysis
V	Volt
WTD	Weighted Total Deposit
ZDP	Zinc Dithiophosphate

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