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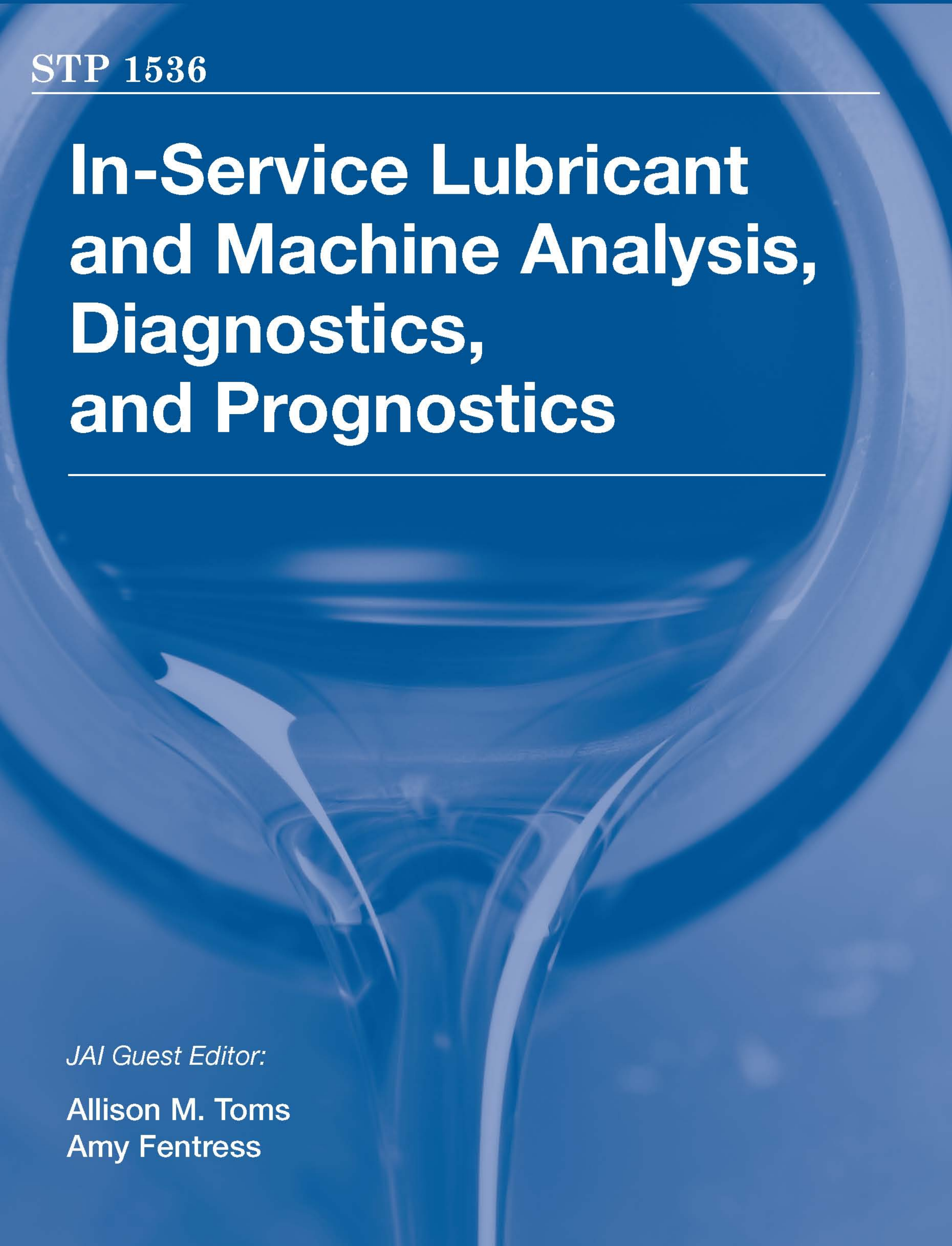
In-Service Lubricant and Machine Analysis, Diagnostics, and Prognostics



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In-Service Lubricant and Machine Analysis, Diagnostics, and Prognostics

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Foreword

THIS COMPILATION OF THE *JOURNAL OF ASTM INTERNATIONAL (JAI)*, STP1536, *In-Service Lubricant and Machine Analysis, Diagnostics, and Prognostics,* contains only the papers published in JAI that were presented at a symposium on In-Service Lubricant and Machine Analysis, Diagnostics, and Prognostics held during December 8, 2010 in Jacksonville, FL. The symposium was sponsored by ASTM Committee D02 on Petroleum Products and Lubricants and Subcommittee D02.96 on In-Service Lubricant Testing and Condition Monitoring Services.

The Symposium Co-Chairs and JAI Guest Editors are Allison M. Toms, GasTOPS Inc., Pensacola, FL and Amy Fentress, Lubrication Engineers, Wichita, KS.

Contents

Overview

This publication contains the presentations delivered at the "Symposium on In-Service Lubricant and Machine Analysis, Diagnostics, and Prognostics," on December 8, 2010 in Jacksonville, Florida, sponsored by D02.CS96. In 1999, D02.CS96, In-Service Lubricant Testing and Condition Monitoring Services Industry Support, was formed to address the needs of monitoring in-service oils. This symposium showcases the progress made in the past decade and highlights the future direction of the CS96 subcommittee.

The standards developed and being developed by this subcommittee provide equipment users with a known basis for the quality of the data they are receiving which Garvey highlights by demonstrating the return-on-investments that can be achieved through proper oil condition monitoring. Toms and Wooton stress the necessity of alarm limits to properly interpret raw lubricant test data and demonstrate how remaining useful life diagnostics of machinery and fluid relies on trending.

With expanding use of alternative energy, comes new oil condition-monitoring demands. Livingstone, Ameye and Wooton address optimizing an oil condition-monitoring program specifically for wind turbines. Pigeon and Abellaneda present the impact of biofuels on lubricant dispersancy and health.

The latest laboratory and field techniques for in-service lubricant and grease analysis were presented. These papers included an alternative use of linear sweep voltammetry for diesel engine oil by Fentress, Sander and Ameye, to monitoring particles, color and water by Canty, and the latest in Fourier transfer infrared lubricant condition monitoring by Pinchuk and van de Voort. The importance of in-service grease analysis was covered by Turner.

Walsh, Barraclough, and Henning offered a historical overview of the role of wear particles in oil condition monitoring, followed by a presentation demonstrating the application of scanning electron microscopy for particle counting and classification by Herguth.

Recent developments in online oil condition monitoring sensors and their alignment with ASTM methods and practices were highlighted by Lunt.

The experiences with ASTM D02 Practices D4378 and D6224 for Turbine oils and Auxiliary Power Plant Equipment Condition Monitoring Programs by Wardlow and Ameye concluded the program.

We wish to acknowledge the prompt response and cooperation received from the authors, reviewers, and the ASTM staff to make for a successful symposium and subsequent efficient publication of this volume. The success of the Symposium and this publication are possible because of the efforts and commitments of the authors, reviewers and their companies. Thank you.

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Ray Garvey[1]

Outstanding Return on Investment When Industrial Plant Lubrication Programs are Supported by International Standards

ABSTRACT: Industrial plant lubrication programs are organized to assure in-service lubricating and hydraulic fluids are kept clean, dry, and fit for use; that these fluids are right for the applications; and that they are filled to the correct levels. These programs can yield outstanding results. For example the lubrication program at an automotive assembly plant reported more than 700 % return on investment with a two month payback period. For another example the lubrication program at a petroleum refinery is credited with reducing maintenance work orders by one-third, from 995 to 674 failure related work orders. Cost avoidance is achieved in at three principal areas: less fluid consumed, less reactive maintenance, and more deferred maintenance. Under-pinning for successful lubrication programs is the use of defined procedures and measurements. Gene Jennings, Condition Based Maintenance Coordinator for Southern Company, highlights the significant role ASTM is fulfilling for industrial lubrication programs in this statement: "Consistency in data gathering is crucial to a successful program and standardization is the foundation for consistency."

KEYWORDS: lubrication, oil analysis, onsite, payback, ROI, standardization

Introduction

One of the main worries in the minds of maintenance managers today has to be "what are my costs going to be next year and how can I possibly create a budget that meets my needs when there are so many unknowns?"

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[1] Emerson Process Management, Knoxville, TN 37932.

Symposium on In-Service Lubricant and Machine Analysis, Diagnostics, and Prognosis in Jacksonville, FL.

Cite as: Garvey, R., "Outstanding Return on Investment When Industrial Plant Lubrication Programs are Supported by International Standards," *J. ASTM Intl.*, Vol. 8, No. 6. doi:10.1520/JAI103526.

Obviously, we can no longer live with a mentality of business as usual. To be successful, maintenance managers must take aggressive steps to control costs and reduce waste. One of the easiest and most cost effective ways to do both of these is by changing the way we deal with lubricants in the plant. Several practices that are in use today, such as time based oil change, replacing used oil with new, and disposing of old oil must be eliminated. Each of these steps has the obvious cost that we account for as well as hidden costs.

To survive the changes that are coming, the practices mentioned above must be replaced with "new" procedures that incorporate the following principals:

- Lubricants will be accepted upon delivery only after third party testing.
- Lubricants will be handled and stored in a manner that reflects their value.
- Lubricants in equipment will have maintenance performed on them only when condition indicates.
- Lubricants will only be discarded when off-line filtering proves ineffective.
- Lubricants leave the plant only as a last resort.

The guiding principle behind these changes in philosophy is the fact that oil seldom fails. It often becomes contaminated and occasionally its additives are consumed. Nevertheless, the oil is still usable if the appropriate measures are taken. In many cases, simply knowing when to filter and ensuring proper filtration can bring the oil back to a condition that is better than new. This information and subsequent action can easily result in a reduction of your oil consumption to a fraction of what it is today—with the surprising benefit of dramatically increased machinery reliability.

The key components in this process are:

- Oil analysis for screening of all incoming new oil.
- Organized storage and distribution of lubricants.
- Oil analysis to determine in-service oil condition, machinery health, and filtering results.
- Filtration to return oil to a clean state.

ASTM Brings Standardization to In-Service Oil Analysis

Oil analysis instruments have long been used to test lubricants and identify problems of lubricant degradation, system contamination, and machinery wear. Until very recently there have not been any international standards for a large number of commonly used oil analysis techniques, instruments and test methods associated with in-service lubricant analysis. For example analytical ferrography and other wear particle analysis techniques have been accepted and practiced since the 1980s without published standards. This has also been the case for integrated testers commonly used in hundreds of industrial plants worldwide. Also there has not been a standard method for trend analysis and for statistically analyzing alarm limits used for industrial oil analysis.

Industry is grateful to ASTM's D02.96 Coordinating Subcommittee which sponsors task groups dedicated to bringing new standards into use covering these and other topics. There is now a wear particle analysis task group actively publishing standard guides, practices, and methods. There are also task groups for integrated testers, for FT-IR, and particle counting instruments.

In addition to standardizing practices and methods concerning oil testing instruments, the industry benefits from the ASTM D02.96 efforts to standardize in-service oil analysis techniques for alarm limit determination and trend analysis practices.

Gene Jennings, Condition Based Maintenance Coordinator for Southern Company, highlights the significant role ASTM is fulfilling for industrial lubrication programs in this statement: "Consistency in data gathering is crucial to a successful program and standardization is the foundation for consistency."

Now, more than ever before, it is important for oil analysis measurements to deliver repeatability and Reproducibility that have been validated by an impartial institution like the American Society for Testing and Materials International (ASTMI). The following are outlines of typical returns being achieved using oil analysis as part of an overall lubrication program. Almost without exception, these successes are a result of motivated technicians following best practices. An important key to widespread acceptance and application of these "best practices" is the standardization which is now well underway!

Returns From Ivestments in Lubrication Programs

Premcor Refinery Reduces Rotating Equipment Failures by 30 %[1]

Jim Crisman and John Gobert at Premcor achieved pacesetter performance through sound lubrication practices contributing to major reductions in rotating equipment failure from 995 in 2001 to 674 in 2004. If the average cost for a work order repair was ~\$6000–\$10 000 then this total cost avoidance was ~$\$2.1 \times 10^6$ to ~$\$3.2 \times 10^6$ each year beginning in 2004!

What was done at Premcor to achieve these outstanding results? Cross functional teams were organized among plant staff with cooperative participation from lubricant, filter, and test instrument suppliers Fig. 1. Oil analysis was performed by both supplier and plant staff to validate delivery of correct lubricants with acceptable cleanliness. Contamination sources were identified and eliminated. A systematic varnish formation problem was identified and corrected by making a substitution of a reciprocating compressor lubricant. Teams were assigned to identify and correct root cause by seeking to recognize and eliminate defects. These things were done by inviting productive conversations inviting advocacy not inquiry. The result was an effective process for organizational learning.

GM Saves $\$1.6 \times 10^6$ with 2 Month Payback at Assembly Plant[2]

Frank D'Arcio and Ed Bohn at a GM Assembly Plant used a viscometer and an onsite oil analyzer to discover that the wrong oil had been used in about half of the machines they sampled. Oil storage points were disorganized, dirty, and poorly marked. To correct this problem, Frank and Ed attached large tags to every oil storage point, storage compartment, and transfer container. Training

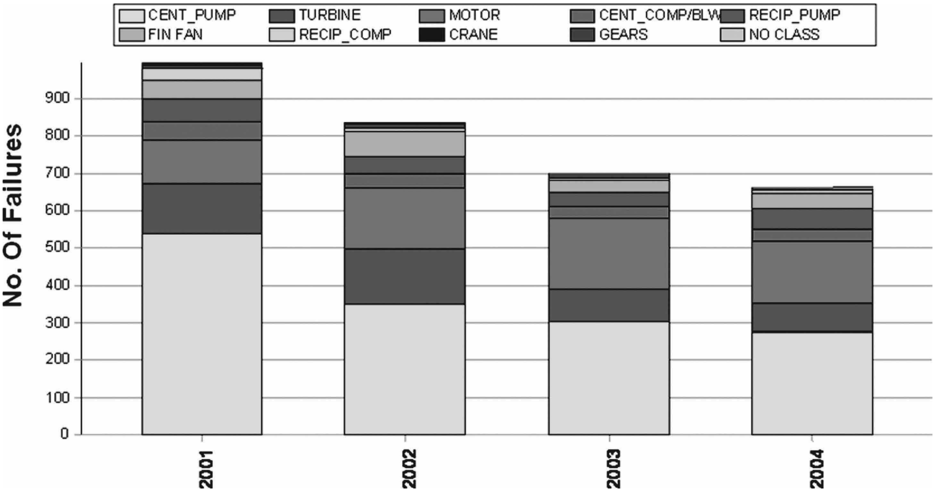

FIG. 1—*Premcor rotating equipment failure count.*

was provided for maintenance and operations. Plant staff took renewed interest in the total lubrication program for plant machinery.

The plant invested $100 800 during the first year of the program to clean up oil storage points, mark oil compartments, provide training, and purchase oil analysis equipment. The lubrication program implemented at the assembly plant was credited with $\$1.6 \times 10^6$ savings during the initial 28 months. The accounting department reported more than 700 % return on investment with a 2 month payback period.

Alcoa Saves $\$1.3 \times 10^6$ in ~18 Months[3]

The lubrication program at Alcoa in Point Comfort, Texas, has undergone significant changes including (1) contamination control, (2) oil analysis, and (3) training and operator certification. Alcoa installed a dedicated and well equipped lubricant storage facility with dedicated pumps and dispensing systems for each lubricant in the building. “There is a place for everything and everything is in its place, including filter carts.” According to Brenda Graham, senior staff reliability engineer, focus for the reliability program is on reducing costs for bearings, lubricants, and repairs of rotating equipment. Graham says, “I think last year we saved more than $800 000 overall and this year, through August, we have saved more than $500 000.” Plant Manager, Allen Ness, says, “Lubrication is a matter of discipline. How well you do the fundamentals is how well you perform.”

Packaging Corporation of America Mill in Tomahawk, WI reduces Downtime 37 and 62 %[4]

Packaging Corporation of America in Tomahawk, Wisconsin, is waging war against contamination for moisture and particulate contamination. Oil analysis, filtration, training and innovative engineering solutions put in place by the staff at this PCA mill have collectively resulted in 37 % reduction of unscheduled downtime for Paper Machine Number 4 (3.5 %–2.21 % in 2009) and 62 % reduction for Paper Machine Number 6 (4.08 %–1.56 % in 2009) according to superintendent of maintenance, Craig Slonski. Those numbers are especially remarkable considering these facts: a paper machine designed to run at 150 m/min has been revved up to nearly 500 m/min and oil life has been pushed out to 3 or 4 years.

Robin Gray, PCA mill's veteran lubricant analyst, provides the measuring stick for contamination and wear. His oil analysis tracks particle counts, wear debris, and water content. These tests validate the performance of installed desiccating air breathers as well as in-line and off-line filtration systems that are strategically positioned throughout the mill. Robin also tests greases, and he tears apart filters for filter debris testing in addition to more traditional oil analysis.

Bowater Tests Oil Rather Than Changing it[5]

Mike Lawson at the Bowater Paper Mill in Calhoun, Tennessee was asked to change the oil in two barking drum gearboxes. But rather than just change the oil, he first tested it to see if it needed to be changed. The test results showed that there was nothing wrong with the oil. It was not degraded and was actually quite clean. The cost avoided for barking drum gearbox included new oil, labor, disposal, and stocking expenses. Each gearbox contains 35 gallons of oil worth \$140. The labor required two people for 3 h or 6 man-hours costing \$240 for each gearbox. It would cost \$50 to dispose of the used oil and another \$50 to store and stock new oil. Total cost is \$480 for each gearbox. Sampling and testing cost is \$15 per sample. Return on investment is (\$480/\$15) × 100 = 3200 %!

Army Oil Analysis Program Achieves 9-to-1 Benefit from Condition Based Oil Changes[6]

The U. S. Army Oil Analysis Program replaced calendar based oil change with condition based oil change. In one study total lube oil costs of \$3 630 641 were avoided as a direct result of \$484 998 investment in the Army's oil analysis. Net savings was \$3 145 643! "In most cases the Army achieved a 9-to-1 benefit ratio for oil analysis coupled with on condition oil changes. The data also indicated that the major source of oil failure was due to contamination.

IBM Achieves Five-Times ROI Expected From Condition-Based Oil Change on Pumps[7]

Joe Meditz of IBM in Essex Junction, New York selected 135 rotary vane pumps that had been on a calendar-based oil change schedule to evaluate the potential for condition-based oil changes. He estimated that if he could extend oil life by 25 % he could save the company \$20 000 per year. A remarkable 5 years later, the oil in 100 of the pumps was still good. An added benefit from monitoring oil condition instead of performing calendar-based oil changes was the discovery of mechanical problems leading to oil contamination in 25 of the pumps. By finding and fixing these problems, Joe saved the company an additional $79 000 on the first round of testing alone.

As mentioned above, an unexpected benefit of this test was that oil analysis revealed mechanical problems and lubricant contamination problems. When these problems were remedied, the company saved 4 times the amount they did by simply extending time between oil changes. Two additional IBM plants implemented testing and validated Joe's four-to-one rule. Altogether Joe's 135 vacuum pump oil analysis project cost $12 000 and saved $99 000 in the first year, and return on investment (ROI) of 825 %.

US Air Force Application of Condition Based Oil Change[8]

In 1993 the Management and Equipment Evaluation Program located at Eglin Air Force Base, Florida evaluated an oil analyzer for use in air force ground fleet maintenance. They determined that the oil analyzer was a useful device for what they termed "bumper testing" oil quality rather than simply hanging oil based on calendar months or equipment usage (miles or hours).

The evaluation confirmed several things. First, lubricants were being discarded that still retained good anti-friction, cooling, cleaning, and corrosion prevention properties. Second, changing oil based on condition was less expensive than changing it based on time or mileage. And finally, an oil analyzer was available to perform this sampling easily in the vehicle maintenance facility itself, eliminating the necessity of shipping lubricant samples to a laboratory.

Implementing and applying onsite oil analysis by the Pacific Air Force Command's Vehicle Maintenance Manager brought benefits other than simple oil savings. "In addition to reducing waste oil generation, collateral benefits include: reduced acquisition of petroleum based lubricants, reduced labor hours in the management of waste oil, a reduction in risk assessment incurred during storage, pumping, and shipping waste oil, and an extension of engine life through improved, in-use, lubricant."

Warren Stamping Plant Saves Oil and Avoids Costly Unplanned Downtime[9]

The Warren Stamping Plant in Detroit purchased equipment for onsite oil analysis. This allowed plant personnel to evaluate samples of oil used in equipment throughout the plant and provide reports to management on the same day. The data showed which equipment needed attention due to oil contamination by water and other materials. Portable filtration equipment was also purchased to clean systems that had become contaminated. Samples were taken before and after filtering to ensure that the oil was up to their company's specifications.

At the onset of this program, press lube oil consumption was over 10 000 gallons per year. By using onsite oil analysis, consumption was reduced to approximately 1000 gallons per year with the added benefit of increased machine availability. Root causes of mechanical problems and oil contamination have been determined and eliminated.

In addition to reducing lubricant consumption, Terry Aikens, reliability engineer at Warren Stamping Plant, avoided more than $100 000 repair expense

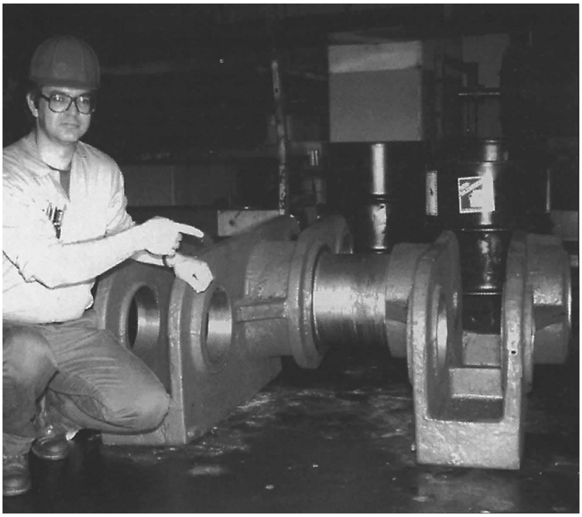

FIG. 2—*Broken rocker arm from 1000-ton press.*

and more than $\$1 \times 10^6$ of lost production when ferrous density measurements revealed problems with two different Hamilton 1000-ton presses.

One press has a cracked rocker arm (see Fig. 2) and on the other, a stud was progressively shearing. Four rocker arms like the one pictured here rotate on studs and press steel sheets into automotive body parts. "Normally we do not know there is a problem like this until the rocker breaks. When that happens there is a lot of damage, there is risk to the operators, and repairs can take several months." Oil analysis showed the problems early, repair of the sheared stud was accomplished in one shift, and repair of the cracked rocker arm was completed in 24 h.

Conclusion

A common thread through these several examples where excellent return on investment has been realized is ownership. Regardless of the industry or particular machinery involved, the success stories are all about plant staff taking direct ownership for their lubrication program. That includes procurement, storage, handling, testing, and disposal of lubricants. It includes the air filtration and oil filtration to exclude abrasive and corrosive contaminants. And it includes testing for evidence and classification of machinery wear processes revealed by in-service lubricant analysis.

These examples reinforce Drew Troyer's observation, "Every successful oil analysis program I have observed has passionate technicians performing the work. And almost without exception each includes some degree of onsite oil analysis."[10] Motivated technicians like the many named in references cited for this article are critically important to the successful implementation of high return lubrication programs.

ASTM's D02.96 coordinating subcommittee on in-service lubricant analysis is providing an excellent service to help these motivated technicians to carry their successes outside their individual plant sites and throughout their respective corporations. This important aspect is best summed up in Gene Jennings' statement, "Consistency in data gathering is crucial to a successful program and standardization is the foundation for consistency."

Endnotes

[1] Is This 'Pacesetter" Performance?" by Jim Crisman and John Gobert, Premcor Refinery, presented at Emerson Global User Exchange 2005.

[2] "GM Invests in Lube Program Upgrades" by Ed Bohn, General Motors Corporation. Machinery Lubrication, September 2001.

[3] "Life on Mars" by Paul V. Arnold, Machinery Lubrication, September-October 2010, pgs. 12–18.

[4] "Lubrication Superheros" by Paul V. Arnold, Machinery Lubrication, May-June 2010, pgs. 14–22.

[5] "The Bill is Coming Due," by Bob Martin and Ray Garvey, Lubrication and Fluid Power, November-December 2005.

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[7] "Case Histories and Cost Savings: Using In-Shop Oil Analysis for Industrial Plant Applications," by Ray Garvey. Posted to Emerson Process Management on the Machinery Health Management website at www.MHM.AssetWeb.com under Expertise/Product Support, Application Papers.

[8] "The Application of Time Resolved Dielectric Instruments to Air Force Ground Fleet Maintenance," by Stephanie Thompson. Posted to Emerson Process Management on the Machinery Health Management website at www.MHM.AssetWeb.com under Expertise/Product Support, Application Papers.

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[10] "Is Onsite Oil Analysis Right for Your Organization?" by Drew Troyer, Practicing Oil Analysis, March-April 2001.

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Allison Toms[1] *and David Wooton*[2]

Guidelines for Alarm Limits and Trend Analysis

ABSTRACT: In 1999, CS96, In-Service Lubricant Testing and Condition Monitoring Services Industry Support, was formed to address the needs of monitoring in-service oils. The standards developed by this subcommittee provide equipment customers with a known basis for the quality of the data they are receiving [Improving Used Oil Analysis Standards: Recent Efforts of ASTM D02 Subcommittee CS96, Bryan Johnson, Practicing Oil Analysis, July–Aug 2006, pp 38–41]. However, test measurements have little meaning in a condition-monitoring program if they cannot be associated with a failure mechanism of the oil or the machine. Maximum reliability of in-service machine components and fluids requires that the condition-monitoring program provide timely indications of machinery and oil performance and *remaining usable life*. In order to address these critical aspects of condition monitoring, D02.96.4, Guidelines, initiated two task forces to develop Standard Guidelines to address Alarm Limits and Trends. Reliable alarm limits for fluid and equipment characteristics are required in order to properly interpret raw lubricant test data. However, these level alarms only state how much damage has occurred. In order to generate meaningful diagnostic and prognostic information on equipment and fluid condition, the rate of change must be trended. The *predictive or forecasting* nature of condition monitoring is based on trending, which determines the remaining useful life of the component and fluid. This paper presents an overview of practical alarm limit calculations using statistical analysis of equipment and fluid condition data and trend analysis of condition data in the dynamic equipment-operating environment. Various trending techniques and formulas will be presented with their associated benefits and limitations. These limit and trend calculations may be applied to all techniques that provide numerical test results and for all types of equipment (diesel, pumps, gas turbines,

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[1] GasTOPS Inc, Pensacola, FL 32503.

[2] Wooton Consulting, Beaverdam, VA 23015.

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industrial turbines, hydraulics, etc.) whether large fleets or individual industrial machines.

KEYWORDS: alarm limits, alarm trends condition monitoring, diagnostics, equipment condition, prognostics, remaining usable life, trend analysis

Introduction

Maximum reliability of in-service machine components and fluids requires a program of condition monitoring to provide timely indications of performance and remaining usable life. Reliable alarm limits are required in order to properly interpret raw test data and generate meaningful diagnostic and prognostic direction. Physical and analytical characteristics of equipment fluids and components such as oil viscosity, oil contaminants, and wear debris are often used as condition indicators for equipment condition or performance monitoring. For reliable machine or fluid performance these parameters must lie within a small well-defined range. Outside this range, the equipment and/or fluid performance degrades until a hard limit reached, after which the machine fails. This physical limit is referred to as a “condemning limit.”

In order to predict equipment and fluid condition, the rate of change of these parameters must be trended. Level alarms only state how much damage has occurred. The *predictive or forecasting* nature of condition monitoring is based on the trending of measured parameters in order to determine the degree of damage and remaining useful life of the component or fluid.

Maintenance engineers expect condition-monitoring information to clearly and consistently indicate equipment condition. There is an expectation of a relationship between the rate-of-change of the equipment’s damage over time and its risk of failure. The data limits and trending procedures must automatically adapt to equipment usage and sampling circumstances and provide numbers that reflect equipment condition change in an incremental fashion.

Trending Interferences

The effect of operation and maintenance activities must be “tuned-out” for the most effective trending. Fluid sampling, component testing, maintenance, and filter and oil changes are rarely performed at precise intervals. These irregular, opportunistic intervals have a profound effect on measurement data and interfere with trending techniques. Effective data trending requires that these interferences be taken into account.

There are a number of events and practices that can affect the results and the trending between samplings

1. The first and most obvious is the equipment operations. Operational intensity or equipment work output can impact how quickly a component wears and how rapidly a fault progresses [1,2]. A relevant indicator of machine usage must be included in any calculations. The selected usage indicator must reflect actual machine usage. A few examples are; stop/start cycles, megawatt hours, hours of use, or fuel consumption. In close association with the operations is maintenance actions performed on

the equipment. Component, filter and oil changes impact the monitoring of machine condition as well as impact wear debris, contamination ingress quantity, and fluid condition. Maintenance events should always occur *after* a sample has been taken (or condition test has been performed). All maintenance events should be documented and taken into account during condition data interpretation. In all cases, maintenance events, if not taken into consideration, will reduce trending relevance and reliability.

2. Sampling procedures can also influence the trending. Improper or poor sampling techniques can profoundly impact quality of test data. A significant difference in the test data could trigger a false trend alarm. Examples of poor sampling techniques are:
   - Stagnant sampling.
   - Sampling after component change out.
   - Sampling after oil and/or filter changes.
   - Irregular sample intervals.
   - Sampling from the wrong location or using a variety of locations for the same piece of equipment.

   A uniform sample cannot be obtained from intermittent or standby equipment without circulating the oil to bring the equipment to operating temperatures. This means that there could be different conditions in the sample obtained from a critical location elsewhere within the system.
3. Laboratory and testing practices are another significant variable. The tools used to perform the condition monitoring tests always impact the data [1]. Analytical instrument differences impact data. It is almost impossible to have two instruments that produce exactly the same results on a test. Bias between them always exists, no matter how hard one tries to eliminate them. Trending should only be performed on results from the same make and model of test instrument. For example, trending atomic emission inductively coupled plasma (ICP) results should be from ICPs with the same sample introduction configuration, plasma energy, and preferably the same manufacturer and model.
4. Differences between testing laboratories will always show the largest bias. Whenever possible, the trending data should all be generated by the same laboratory. If a new laboratory is going to be used, overlapping tests should be performed. When multiple laboratories are utilized, a correlation program between them should be established. Documentation of any laboratory changes is important.
5. Analytical instruments with poor measurement repeatability and reproducibility will result in correspondingly poor trending. Testing repeatability should also be included with the trending studies. Inappropriate analysis techniques can hide or distort interpretational conclusions. The condition-monitoring tool chosen must provide evidence of the critical failure modes under review.
6. Equipment wear process themselves have been shown to affect the trending if not properly considered. Wear metal concentrations in oil are subject to variability.

- Filters remove the majority of debris particles greater than filter pore size. Thus a circulation fluid sample only contains new wear and small, suspended, old wear.
- Wear particle formation is "event driven," e.g., increased load or speed can result in increased wear.
- The rate of wear debris formation is not linear with time and many wear debris formations occur in bursts from a fault mechanism. Wear debris will also assist the production of more wear debris, thus accelerating the process.
- Wear metal analyses methods can have particle size limitations that should be included in the evaluations. For example, ICP metal analyses are limited to those particles below nominally 8 μm and rotary disk emission (RDE) spectrometry cannot detect below nominally 20 ppm.

7. Reservoir/sump volumes will also enter into the mix. Fluid and wear condition parameters are concentration measurements that are affected by reservoir/sump size. Varying the oil volumes in a reservoir can impact the trending analysis. For example, infrequent top ups allows the oil volume to decrease and thus concentrate the wear debris and contaminants. Alternatively, large volumes of make-up oil dilute the concentrations. Small, routine oil top-ups reduce this interference. The fluid make-up rate should be assessed as apart of the evaluation procedure.

When trending for a specific piece of equipment, one should look at the difference between the current sample and an average of a group of previous samples from that piece of equipment or a group of samples from as many similar units as possible. Basing a trend on just two data points can leave significant room for error and misjudgment. In addition, when samples cannot be taken in exact intervals, techniques should be applied that overcome these irregular intervals.

Trending Procedures

For in-service lubricant and machinery condition monitoring, there are numerous techniques to calculate trends from the very simple to the more complex [1,3]:

- Difference (delta) trend.
- Percent change trend.
- Rise-over-run.
- Cumulative trend.
- Adaptive trending.

There are advantages and disadvantages to each method.

- Difference (delta) trend: The difference trend (T) is between sequential samples and is the current sample (C) value minus the previous sample (P) value

$$T = C - P \tag{1}$$

The difference trend procedure is the easiest to calculate. As disadvantages, it does not account for machine usage and is of little value for the determination of the rate of wear or oil deterioration. This is the traditional "eyeball-it" method where gross changes can be noted. This formula does not factor in the equipment duty cycle or operating conditions and is a poor indicator of equipment or fluid condition.

- Percent change trend: The % change trend is the current sample minus the previous sample, divided by the current sample value, times 100

$$T = (C - P)/C * 100 \tag{2}$$

The % change since the last sample can be a better indication of trend but still does not account for equipment usage or duty cycle. This calculation can generate ambiguous numbers for fractional data since an increase from 0.1 to 1 is the same % change as from 10 to 100. Percent change is only suggested for large trend changes (for example trending intervals that yield *C* or *P* of 100) and where the equipment is used continuously with rigorously constant sampling interval.

- Rise-over-run: The rise-over-run trend is the current sample minus the previous sample, divided by the usage metric (*H*), times the standard sample interval (SSI)

$$T = [(C - P)/H] \times SSI \tag{3}$$

The rise-over-run trend calculation factors in the equipment usage, Fig. 1. It can be effective for continuous duty and intermittent duty equipment, as long as the sampling interval remains constant and the samples are taken as close to the correct time as possible.

The optimum sampling intervals are derived from known failure profile data. The sampling interval is a fraction of the time between initiation of a critical failure mode and equipment failure. In general, sample intervals should be short enough to provide at least two samples

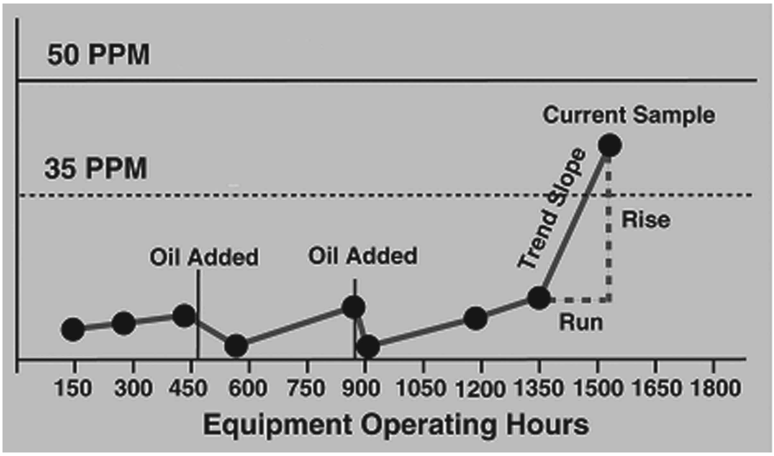

FIG. 1—*Trend plot demonstrating rise-over-run.*

prior to failure. The interval should be defined to account for the shortest critical failure mode. Samples not taken at the correct intervals (very short or very long) will generate ambiguous results due to multiplication factor of this standard sampling interval.

- Cumulative trend: The cumulative trend is the sum of previous and current sample

$$T = \sum\nolimits_n C \tag{4}$$

  The cumulative trend plot provides a quick indication as to whether a series of data points are maintaining a linear trend slope or are beginning to deviate. The cumulative trend plot is most effective when equipment is used continuously. It also works well for real-time data where there is a high sample rate, Fig. 2, such as on-line sensors.

- Adaptive trending: In practice, there is considerable difference between the standard sample interval and the actual sample interval. Because of intermittent equipment usage and/or irregular sampling and maintenance, the above trending techniques are incomplete for sampled condition monitoring. It is not practical to expect all equipment to have equal duty cycles, be sampled at specific, uniform intervals, or be maintained at regular intervals. Maintenance and sampling operations are generally opportunistic in order to meet production goals.

A solution can be to utilize an adaptive trend calculation that can compensate for varying sample intervals and reasonable makeup oil additions. When the sample interval is between 0.5 and 1.5 times the standard interval, a conventional rise-over-run trend formula provides reliable trend data. When the actual sample interval is shorter than half the standard interval or longer than

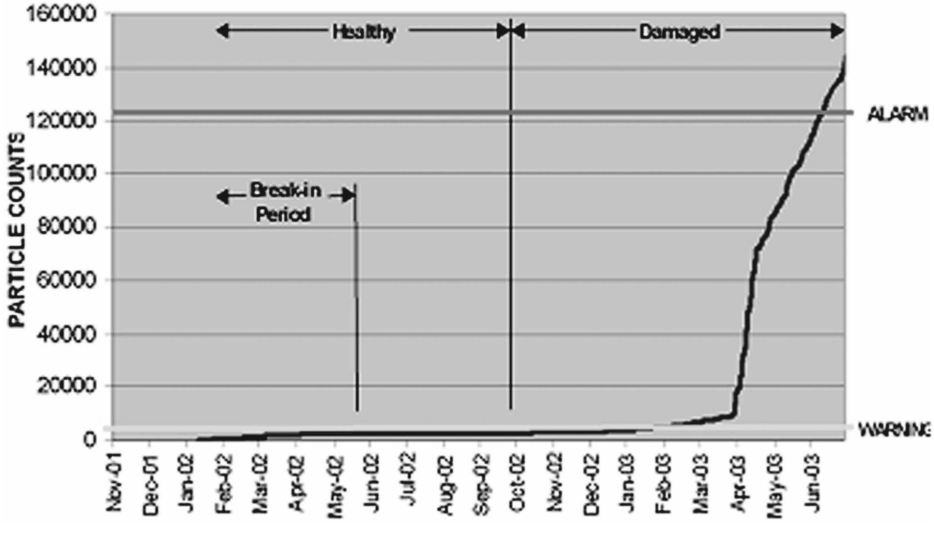

FIG. 2—*Cumulative trend plot of real-time sensor data.*

1.5 times the standard interval, the rise-over-run formula no longer provides satisfactory results. In these cases, a predicted last sample value can be determined from a linear regression that "predicts" the last sample value based on the standard interval, Fig. 3.

The following five rules can be used to help select the most appropriate formula based on sample interval and oil change information.

1. For routine samples *taken at 0.5 to 1.5 times the standard sample interval* and the oil has not been changed since the previous sample, it is recommended to use the *rise-over-run*.
2. If the sample *interval is smaller than 0.5 times the standard interval* and there are sufficient samples, it is recommended to use a linear regression (1) or cubic spline value calculation to predict the prior sample value (*PP*), one standard interval prior to the current sample (*C*). One can calculate the test data trend (*T*) for the current sample (*C*) using *a rise-over-run* equation and the standard sample interval (*SSI*).

   (1) A linear regression over 10 previous samples is suggested

$$T = \frac{C - PP}{SSI} * SSI \tag{5}$$

3. If the sample *interval is greater than 1.5 times the standard interval* and there are sufficient samples, it is recommended to use a linear regression or, if the data is nonlinear as it often is near the end-of-life, a cubic spline calculation [4] to predict the prior sample value (*PP*), one standard interval prior to the current sample (*C*) as above. One can calculate the test

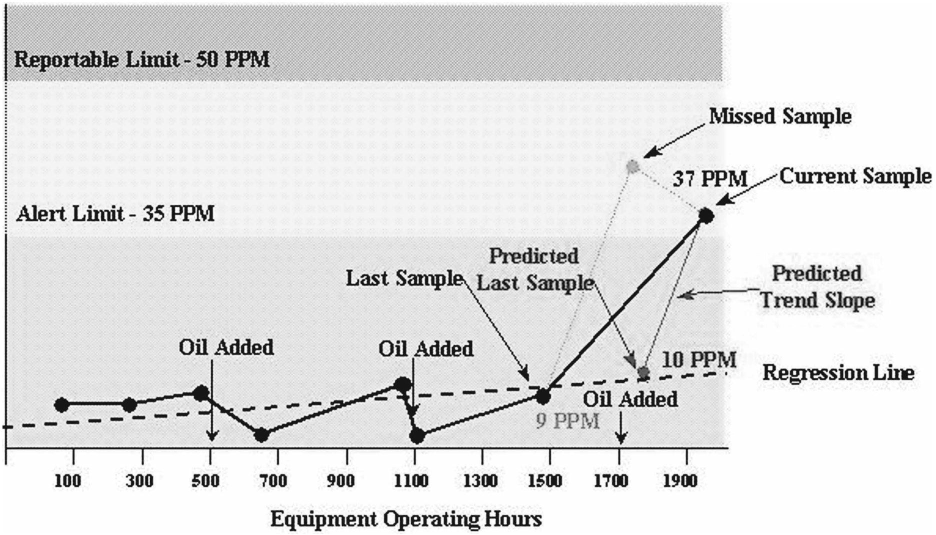

FIG. 3—*Adaptive trend plot when sample interval is greater than 1.5 times the standard interval.*

data trend (T) for the current sample (C) using *a rise-over-run* equation with the standard interval (SSI) and Eq 5.

4. If the sample is *the first after an oil change* or if there are insufficient samples since the last oil change to perform a linear regression calculation, it is recommended to calculate the trend for the current sample **(C)** using a *rise-over-run* equation (Eq 5), the time-on-oil interval **(OI)** and the standard sample interval **(SSI)**. When there is no prior sample, it is recommended to use the average data of all samples taken immediately after an oil change for that equipment type (Eq 6)

$$T = \frac{C - Avg}{OI} * SSI \tag{6}$$

5. Alternatively, if an *oil change occurs immediately* before the current sample is drawn, destroying the history of an abnormal trend, it is recommended to trend data before the oil change to estimate or predict a usable trend. It should be noted that destroyed trend data can never be fully recovered.

Predictive Forecasting

The second part of any trending operation is to understand or predict how the behavior of the fluid or machine will progress into the future. One wants to know how long the fluid or machine is "predicted" to last, based on how it has behaved in the past. The formulas for a prediction model can be developed based on studying the single variables or polynomials representing on all the data acquired. The prediction is a regression equation that connects the past data with the future. Regression analysis allows one to develop these model equations and the coefficients equation that can be used to predict the future.

There are many mathematical models that can be used to develop these predicting equations. The extrapolation of a trend plot or least-square calculated line will yield an extension of the data. However this approach may not actually account for what is happening within the last several samples. By nature, these equations take into consideration all the data within a range of measurements yielding an averaging to the data—beginning to end. This may not be what one wants to study. One should "weight" the last several results to learn where they are taking the trend.

This approach can be accomplished by applying a recursive mathematical approach similar to Kalman equations [5,6] or by efficient organization of a mathematical regression search calculation for optimum structure of a developed mathematical model such as that used in the group method of data handling (GMDH) [7]. These techniques employ matrix or polynomial mathematics to develop processing equations that are based upon previously determined results for the system (or equipment) being studied. This process is like using the previous samples to develop a calibration model of the data flow, thus allowing a calculation or prediction of what the next sample's results should be. These new equations then become adaptive and predictive in nature because they are constantly changing throughout the evolution of the system's (or equipment's) life span. Essentially an equation of the results is designed toward estimating the flow of the data for the life span and allows improved end of life prediction.

Limits Setting Procedures

With the trending approach established, the equipment maintenance engineer's next objective is to establish guidelines for limits or extremes to which the results may progress to before requiring maintenance actions to be taken [1,8–10].

The calculation of alarm limits should initially be developed based on the ideal conditions and limitations from a sample population of condition data. It is understood that ideal conditions are not often met. The scientific basis of this method has been developed from large equipment fleets and can be applied to smaller fleets with longer history or with expert knowledge of condition monitoring. Extension of these alarms outside the ideal conditions can only be achieved after extensive and proper evaluation of the data and the equipment's operation are achieved. The general assumptions to follow in setting these limits and alarms are:

- The equipment process is a closed loop system whereby test measurements are only affected by operations, maintenance or the onset of a failure mode.
- The equipment fleet is a well-maintained population of like machines.
- The equipment in the sample population operate in the same environment, under the same duty cycle and load conditions and have the same mechanical specifications, e.g., oil capacity.
- A nominal sample interval has been established that accounts for the critical failure modes with at least two samples between failure mode initiation and its terminal phase.
- The sample population should cover at least one overhaul interval or in the case of a large fleet, should cover all operational phases from new to overhaul.
- Each established failure indicator is unambiguous in its prediction and free from interference.
- The sample population includes a complete range of failure indication levels from problem initiation through component failure for each critical failure mode.
- Nominal condition data are expected to fall *within two standard deviations* of the mean or represent about 94 % of all samples taken.
- Abnormal condition data are expected to fall *outside two standard deviations* of the mean and represent about 6 % of all samples taken.

Alarm Limits Procedure

The application of statistical analysis on the conditional data for calculating the practical alarms is a recommended practice. The alarm limits that indicate physical characteristics, i.e., state of equipment fluids and components are often initially established by the original equipment manufacturer (OEM) guidelines. However, these physical characteristics alarms should be reviewed periodically throughout the trending process—for in many cases they were established before extensive operation of the equipment in the field was completed.

It does not matter how different or complex the failure modes are, the process for developing limits becomes the same. The failure mode is initiated/caused by some factor (age, lack of lubrication, contaminant, etc). Each factor has a definable path to failure. The failure is manifested in the data through its signature. The failure mode will need to be tracked by a given measurement test that reflects its behavior.

Practical alarm limits are calculated based on statistical population distributions of the historical samples:

- 1-Sigma limits: 68.27 % of samples will fall within one standard deviation of the population mean.
- 2-Sigma limits: 94.45 % of samples will fall within two standard deviation of the population mean.
- 3-Sigma limits: 99.73 % of samples will fall within three standard deviation of the population mean.
- 4-Sigma limits: 99.99 % of samples will fall within four standard deviation of the population mean.

Most sample data from a properly designed and maintained equipment population can be expected to fall within the 2-sigma limits (~94 %), with the remaining ~6 % likely to represent a range of abnormal to failure conditions. Therefore, alarm limits of 2-sigma can be used to reliably detect and diagnose equipment and fluid problems before failure occurs.

Defining the practical limits can be achieved by following a simple series of steps. The first four steps aid to defining the alarm limits with their action levels. Steps 5 and 6 define the population of samples that are being used in step 7 and 8 to define the distribution ranges for the alarm limits. Step 9 is the validation of the prior steps.

Step 1—Determine the critical failure modes. Generally these failure modes result in process shutdown or individual component failure.

Step 2—A fault will need to have a definable test parameter that is measurable for the trending process and reflects the cause of the failure mode. Any failure mode may have more than one test measurement; however, the parameters must be reliable. If multiple parameters have been determined, define the primary and secondary ones. The fault is confirmed when the prime indicator and the related indicators are all present.

Step 3—Establish a series of warning levels that are based upon severity of the indicated condition. These indicators are used to define the condition of the equipment or fluid. For example:

Alert level: First warning of developing problem
Reportable level: Fault is at a serious stage

Moderate trend: Fault has progressed higher; action should be required
Rapid trend: Fault is increasing rapidly; action is required.

Step 4—Define the criteria for each alarm status category
Alert level: 2-Sigma limits: (encompasses 94.45 % of samples)
Reportable level: 4-Sigma limits (encompasses 99.99 % of samples)
Moderate trend: 60 % of the alert level (change in one sample interval)
Rapid trend: 90 % of the alert level (change in one sample interval).

Step 5—Define the equipment that will be using the fault just established. Candidates must be segregated by mechanical and operational characteristics including make, model, duty cycle, and sump capacity. In addition, remove any data from components with differing characteristics such as different metallurgy, different fuel consumption, etc.

Step 6—From the candidate histories, pull together the relevant condition data. For best performance select all available samples. For large equipment fleets, select ten [10] or more consecutive samples from each machine in the population. Ensure each sample population contains as much data as possible. It is recommended to have several hundred samples to be properly representative of the failure mode. *Do not choose random samples*. Randomly picked samples do not always reflect the failure modes and usually generate unreliable limits.

Large fleets are not the norm for many applications. Limits can be set with these smaller populations (e.g., 10 samples); however, these limits should be adjusted through experience with similar equipment.

Step 7—Since all the limits are based on a statistical calculation from the mean values, the mean and standard deviation for each condition indicator (measurement) should be calculated. A normal distribution is typically desirable. The type of distribution can be determined by plotting a frequency distribution of the data (nominal and failure). A “normal distribution” is when a well-defined bell curve is present. A low-flattened or multimodal pattern in the distribution generally indicates the presence of more than one failure mode. In this case, the population should be culled of samples containing the interfering failure mode and the statistical analysis recalculated.

Note a high proportion of samples with abnormal (failure) results will raise the mean and standard deviation values, generating higher alarm limits. In this case, the frequency distribution is broad and flat or may have a distinct curve at the high end. To overcome this problem, the samples with *greater than the average plus 5 or 6 standard deviations* should be culled from the population. The recalculated mean and standard deviation on the remaining samples usually provide improved limits.

Step 8—From the average and standard deviation data for each test parameter, calculate a series of tentative limits based on the established formulas; for example:
Alert level: Mean + 2 standard deviations
Reportable level: Mean + 4 standard deviations

Moderate trend: 60 % of the alert level
Rapid trend: 90 % of the alert level.

Step 9—Validate the tentative limits by reexamining the historical data for each equipment problem represented in the test population. Compare results against:

1. The original recommendations for the sample to determine correlation. Look for valid instances of agreement and disagreement.
2. Look for any equipment or fluid problems that were repaired to determine viability of the new alarm limits.
3. Look for false positive when a "no problem found" exists to determine if the new alarm is too sensitive.

Note: Avoid the trap of requiring a 100 % correlation between the new limit performance and previous limits or recommendations. Sample recommendations are based on data interpretation that includes much more than limits. The purpose of this step is to find gross discrepancies. The validation evaluation should show a minimum of false positives and each one should be readily explainable. If there are too many false positive alarms the limits are *set too low*. False positives can also result when the critical failure was not adequately represented in the population or the sample interval is too long. Failure of equipment in the absence of an alarm indicates the limit is *set too high* or the measurement tool was not proper. This is also a problem if the limits were calculated from a population where the critical failure modes were *over represented* in comparison to the normal samples.

Conclusion

Establishing proper trending and limit alarm levels can be a valuable asset for interpretation of test results to reflect the equipment's operation. When achieved, these level and trend alarms can assist the maintainer with equipment reliability control and improvement.

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Optimizing a Wind Turbine Oil Condition Monitoring Program

ABSTRACT: The logistics and expense of obtaining oil samples from wind farms are among the conditions that make it challenging to establish a valuable condition monitoring program. Optimizing the oil condition monitoring program for a wind turbine is a critical step towards achieving forecasted ROI for the wind farm. To advance the current oil condition monitoring practices for most wind turbines, 150 identical wind turbine systems in the field for up to four years were studied. Several oil analysis tests were performed to understand the depletion of additives and changes in fluid chemistry. Basic statistical analysis was obtained on the data set to identify correlations in the data. The result of the analysis was a better understanding of the wind turbine oil's formulation and the relationship to fluid degradation and gearbox condition. The statistical methodology that we performed can be applied to other wind turbine farms to improve the knowledge derived from their oil analysis tests, both for gear as well hydraulic oils. In addition, other innovative tests were suggested which may augment a current oil analysis test slate to improve the value of their program.

KEYWORDS: wind turbine, condition monitoring, additive depletion, statistical analysis

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Introduction

Wind turbines have received much attention of late due to lower than expected reliability and higher than forecasted maintenance and operational costs. Although there are many factors that contribute to this phenomena, one wind turbine component in particular and a recipient of this attention from lubrication engineers is gearbox reliability. Although the failure rate of the gearbox is relatively low, when it does fail it is extremely costly for the wind farm operator. The gearbox is the most expensive component in a wind turbine and replacing it involves the use of up to two expensive cranes and considerable manpower. Although there are many factors involved with the reliability of a wind turbine gearbox, lubrication plays a fundamental role. Like in most other lubricant applications, oil condition monitoring is an integral part of trending the condition of the fluid and gearbox.

There are several wind turbine components that require lubrication, including the main bearing, pitch adjustment, hydraulics, yaw bearing, yaw gear, generator bearing, and main gearbox. This paper will only focus on the oiled components (gearbox and hydraulic system), since the other components are typically lubricated with grease and involve another set of challenges to set up a condition monitoring program.

Unfortunately, establishing an effective oil condition monitoring in a wind turbine is more complex than many other applications because of sampling frequency, equipment access, formulation chemistries, and a wide range of component original equipment manufacturers (OEM).

Sampling Frequency

Maintenance is an expensive activity to schedule for wind turbines due to their remote locations and the physical challenges of accessing the nacelle. There is a general trend in the industry to minimize maintenance activities and increase the time interval between maintenance events. Typically, maintenance activities are scheduled every 6 to 12 months, depending upon the requirements of the OEM. There are some OEMs that are looking at extending this even further to 18 months. Oil sampling is scheduled during this maintenance activity, so increasing this time interval has a direct impact on the amount of data that is generated, as well as the chance of making the right decisions at the right time. It is conceivable that the wind turbine oil that lasts for 6 years may only have 4 in-service oil analysis data points. This low amount of data makes trending challenging and lowers the potential value of an oil condition monitoring program [1]. By establishing multiple data points, lubrication professionals would be able to discern accumulated patterns and/or identify acute events occurring within a specific lubricating oil system, or per type of equipment. At any point in a component's life, conditions may exist that will shorten the life if left unattended. The promptness with which these conditions are corrected directly affects the component life. The longer test interval has excluded all of these advantages of condition monitoring.

Drawing Conclusions From Insufficient Amounts of Data

It is well known that water decreases the life of bearings and can be a cause or catalyst to micropitting [2]. Research on failed wind turbine gearbox components has shown that micropitting has been present and is considered a primary failure mode, responsible for several secondary failure modes [2]. Is water a problem in wind turbines? There does not seem to be a uniform consensus. Many oil analysis laboratories with thousands of data points suggest that water is not a problem. However, the frequency of attaining the samples from the wind turbine is between 6 to 12 months. Not surprisingly, the time that maintenance activities are planned is in traditionally drier, less stormy months. Who would want to go up-tower in stormy weather if there was a choice?

A wind farm that concluded that water was not a problem due to their oil analysis results, decided to test a few of their units on a more regular schedule to validate their assumptions. Sample data was collected on four wind turbines over a 6 month period of time.

It becomes evident when viewing the multiple data points in Fig. 1 that there is a relationship between the water levels of all four wind turbines. The common trending of water levels points to environmental conditions that are commonly impacting moisture levels in all of the gearboxes.

Figure 2 is a graph of the precipitation for the region on top of the water analysis. The times when sample results show higher water levels appear to coincide with precipitation just before the sample is analyzed. This suggests that there is moisture getting into the gearboxes from precipitation. However, the biggest finding was that the elevated moisture levels were not evident when the wind farm only looked at annual oil analysis results, pinpointing one of the largest oil analysis challenges in the wind turbine market.

Wide Range of Oil Formulations

The wind turbine market is a relatively new market compared to most other industrial lubrication markets. One unscientific way of evaluating the maturity of a market is to look at the chemistry of lubricant technologies that are being offered. With wind turbines, we see a wide range of formulation chemistries, e.g., additives and base oils, all being offered to the same gearbox—an indication that a consensus of the lubricant behavior is still being developed.

The various base oils that are commercially available in current product formulations include:

- Mineral
- Poly-alpha-olefin (PAO)
- Polyalkylated glycols (PAG)
- Esters.

Table 1 highlights the wide range of formulation chemistries as shown by the differences in the base oils used, additive metal concentrations, acid number from formulation additives, and antioxidants.

With such a wide range of base oil and additive technologies in use, it makes it much more challenging to apply a common set of condition monitoring rules that are relevant to all of the lubricant formulations in use.

Various Component Manufacturers

Globally there are over 60 wind turbine manufacturers and gearbox manufacturers, and yearly new players are added mainly in China. Each component

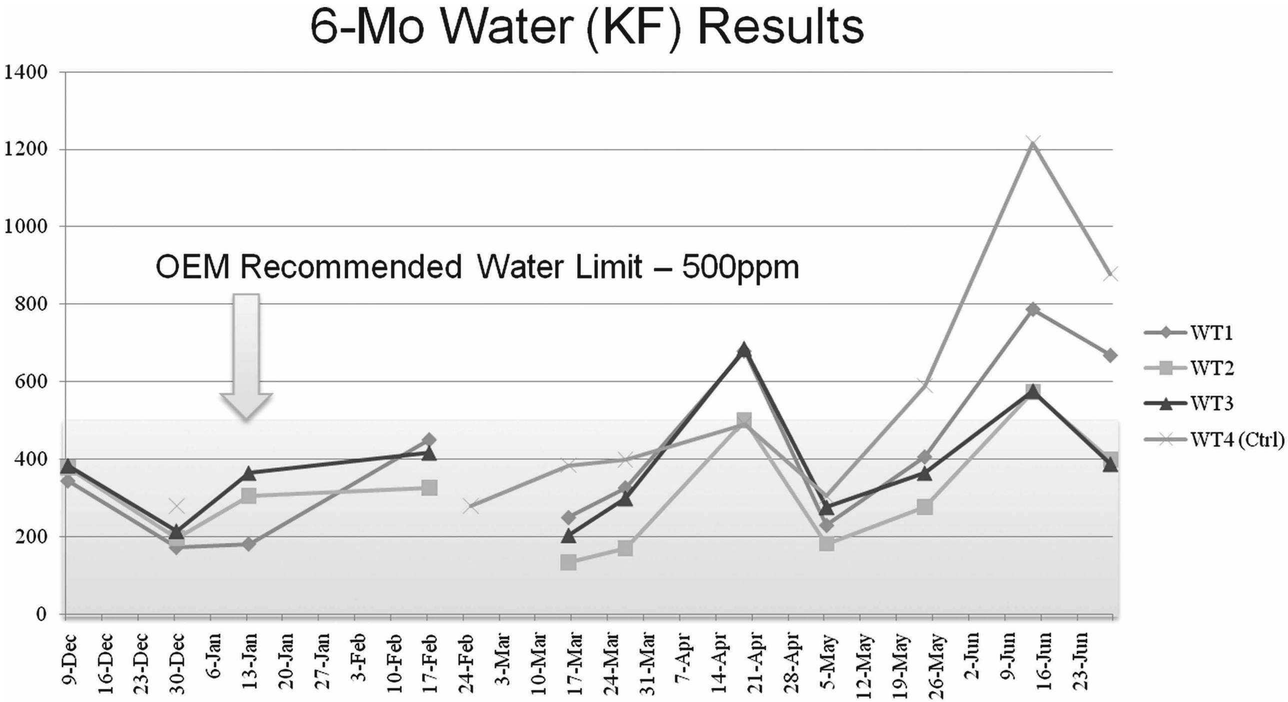


FIG. 1—*Moisture levels in four wind turbine gearboxes over 6 months.*

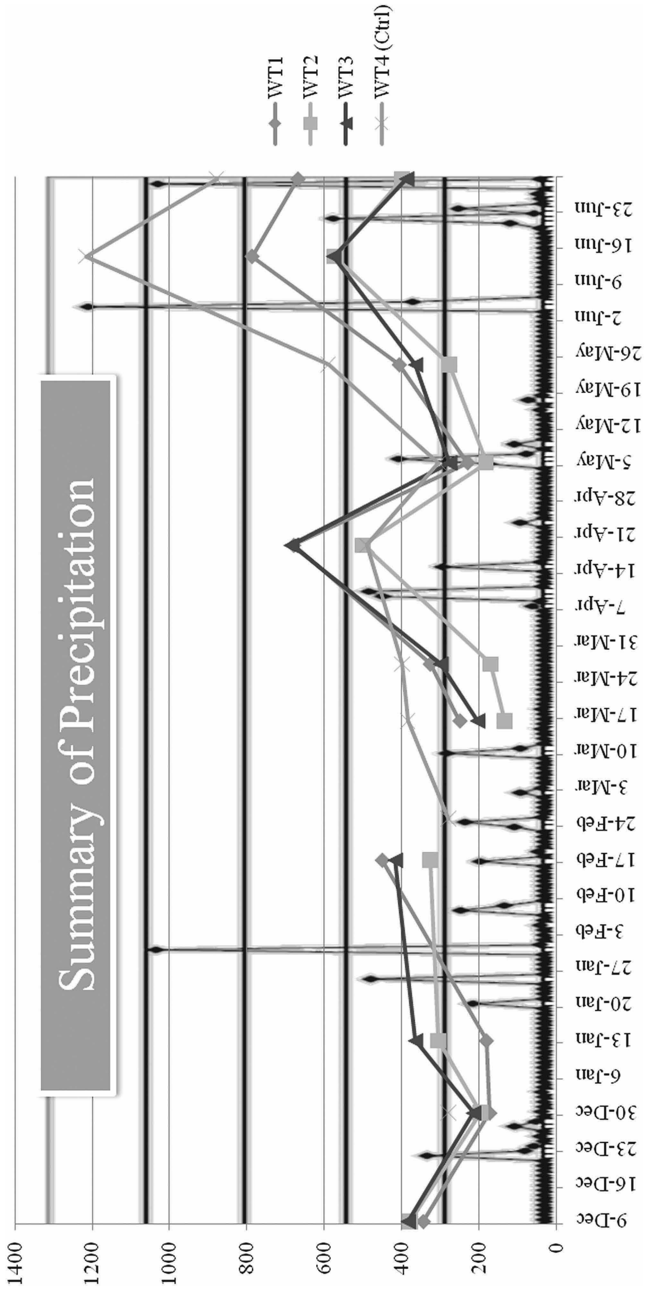


FIG. 2—*Moisture levels in four wind turbine gearboxes over 6 months compared to precipitation.*

TABLE 1—*An example of formulation chemistries in commercially available wind turbine oils.*

Oil Type	API Gp.	Ca	Mg	P	Mo	AN	Aminic	Phenolic
Gear oil 1	I	ND	ND	200	0	0.42	no	yes
Gear oil 2	IV	ND	ND	253	0	0.59	no	no
Gear oil 3	IV	ND	ND	338	0	1.01	no	no
Gear oil 4	IV	ND	ND	180	0	0.57	no	no
Gear oil 5	V	ND	ND	382	0	0.63	no	yes
Gear oil 6	I	11	ND	174	0	0.54	no	yes
Gear oil 7	IV	13	ND	177	0	0.58	yes	no
Gear oil 8	V	ND	ND	273	0	1.37	no	yes
Gear oil 9	I	ND	1435	1193	1042	3.21	no	no
Gear oil 10	I	ND	ND	171	0	0.53	no	no
Gear oil 11	IV	1986	ND	339	1176	1.46	yes	no
Gear oil 12	IV	ND	ND	420	0	0.91	no	no
Gear oil 13	V	51	ND	2484	0	0.92	yes	yes
Gear oil 14	IV	ND	ND	446	0	0.99	no	no
Gear oil 15	I	ND	ND	241	0	0.67	no	no
Gear oil 16	IV	12	1530	1249	1291	3.22	no	no

manufacturer may have their own testing standards and approval processes. Also, each gearbox component may have different materials of construction, design criteria, and lubrication tolerances which all play into understanding the data from an oil condition monitoring program. Table 2 lists some of the manufacturers of wind turbines and components.

TABLE 2—*Wind turbine and component manufacturers.*

Wind Turbine OEMs	Wind Turbine Component OEMs
Acciona	Winernergy/Flender
Clipper	Eickhoff
Ecotecnia	Jahnel-Kestermann
Gamesa	Hansen
GE Wind	Dorstener
Lagerwey	Lohmann and Stolterfoht
Mitsubishi	MAAG
Nordex	Metso
RE Power	Cincinnati Gear
Siemens Wind	Nanjing Gear
Sulzon	GE Transportation
Vestas	Bosch-Rexroth
Goldwind	David Brown

Oil Analysis Data

Statistical analysis was obtained on a data set from 150 wind turbines, all located in the northern United States. The wind turbines were all from the same manufacturer, although different gearbox manufacturers are included in this population. All of the gear and hydraulic oils used at this wind farm are the same type from the same manufacturer. The sample interval was between 6 and 12 months per turbine with between 2 to 4 years worth of analytical data from each unit.

Oil analysis was performed on the new wind turbine oil sample to better understand its additive chemistry and oil properties. The following tests were performed to gather this information:

- Elemental analysis by ICP (D5185)
- Water (D6304 Procedure C)
- ISO particle count (ISO 4406-99) pore blockage
- ISO particle count (ISO 4406-99) laser
- Viscosity at 40C (D445)
- Membrane patch colorimetry
- Linear sweep voltammetry (D6971) (green and blue solvent)
- FTIR (custom interpretation)
- ^{31}P - nuclear magnetic resonance
- Total acid number (D664)
- Total base number (D2896)
- Foam (D892)
- Demulsibility (D1401).

Based on this analysis, identification of several of the additives in the lubricant's formulation was achieved, as summarized in Table 3.

A series of statistical analysis methods were performed on the data set including principle component analysis, principle factor analysis, and agglomerative hierarchical clustering [3]. The method that proved most useful with this data set was principle component analysis, specifically the Pearson correlation data within this analysis set.

Table 4 lists some of the oil properties that were analyzed. The components that had extremely low values were discounted.

Product Formulation Information

The Pearson correlations showed a positive relationship between total acid number and the additive metals phosphorus, zinc, and molybdenum. This is illustrated in Fig. 3. This correlation suggests that these additives components are acidic in nature and are contributing to an elevated acid number in the final formulated product.

In Fig. 4, a strong relationship (0.9 correlation) is noted between zinc, phosphorus, and molybdenum. The correlation between zinc and magnesium was evident; however much lower. This suggests that zinc, molybdenum, and phosphorus are part of the same additive package/component.

It can be speculated that this additive package has a viscosity that is higher than the oil's 320 cSt at 40°C due to the strong correlation between zinc,

TABLE 3—*Components in the new wind turbine oil.*

Lubricant Component	Function
Polyalpha olefin	Base oil
Magnesium salicylate	Contributes to TBN, designed to neutralize acidic by-products in the oil from oxidation and acts as a detergent.
Overbased zinc dialkyl dithiophosphate (ZDDP)	Antiwear agent and secondary antioxidant
Neutral zinc dialkyl dithiophosphate (ZDDP)	Antiwear agent and secondary antioxidant
Phenol	Primary antioxidant
Molydithiocarbamate	Moly functions as both an antiwear and extreme pressure additive. It is also known to behave as a secondary antioxidant.
Phosphite	Antiwear additive with antioxidant characteristics
Alkyl phosphate	Antiwear additive with antioxidant characteristics
Polysulfide	Extreme pressure additive
Phenate	Contributes to TBN and detergent with some antioxidant properties.
Synthetic ester	Base oil modifier

molybdenum, and phosphorus. This is illustrated in Fig. 5 showing the correlation between viscosity and additive metals.

Changes in the In-Service Fluids

Correlating the oil's properties to wind turbine operating hours will result in valuable information related to the lubricant aging process. This may be useful in targeting key properties of the oil to be monitored with individual trends, as well establishing their condemning limits [4]. The properties that have good correlation to wind turbine operating hours as displayed in Fig. 6 are:

- ISO particle count—It is reasonable to expect to see particle counts increasing over time as dirt accumulates and wear particles are generated. This may signal a requirement for more attention on the gear oil's filtration system so that the fluid is maintained at its same ISO code throughout its service life.
- Oxidation and nitration—The oxidation and nitration bands in the Fourier transform infrared (FTIR) analysis showed a correlation to machine hours, indicating fluid degradation.
- Iron—The only wear metal that had a high correlation to time was iron. This is a key wear metal to trend in a condition monitoring program and earlier literature and research has proven the relationship of this parameter to the health of the gearbox.
- Color—The FTIR soot band is not actually measuring soot in these samples, but color changes and particle content increasing within the fluid

TABLE 4—*Summary of data points and oil properties.*

Variable	Minimum	Maximum	Mean
Fe	0.0	361.0	46.3
Cr	0.0	23.0	0.1
Pb	0.0	60.0	3.0
Cu	0.0	52.0	9.1
Sn	0.0	17.0	1.4
Al	0.0	54.0	16.5
Ni	0.0	7.0	0.0
Ag	0.0	4.0	0.0
Si	1.0	71.0	14.6
B	0.0	3.0	0.0
Na	0.0	87.0	6.2
Mg	45.0	1513.0	717.6
Ca	0.0	274.0	14.3
Ba	0.0	39.0	0.2
P	0.0	2406.0	1393.8
Zn	0.0	2212.0	1390.3
Mo	28.0	1611.0	810.8
Ti	0.0	26.0	0.2
V	0.0	22.0	0.2
K	0.0	25.0	2.4
Viscosity @ 40°C	199.6	408.0	305.2
TAN	1.1	3.8	2.1
FTIR oxidation	3.0	30.0	11.3

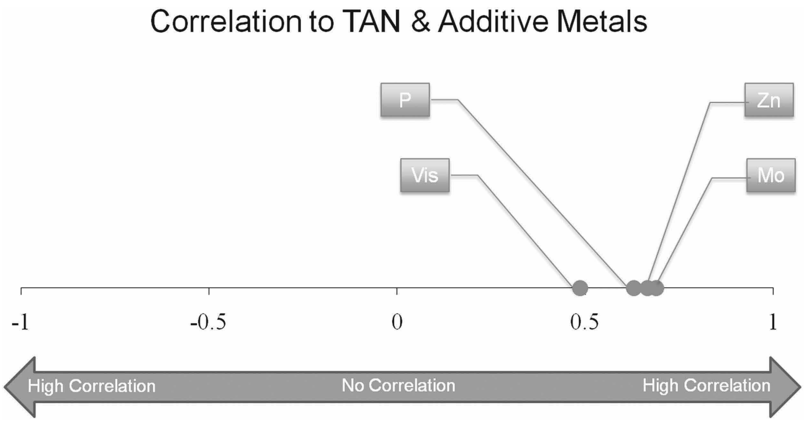


FIG. 3—*Correlation between TAN and additive metals.*

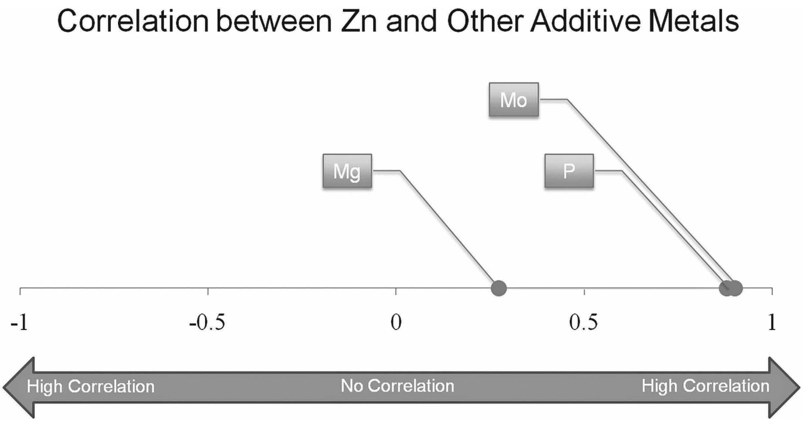


FIG. 4—*Correlation between zinc and other additive metals.*

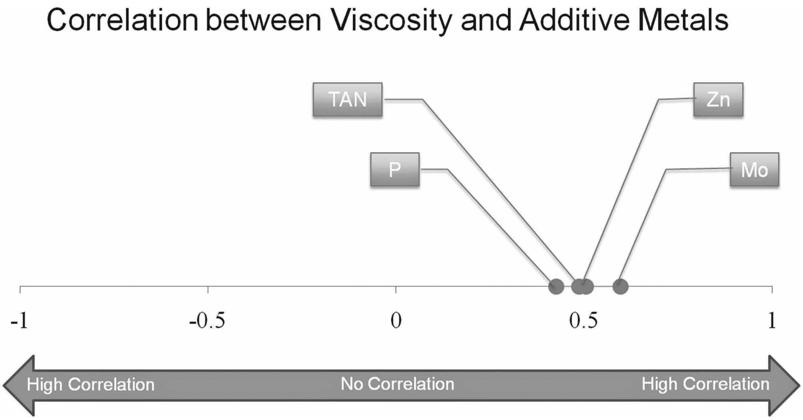


FIG. 5—*Correlation between viscosity and additive metals.*

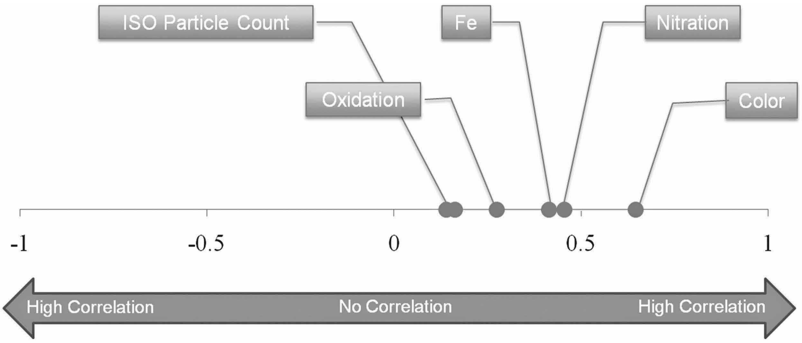


FIG. 6—*Correlation between oil parameters and wind turbine operating hours.*

from oxidation. It is intuitive to believe that the fluid will get darker as it oxidizes and ages and this data correlation confirms one's intuition. With sufficient data, color may be a basic screening tool to approximate the age of the fluid.

It is also interesting to note what oil properties did not have a strong correlation to the lubricant operating hours. It was initially surprising that additive metals did not have a strong correlation to the wind turbine's operating hours. Experience by the authors when looking at individual wind turbines showed a consistent downward trend in additive metal levels as the fluid aged. This trend is visible in Fig. 7. This investigation went further by plotting all of the "new" oil values (machine hours equals 0) from the 150 sites. The levels of Mg, P, Zn, and Mo are graphed in Fig. 8. One can clearly see in this graph that the starting values of these additive components have a very wide range of fluctuation. Without further normalizing done to this data range, it is evident why the statistical analysis showed no correlation between the varying levels of additive metals and machine operating hours. These data pointed to potential quality control issues that the wind farm had with either receiving a consistent blend of new wind turbine oil or their storage practices. It recommends the implementation of a proper quality control program for the incoming fluid.

The final correlation that was studied focused on iron, which in the previous analysis showed a strong correlation with wind turbine operating hours and is a key component in the wind turbine gearbox. The iron correlation was illustrated in Fig. 9, which showed a negative correlation between zinc, molybdenum, phosphorus, total acid number, and magnesium, in that order. These data suggest that wear occurs more frequently in a wind turbine gearbox with the depletion of these additive metals. It is important to note that the depletion of the additive metals is not necessarily related to when the additives were consumed. The additive metal levels drop in the fluid because they are no longer soluble in the fluid and measurable by the inductively coupled plasma atomic

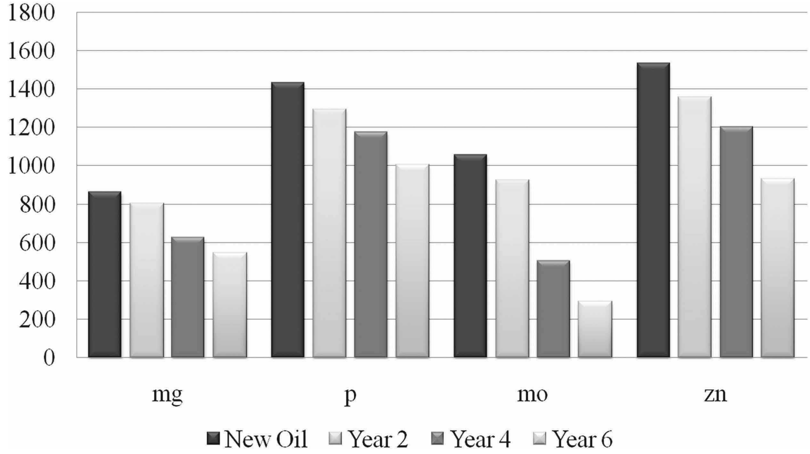


FIG. 7—*The trending of additive metals in wind turbines over 6 years.*

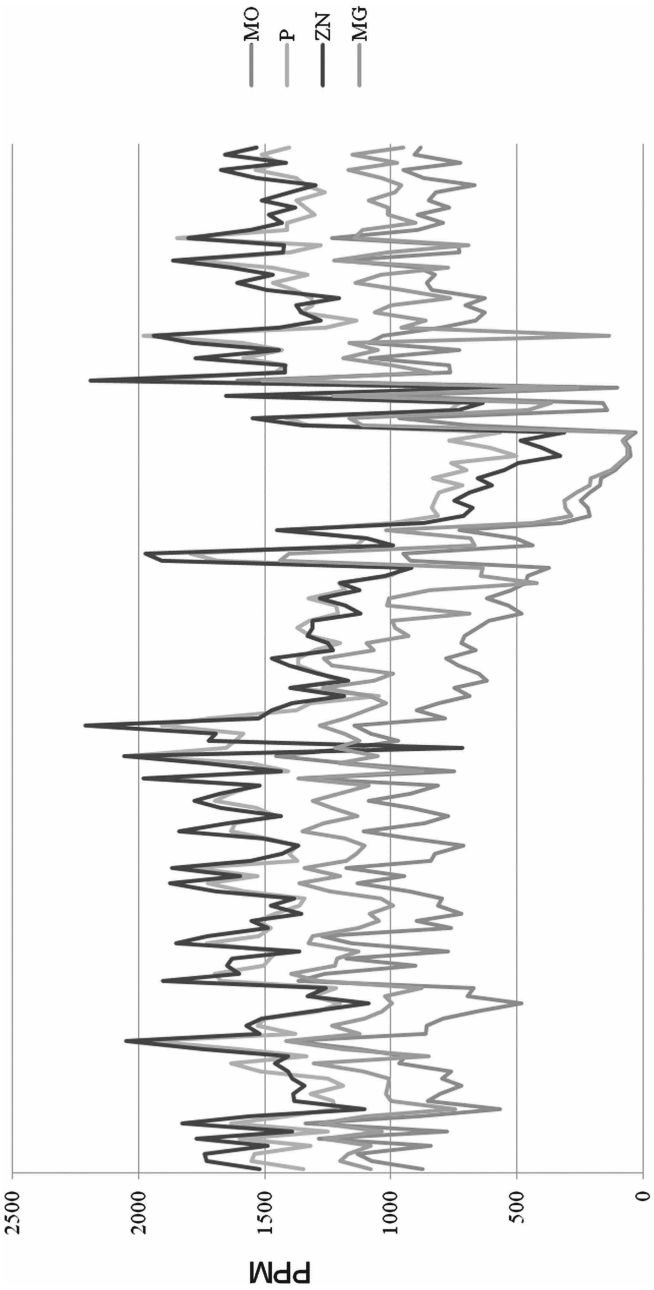


FIG. 8—*Plotting of additive metal content on 150 wind turbine oil samples.*

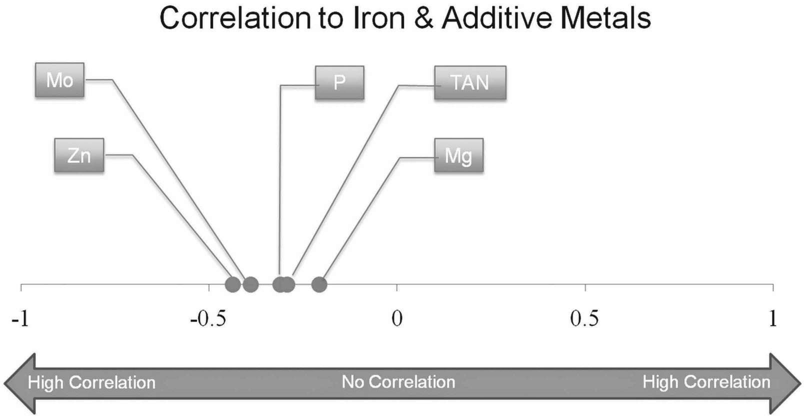


FIG. 9—*Correlation between iron and additive metals.*

emission spectrometer. It is logical that the reduction in additive metals is related to the formation of sludge in the oil and related deposits in the system. It is not known from this data if gear wear is occurring and iron is being generated due to the depletion of key protective additive components or because these degraded additives are creating sludge which is increasing the wear rate in this component. With further investigation, one could also ascertain the reason that zinc and molybdenum have a higher negative correlation to iron as compared to phosphorus and magnesium.

Hydraulic Oil Data Correlations

The analytical data from the hydraulic oil was also statistically analyzed. The correlation between oil condition and operating hours is illustrated in Fig. 10. Here one can see a positive correlation between the wind turbine's operating hours to molybdenum, magnesium, iron, copper, and fluid color. The correlation values of the wear metals iron and copper are not surprising. These are the two most important wear metals to trend in this hydraulic system. The color/haze of the fluid is also expected to increase as it ages and this correlation is clearly visible. The surprising value was the correlation of magnesium and molybdenum to machine hours, because these additive metals are not part of the new oil additive system. The correlation between molybdenum and magnesium was also extremely high at 0.926—indicating that the two metals are getting into the fluid at the same time and rate. These data suggest that the same oil handling equipment is being used for pumping gear oil and hydraulic oil up-tower and that insufficient flushing is done on the equipment after each use of the individual lubricant. The data showed a need for improved maintenance practices at the site to avoid cross-contamination issues. Although cross contamination of the gear oil into the hydraulic oil did not seem to correlate to an increase in wear, contaminating hydraulic oil into the gear oil system would most likely prove to be more damaging.

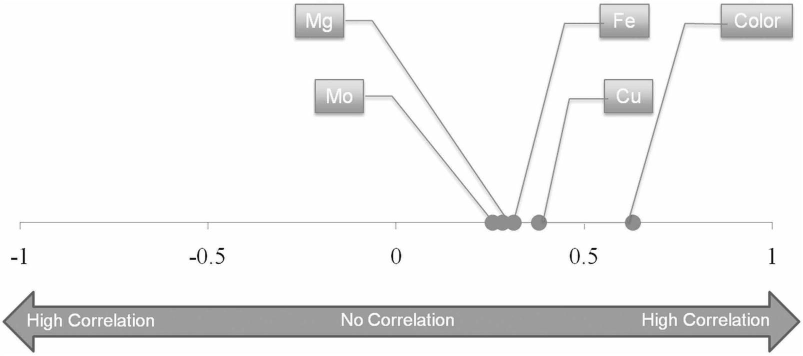


FIG. 10—*Correlation between hydraulic oil analysis data and wind turbine operating hours.*

Other Analytical Tests for Consideration

A large factor in determining appropriate testing methodologies is the product formulation. Based on the chemistry of this formulation, other analytical tools may be of value to the condition monitoring program.

Figure 11 shows that measuring the total base number of a fluid may also provide some value. The decrease in TBN may indicate the formation of sludge and deposits in the system.

Figure 12 shows the results of a patch test from an used wind turbine oil with a high amount of sludge. In order to generate this patch, 10 ml of oil was mixed with 50 ml of petroleum ether and was pulled through a 0.45 μm nitrocellulose patch by vacuum. This may be an appropriate screening tool to measure the deposit tendencies of the in-service wind turbine oil. The color and appearance of this patch indicated the materials being isolated are not varnish but sludge type materials. One can observe that the color is white/gray, a typical color of inorganics and carboxylate salts. The physical appearance of the material show that is was thick and soft during the separation and "dried" on the

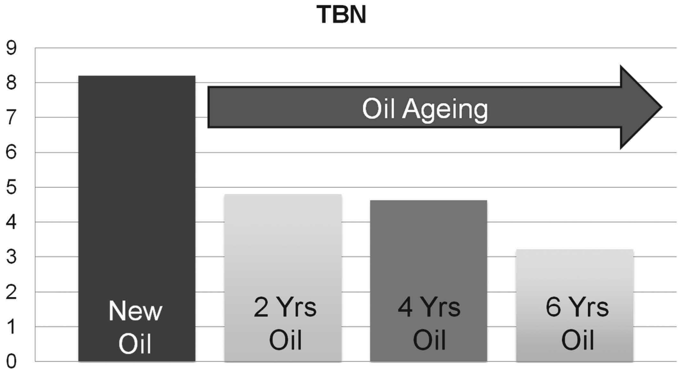


FIG. 11—*The trending of TBN over time can provide value in some formulations.*

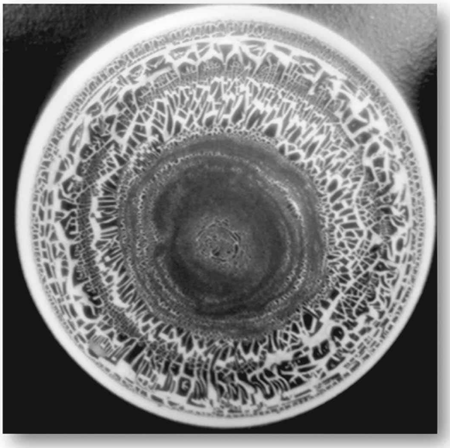


FIG. 12—*A patch from an in-service wind turbine gear oil suggesting high deposit tendencies in the fluid.*

patch causing the cracking appearance, a behavior of sludge type materials. This observation supports the metal analyses observation where there is a loss in additive metals to form sludge.

Figure 13 shows the value in using ^{31}P nuclear magnetic resonance (NMR) analysis in better understanding the behavior of phosphorus containing

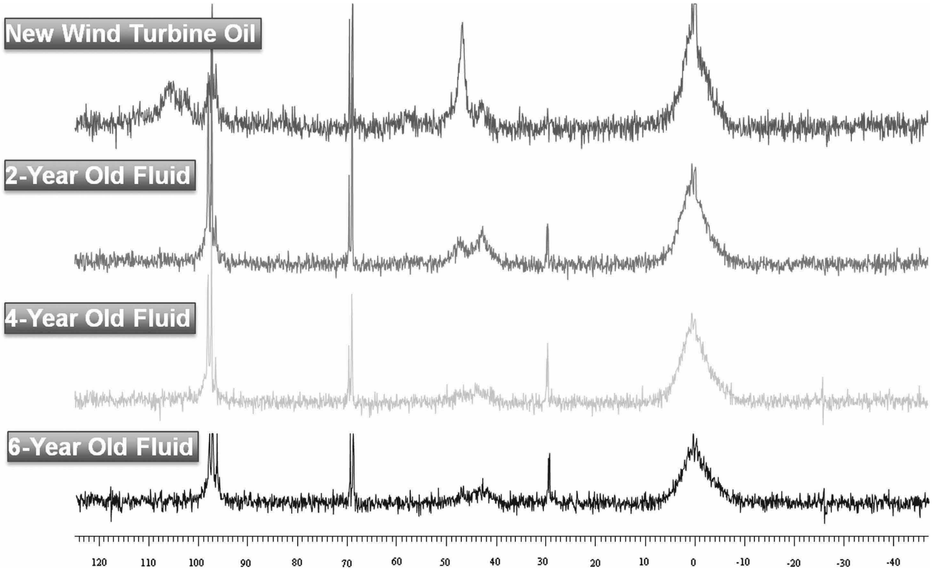
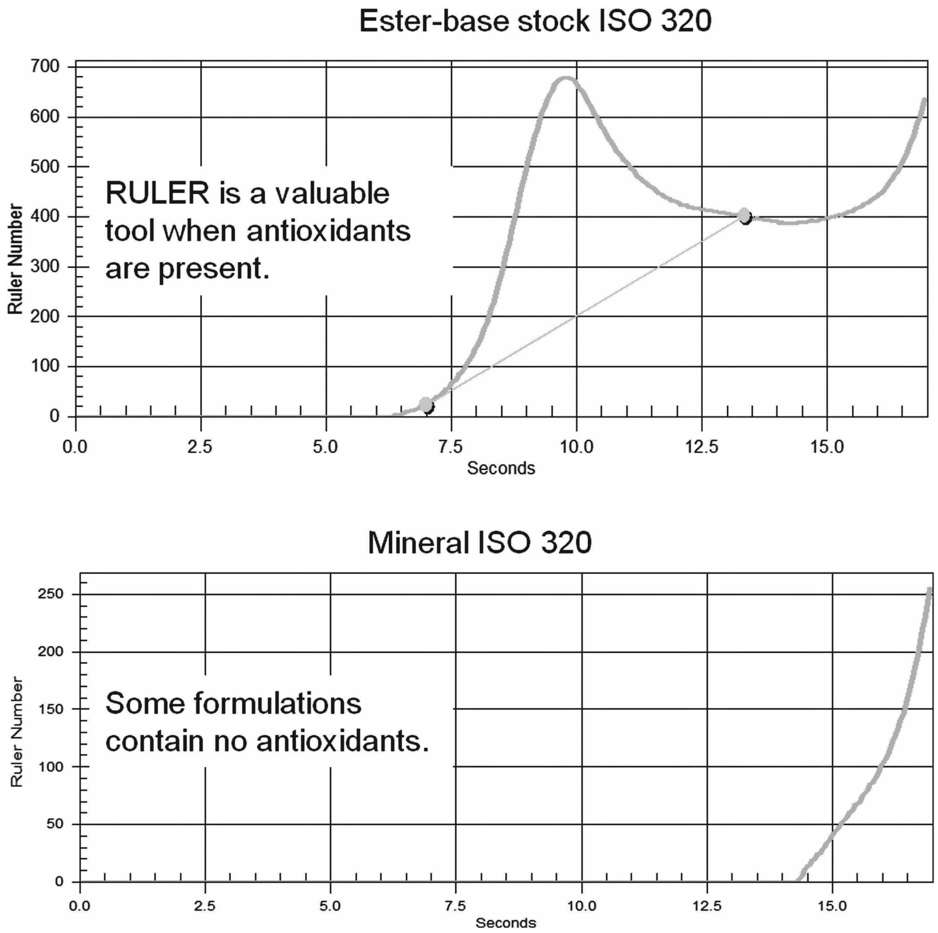


FIG. 13—*^{31}P NMR analysis can be valuable to understanding how phosphorus components degrade over time.*

additive components as the fluid ages. In this example, ^{31}P NMR was done on new fluid and compared to fluid that had been in service for 2, 4, and 6 years. Some interesting observations can be made from this analysis, such as:

- Overbased zinc dialkyl dithiophosphate (ZDDP) depleted very rapidly, whereas the neutral zinc dialkyl dithiophosphate decomposes over a much longer period of time.
- The intermediaries formed from the phosphite additive are decomposing due to oxidative mechanisms rather than wear mechanisms, suggesting a primary mode of degradation for the fluid.

The FTIR study of these samples showed that the polysulfide additive (which is the only purely extreme pressure additive component in the formulation) showed virtually no depletion over 6 years.



Ester-base stock ISO 320

RULER is a valuable tool when antioxidants are present.

Mineral ISO 320

Some formulations contain no antioxidants.

FIG. 14—*The use of linear sweep voltammetry is valuable if antioxidants are present in the new oil formula.*

Combining ^{31}P NMR information with FTIR and other analytical techniques can suggest primary modes of fluid degradation. In this particular case, the data suggested that the fluid was not degrading from thermal events due to boundary or elastohydrodynamic lubrication regimes, but rather from oxidative mechanisms.

Linear sweep voltammetry (ASTM D6971) is also a valuable trending tool to consider if the new oil formulation contains antioxidants. Figure 14 shows two examples of using linear sweep voltammetry with a fluid formulated with antioxidants and another brand of fluid devoid of antioxidants. This test also showed that the above series had no antioxidants remaining after only two years in service.

What about Oil Condition Monitoring Sensors?

Oil condition monitoring has shown tremendous value and rapid ROI in virtually all other lubrication applications. There are some inherent challenges in the wind turbine market due to the maintenance expense of obtaining samples on a frequent basis. This suggests that online sensors could play a valuable role in monitoring in-service wind turbine oils.

To date however, there is reluctance by wind farm operators and equipment OEMs to invest in sensors that are commercially available. One reason is that wind farm operators go to great lengths to minimize their maintenance and operational costs in order to keep their cost of power competitive with fossil-fuel derived power sources. A second reason is that the value of inline sensors in wind turbines has not been adequately demonstrated, making it hard to justify the expense. Until wind farm operates can justify the investment into real-time oil analysis tools, we must rely on off-site laboratory analysis. It is therefore important to optimize the value derived from this off-site analysis.

Conclusion

There are several inherent challenges in setting up an effective oil condition monitoring program for wind turbines. The wide number of formulation chemistries and fluids commercially available is one obstacle, since each fluid type should have tests chosen specifically for that formulation if one is going to follow the fluid's condition. There are also a wide number of component manufacturers, each utilizing different metallurgy and having slightly different lubrication requirements. The biggest challenge however is in collecting enough good data to produce trends. The trend of extending maintenance intervals from 6 to 12 to 18 months makes it even more challenging to obtain enough data in an oil analysis program.

Due to infrequent oil sampling and limited data, it is important to not make assumptions in your data set. An example of a wind farm has been presented assuming that water contamination was not an issue, but after collecting two samples per month for 6 months, realized that water contamination was impacting the condition of their lubricant.

Selecting appropriate analytical tests for a wind turbine oil condition monitoring program depends upon the oil formulation, OEM standards, and data collected from in-service fluids. It should be an evolving program that is continually improving based on a regular stream of new data inputs.

Statistical analysis can play an important role in measuring the importance of oil analysis data. Trending is the typical form of statistical analysis utilized by commercial laboratories. The use of additional statistical methodologies such as principle component analysis can be of value to provide additional information on the formulation of the fluid in addition to the understanding what happens to the fluid over time while it is in service.

The importance of improving the quality and frequency of oil analysis data cannot be understated. The better ability that we have to adequately monitor the condition of wind turbine oil, the higher reliability we can expect out of these assets and the more competitive this form of energy will be.

Acknowledgments

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The Use of Linear Sweep Voltammetry in Condition Monitoring of Diesel Engine Oil

ABSTRACT: One of the main ways to decrease engine oil changes is to increase drain intervals. Although it has been a controversial subject for years, increased drain intervals can be accomplished safely using lubricant analysis. Over the years, many diesel engine operators have effectively employed used oil analysis to extend engine oil drain intervals safely by monitoring the condition of the oil and the engine. Various tests have been used for the monitoring of engine oil. However, although it has been used for years to evaluate the remaining useful life of industrial lubricants, linear sweep voltammetry has not been used for routine monitoring of engine oil. This paper will show how linear sweep voltammetry can be an effective method for maximizing extended drain intervals in an over-the-road heavy-duty trucking fleet.

Introduction

Most equipment users desire and expect reliable performance from the equipment they use. This is especially true for over-the-road trucking fleets, construction and mining companies, and racing teams. One thing that all of these users have in common is that the equipment they use is powered by an internal combustion engine. For quite a few years, many of these users have employed oil analysis to monitor the condition of both the oil and the engine. For race teams, the analysis is mostly used for monitoring the condition of the car's engine. The oil is generally changed prior to a race. For the trucking fleet and construction and mining companies, if the equipment is not running, then it is costing them

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money rather than earning them money. As a result, minimizing downtime is crucial. This downtime can include repairs as well as normal maintenance.

Some of today's engine oils are formulated to provide excellent protection and extended drain performance, which helps to minimize maintenance downtime. Because of varying equipment operating conditions, safe extended drain intervals can only be achieved when coupled with used oil analysis. Various tests have been employed to monitor the condition of used engine lubricant. Linear sweep voltammetry, ASTM D6971 [10] for turbine lubricants, has been introduced recently as an additional test for obtaining even more detail about the remaining useful life of in-service engine oils.

When it comes to lubricant formulations, engine oils are probably the most complex. They are formulated to protect against wear, oxidation, contamination and corrosion. Corrosion prevention is usually only measured during product design and production quality control analysis. Most used oil analysis techniques are devised to evaluate contamination and degradation of the oil.

Because of the fuel combustion process, engine oil often needs to be changed to remove contamination before it is actually degraded to the point that it is no longer useful. Today, many companies are beginning to use efficient filtration techniques in combination with high-performance engine oils. This makes it possible to use the oil until it is worn out.

Following is a list of some of the traditional in-service engine oil tests:

- Kinematic Viscosity (usually at 100°C and sometimes also at 40°C)
- Total Acid Number (TAN)
- Total Base Number (TBN)
- Elemental Spectral Analysis: Wear Metals; Organometallic Additive Metals; Dirt
- Fourier Transfer Infrared (FTIR) Spectral Analysis: Oxidation; Water; Additive; Depletion; Nitration; Glycol Contamination; Organic Cross-Contamination; Soot (Carbon) Contamination.

So, how does the user know when engine oil is worn out? Over the years, many analysts have used a combination of viscosity change, TAN increase, TBN decrease, and FTIR to predict remaining useful life. A common rule of thumb has been to condemn the oil when the TAN value climbs higher than the falling TBN value. This is called the crossover point. The acid number climbs because of the formation of acidic byproducts that collect in the oil either from contamination from fuel combustion or from engine oil oxidation. Basic additives are incorporated into engine oil formulations to neutralize these acids.

At the beginning of 2007, fuel refiners were required to produce ultra-low sulfur content fuels. The refiners forced sulfur dioxide compounds into the oil, where the compounds mixed with water vapor to form sulfurous acids in the oil. With most of the sulfur removed from the fuel, the only source of acid formation is carboxylic acids formed from the oxidation of the engine oil. Under normal engine operations, oil oxidation acids should not accumulate as rapidly as when higher sulfur diesel was burned. Therefore, the question can be asked, "Is the TAN/TBN crossover still a good indicator of an oil's remaining useful life?"

Coincidentally, the additives used to neutralize the oxidation acids that form in the oil are not antioxidants. So, the question needs to be addressed, "Does consumption of the TBN indicate that the oil's oxidation resistance by antioxidants consumed?" Linear sweep voltammetry directly measures the remaining concentrations of antioxidants in used engine oil.

In this study, engine samples were collected at controlled intervals to optimize the drain intervals for over-the-road trucking units. Along with the traditional engine oil tests listed above, linear sweep voltammetry was conducted on the samples to determine the remaining useful life in the samples. With the new ultra-low sulfur fuels and new low-emission engine lubricant formulas the question being addressed is, "Do the traditional tests provide the full story when it comes to extended drains and remaining useful life?"

Linear Sweep Voltammetric Method for Engine Lubricants

The linear sweep voltammetric method selected for this research paper was performed with a portable, commercially available RULER voltammograph (RULER is a trademark of Fluitec International), equipped with a three-electrode system and a digital output. The combination electrode system consists of a glassy carbon disc (3-mm diameter) working electrode, a platinum wire (0.5-mm diameter) auxiliary electrode, and a platinum wire (0.5-mm diameter) reference electrode, as described in ASTM D6447 [11].

The RULER instrument uses linear sweep voltammetric techniques applying a controlled voltage ramp, 0 to -1.8 V range with respect to the reference electrode (at a rate of 0.01 to 0.5 V/s), to the auxiliary electrode through the electrode inserted into the diluted oil sample.

To perform a test, the new and in-service oil samples (400 μl) were diluted with an alcohol-formulated analytical test solution containing dissolved electrolytes and a suspended solid substrate [1].

When the hydrocarbon-based oil/solvent/solid substrate mixture was shaken, the suspended solids quickly precipitated the insoluble oil, leaving a clear solution for analysis. This solution was free of interference from fuel, soot or contaminants. As the voltage potential increases, the antioxidants and anti-wear additives (such as ZDDP) become chemically excited (at their respectively oxidation-reduction potential), causing the current to increase until it reaches a peak. It then decreases as the potential continues to increase. (See Fig. 1 below.) In the voltage/current relationship, the voltage potential range is related to the identity of the type of the antioxidant or anti-wear additive (such as ZDDP, amines, phenols, phenates, salicylates, etc.), and the current peak indicates the concentration of the additive.

The current wave or peak is measured and stored in the data acquisition software as a value. This RULER number, when compared to its standard (fresh oil), will indicate how much additive depletion has occurred.

By tracking the RULER readings over time, the user is able to observe a decrease in the RULER number to determine when a critical condition is likely to occur and to estimate the remaining useful life of the oil, before acid number or viscosity indicates significant change.

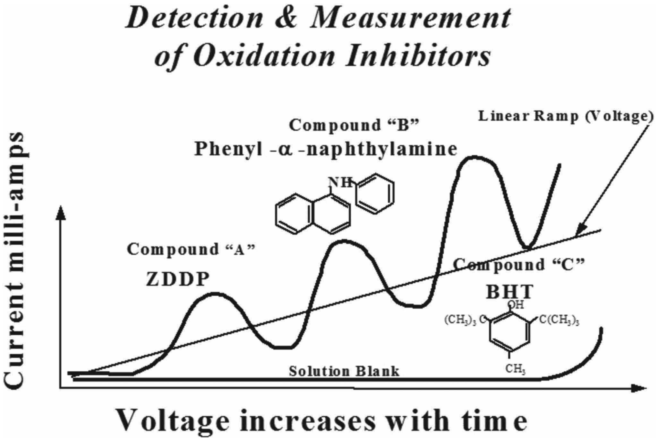

FIG. 1—*Voltage/current graph for voltammetric test method, with time/voltage in X-axis and current in Y-axis for three types of antioxidants, ZDDP, amines and phenols in a neutral-type of voltammetric test solution.*

RULER as an Instrument for the Detection of Additives

An engine lubricant has several functions, such as protecting engines from wear, maintaining cleanliness and acting as a coolant. The components of a lubricant are predominantly base fluid(s) containing specially designed additives. These additives provide the lubricant with its attributes and protect its base fluid from degradation, an important function because the base fluid is the carrier used to transport the additives around the engine. Most base fluids used in modern diesel and gasoline engines are hydrocarbon-based and are prone to oxidative degradation.

Three additive types are used to control such degradation:

- Radical scavengers (sterically hindered phenols and aromatic amines),
- Hydroperoxide decomposers (organosulphur and organophosphorous),
- Synergistic mixtures of these (ZDDP, phenates, salicylates, etc.).

The RULER method of linear sweep voltammetry monitors these types of additives and gives a measure of the effective antioxidant level in the oil as it ages. Because each additive chemistry is activated at a different voltage, many formulated oils show a characteristic RULER voltammogram. This could be useful in the field when top-up with a different oil is suspected. Typically, two to three additive peaks are detected: one from ZDDP, one from amine, and one from phenolic antioxidant. The data allows the user to monitor the depletion of both additives, as Fig. 2 shows. If the total effective antioxidant capacity depleted rapidly to a low level, this could suggest the oil is inappropriate for the engine's particular duty cycle. For this study, the antioxidants' RUL% numbers and the total RULER area (representing total antioxidant concentration) were used to compare results with other standard used oil analytical techniques.

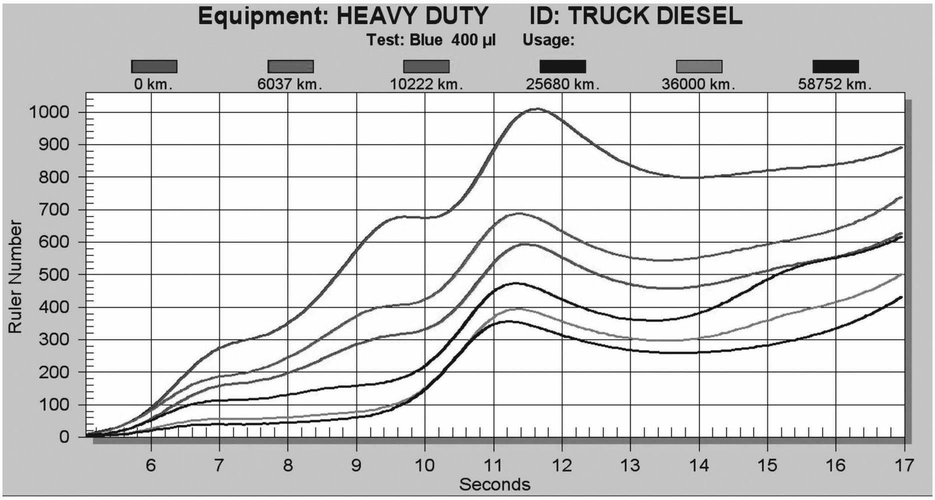

FIG. 2—*Depletion trending curve for antioxidants in truck diesel oil from 0 to 58,752 km.*

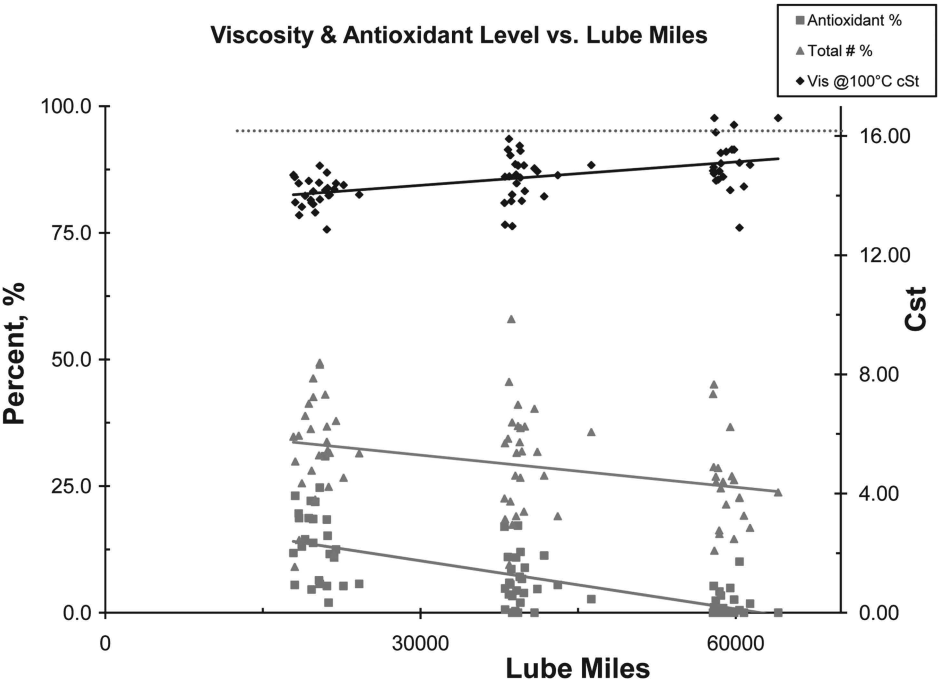

FIG. 3—*Antioxidant levels (RUL%) and viscosity at 100°C versus lube miles.*

Experimental

The units used in this experimental study were Caterpillar C-15 engines, used in over-the-road, long-haul trucking service. The background information collected for each unit and sample include engine type, total miles, lube miles, and date of the sample. The testing data collected for comparison included viscosity at 100°C, Total Acid Number (TAN), Total Base Number (TBN), Fourier Transform Infrared Spectroscopy (FTIR) (oxidation, nitration and sulfation), metals (iron and chromium), silicon, and additive levels using linear sweep voltammetry (RULER). The laboratory test equipment used for the analysis includes:

- *TAN and TBN*—A Metrohm Model: 751 GPD Titrino with full automation was used to acquire the results per ASTM D4739 [2], D2896 [3] and D974 [4].
- *Viscosity*—The viscosity was manually acquired using a Cannon-Fenske Opaque Viscometer for Transparent and Opaque Liquids per ASTM D445 [5] and D446 [6].
- *FTIR*—The oxidation, nitration and sulfation values were acquired using a Bruker Tensor 27 along with Spectro's Oil Analysis program per Joint Oil Analysis Program (JOAP) protocol [9].
- *Optical Emission Spectrometry (ICP)*—The silicon and metals; iron and chromium results were collected using and PerkinElmer Optical

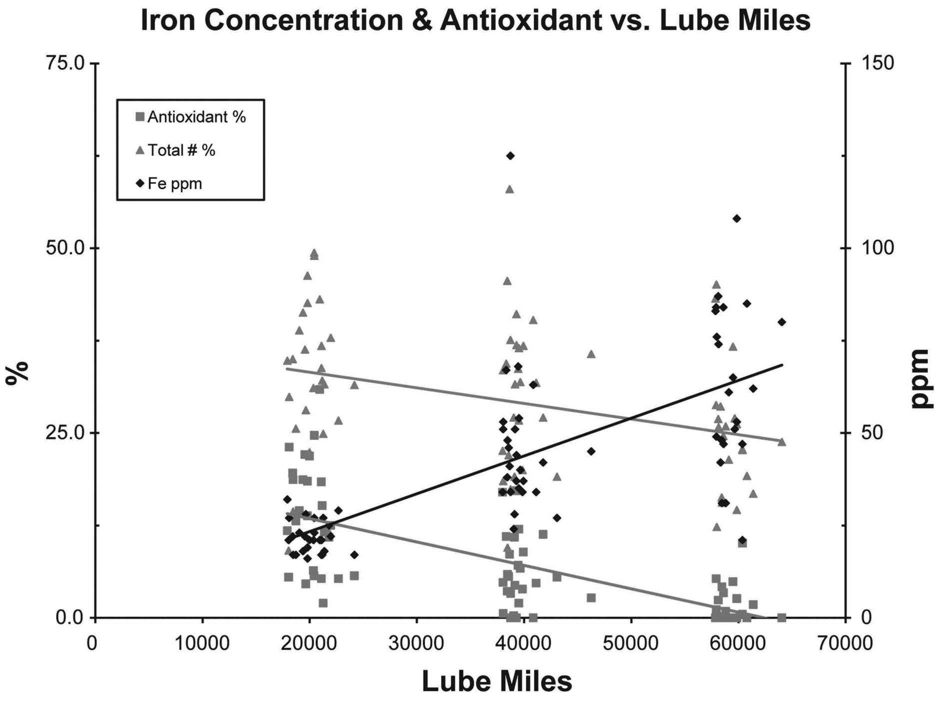

FIG. 4—*Close-up comparison of antioxidant additive levels (RUL%) and Fe (iron) concentration versus lube miles.*

Emission Spectrometer Model: Optima 4300V per ASTM D4951 [7] and D5185 [8].
- *Linear Sweep Voltammetry (RULER)*—The additive levels were collected using Fluitec's RULER, Model CE320 per the user's guide and operation and instruction manual.

Data was collected for each unit when there was a minimum of three sample dates, representing a complete cycle for the oil. The oil was run in each unit for approximately 60,000 miles, and the sampling was completed at approximately 20,000, 40,000 and 60,000 miles. The filters and oil were not changed until approximately 60,000 miles.

Results

There were 74 samples taken from the units, with a minimum of three samples per unit. The results can be seen in Fig. 3. It shows the antioxidant additive levels and viscosity at 100°C versus lube miles, the dotted line indicates the cutoff for the SAE40 range. It shows that the antioxidant additive levels are depleted before the viscosity reaches the cutoff point.

In Fig. 4, the iron concentration trends very well with the remaining antioxidant additive level trends. The iron levels show how easy the trending for the

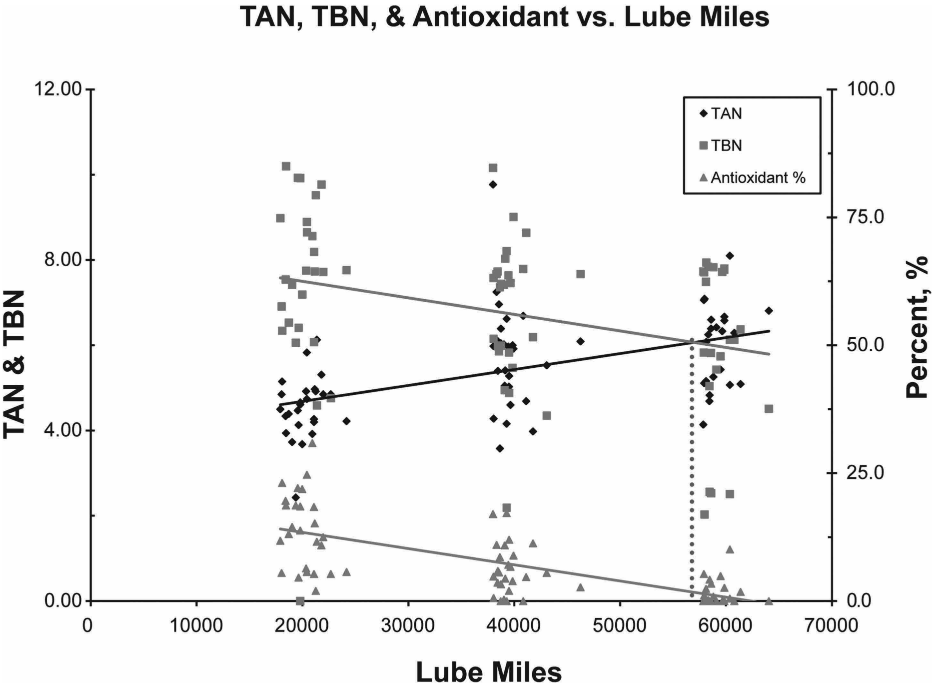

FIG. 5—*Close-up comparison of antioxidant additive levels (RUL%), TAN and TBN versus lube miles.*

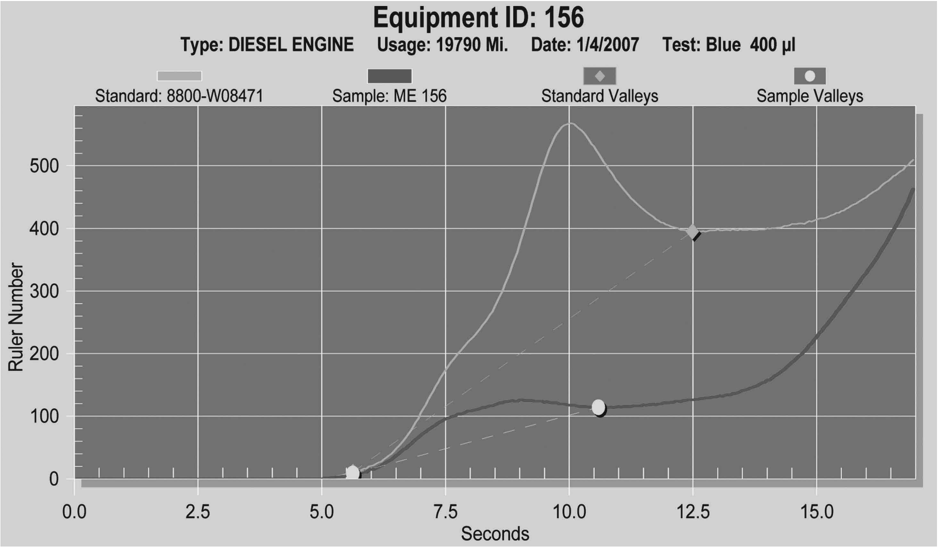

FIG. 6—*Voltammograph from Unit 156, collected at 19,790 lube miles and 18.5% remaining antioxidants.*

FIG. 7—*Voltammograph from Unit 156, collected at 39,927 lube miles and 8.9% remaining antioxidants.*

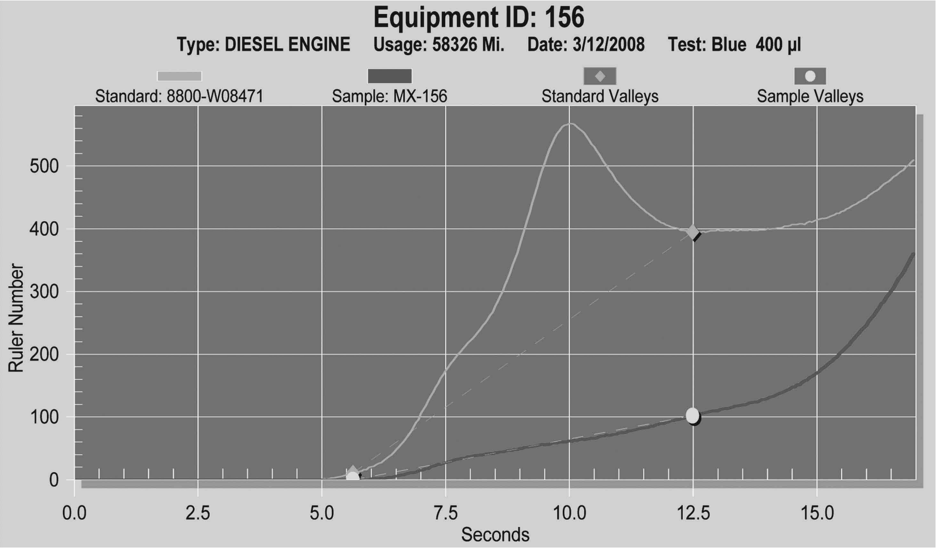

FIG. 8—*Voltammograph from Unit 156, collected at 58,326 lube miles and 0.2% remaining antioxidants.*

iron would be and how easily it would be to set limits for normal service and end of oil useful life.

The antioxidant levels—compared to the TAN and TBN, when plotted versus lube miles—shows that the TAN and TBN would meet at less than 60,000 miles (as seen in Fig. 5). In individual units, this result was before and after the endpoint seen for the additive levels.

Discussion

Each sample was tested using the RULER and the data and graphs collected. The data shown was collected from unit 156 from approximately 350,000 to 450,000 engine miles. Figure 6 represents the oil sample that was collected at 19,790 lube miles, and the % remaining antioxidant additives (or RUL%) was 18.5.

Figure 7 shows the sample that was collected at 39,927 lube miles, and the % remaining antioxidant additives was 8.9. Figure 8 shows the oil sample collected at 58,326 lube miles, and the % remaining antioxidant additives was 0.2%.

The results from the graphs above produced by the RULER for unit 156 were plotted linearly to show how the results related to the other data collected.

Table 1—*R^2 values for test data collected on Unit 156.*

	Antioxidant Level RUL%	Total Antioxidant Level	Viscosity @100°C	TAN	TBN	Iron	FTIR: Oxidation	FTIR: Nitration	FTIR: Sulfation
R^2	1	0.9845	0.9997	0.9091	0.9904	0.9186	0.9733	0.9733	0.9733

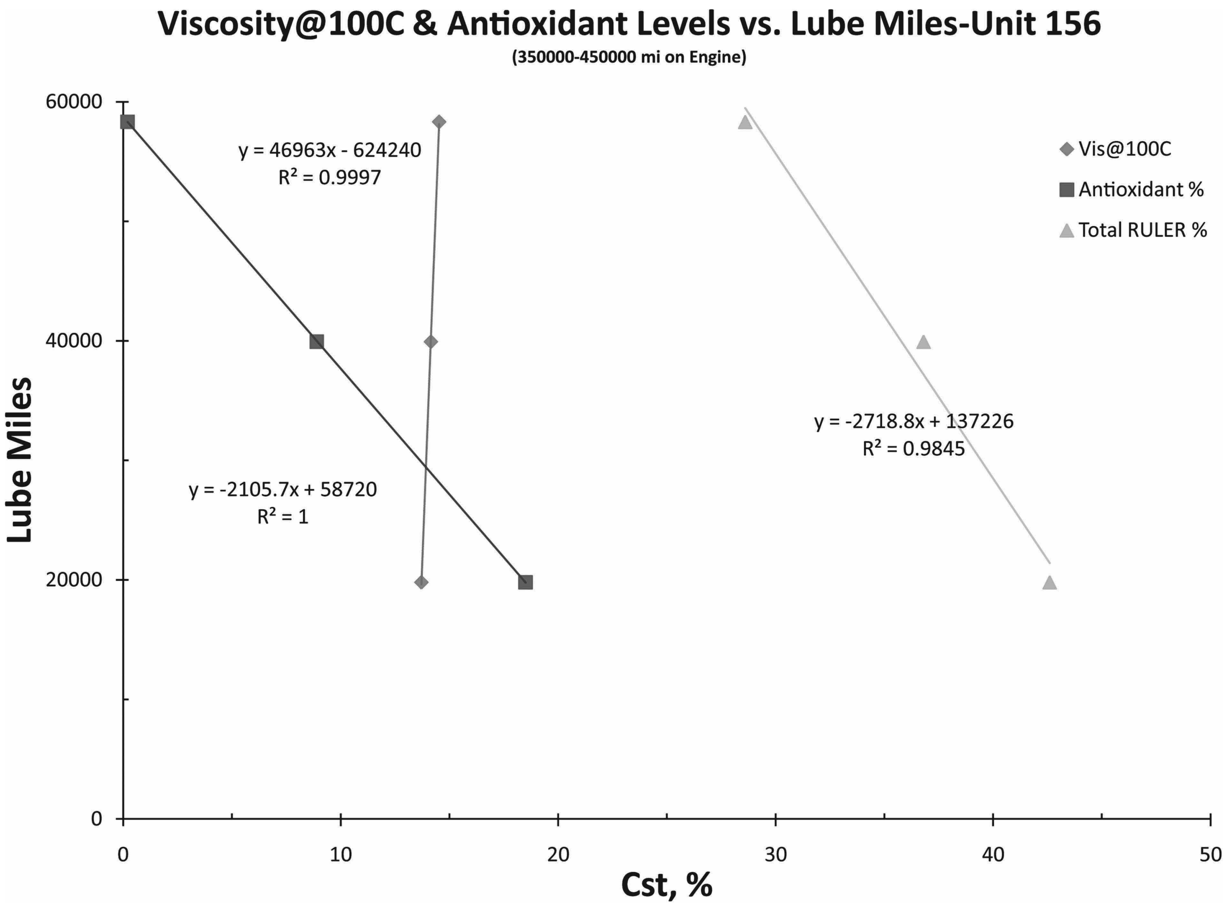

FIG. 9—*Antioxidant levels and viscosity at 100°C versus lube miles.*

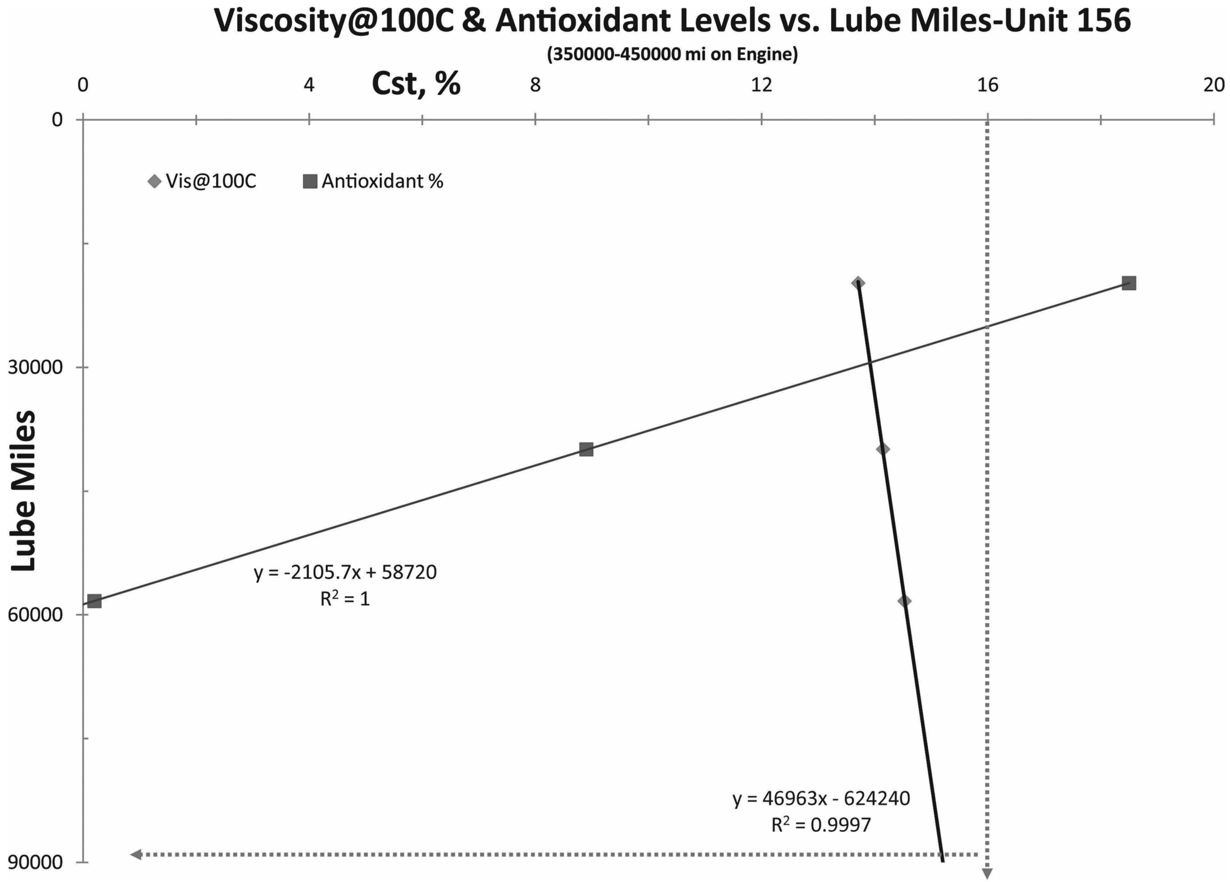

FIG. 10—*Close-up comparison of antioxidant levels and viscosity at 100°C versus lube miles.*

The R^2 values were calculated for each test, found in Table 1. The remaining antioxidant (additive #1) includes ZDDP, amines and Ca-phenates. The phenols are included in the total antioxidant RUL% calculation

In Fig. 9, the first test graphed in relation to lube miles and antioxidant levels was the viscosity at 100°C. The graph shows the data as a trend line. The R^2 was determined along with the line's equation for each of the data sets.

If you take the graph and enlarge the area where antioxidant % and the viscosity reach the end of the lube cycle (see Fig. 10), it can be seen that the trending of antioxidant additive #1 shows the end of the oil's useful life better than the trending done for the viscosity. The pink vertical dotted line shows where the viscosity value would exceed the SAE40 range. The pink horizontal dotted line shows where the viscosity would be at 90,000 lube miles.

The graph shows that for the viscosity to be heavier than the SAE40 range, the engine would have lube miles greater than 90,000 miles. The antioxidant additive level is approaching the 0 value at approximately 60,000 miles. The total antioxidant level is less than 45%, which compared to the additive level is not very much additive. The remaining antioxidant is the phenol additive for engine additive protection.

The antioxidant levels—compared to the TAN and TBN, when plotted versus lube miles— shows that the TAN and TBN would meet after 75,000 miles (Figs. 11, 12, and 13). This would be far beyond the oil's useful life. Even if

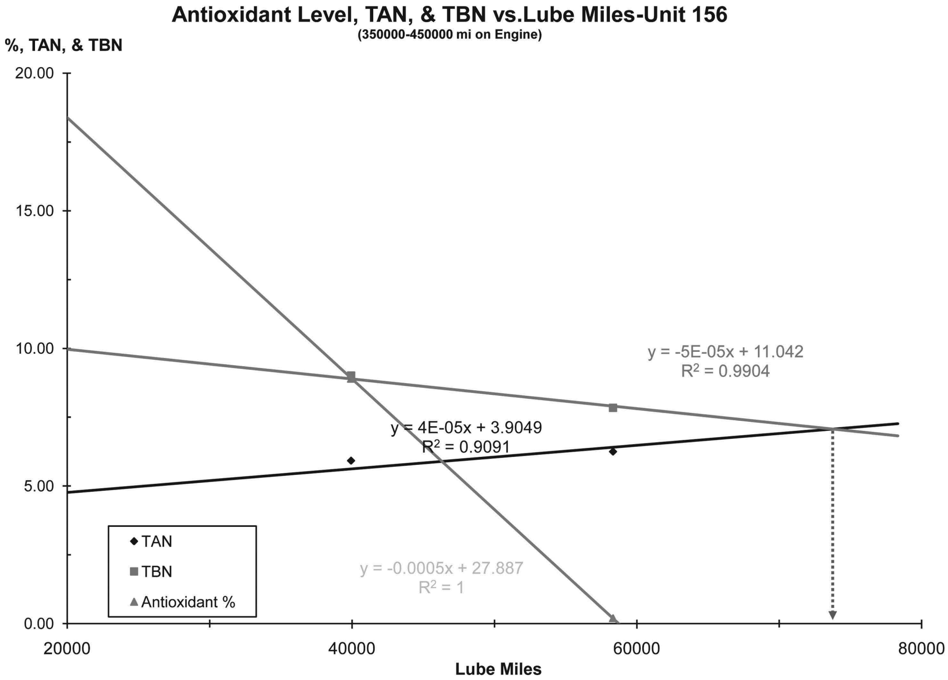

FIG. 11—*Close-up comparison of antioxidant levels, TAN and TBN versus lube miles.*

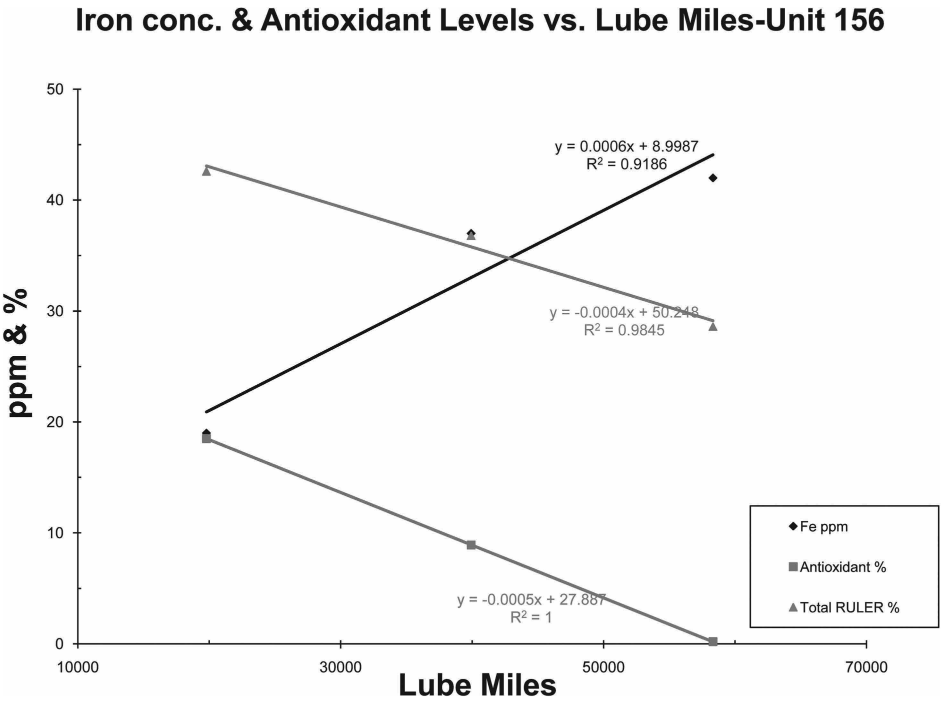

FIG. 12—*Close-up comparison of antioxidant levels and Fe (iron) concentration versus lube miles.*

putting the viscosity results and the TAN and TBN results together, this does not paint an accurate picture of the oil's remaining useful life.

The iron concentration trends very well with the antioxidant level trends. The iron levels show how trending the iron would allow limits (ppm) to be set for normal service.

The nitration, sulfation and oxidation results from the FTIR trend very well with the iron concentration and antioxidant RUL%. The FTIR results show how trending this data would allow limits to be set for normal service or continued use and end of useful life of the oil.

Conclusion

For this experimental study, Caterpillar in-service C-15 engines used in over-the-road and long-haul trucking service were sampled at 20,000-mile intervals up to approximately 60,000-mile drains. A total of 74 oil samples were collected from these units, with a minimum three per unit. The objective was to see if all the traditional testing for used engine oil monitoring adequately shows remaining useful life of in-service engine oils.

A useful tool in determining remaining useful engine oil life is linear sweep voltammetry, commercially available as the RULER method. The RULER

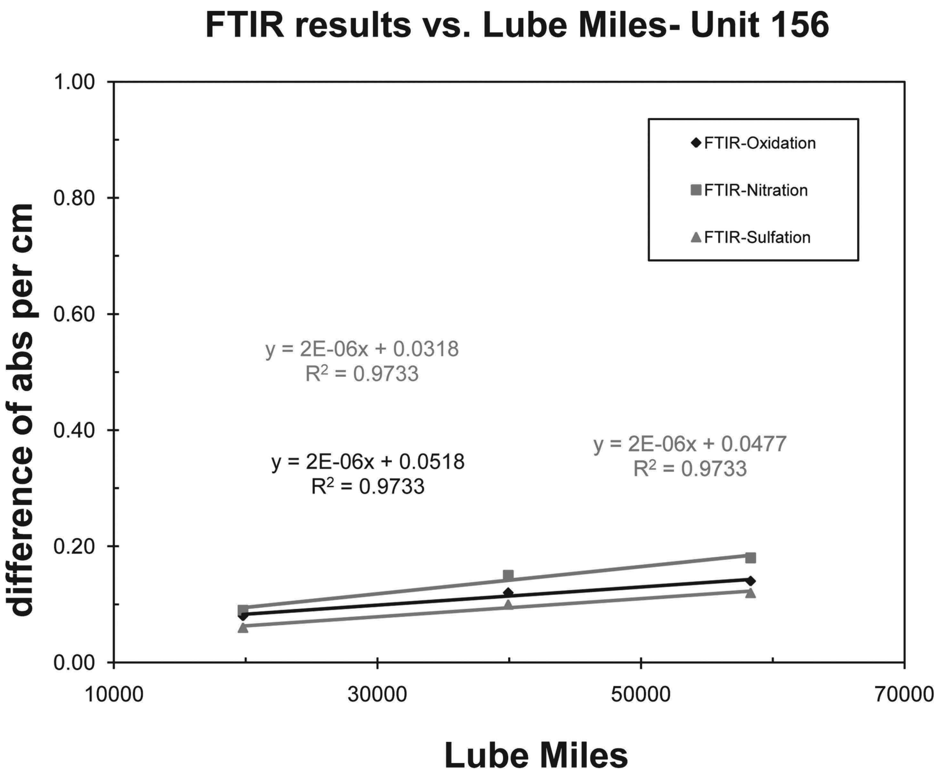

FIG. 13—*Close-up comparison of antioxidant levels and FTIR results versus lube miles.*

results were compared to results from traditional used engine oil tests, providing answers to the questions on p. 42 of this report.

Q: *Is the TAN/TBN crossover still a good indicator of oil remaining useful life?*

A: In the work done, it appears the TAN/TBN crossover is not a good indicator of oil remaining useful life, and neither is viscosity.

Q: *Does consumed TBN indicate that the oil's oxidation resistance is used up?*

A: The TBN being consumed does not indicate oil's oxidation resistance being used up as reliably as the RULER does. However, the wear metal monitoring and the FTIR (oxidation, nitration and sulfation) do appear to trend right alongside the RULER results.

Q: *With the new ultra-low sulfur fuels and low-emission engine lubricants, do the traditional tests provide the full story when it comes to extended drains and remaining useful life?*

A: The traditional tests do not appear to provide the full story when it comes to extended drains and remaining useful life of oil.

Q: *How does the user know when engine oil is worn out?*

A: This question can be answered by many of the tests working together in a monitoring system that includes the RULER method.

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An Overview of Progress and New Developments in FTIR Lubricant Condition Monitoring Methodology

ABSTRACT: Fourier transform infrared (FTIR) spectroscopy has found an important analytical niche in the field of condition monitoring (CM) of lubricants, lubricant quality being a primary determinant of the wear and operational efficiency of engines and machinery. The ability to track a wide range of functional groups associated with chemical changes in lubricants by FTIR spectroscopy allows the overall quality of lubricants to be assessed and informed decisions made as to whether a lubricant needs to be replaced or not. Newer automated FTIR systems in centralized laboratories are now capable of analyzing oil samples at rates of >120 samples/h, providing trending information on soot, moisture, glycol, oxidative status, anti-wear additives, and nitration, amongst other measures. The current FTIR CM methodology is based on ASTM Standard Practice E2412-10 and is restricted primarily to petroleum or mineral oils; however, the ability to spectrally classify oils could overcome this limitation and broaden the scope of the methodology substantially. Quantitative FTIR methods have come to the fore to carry out more specific measures related to oil condition, specifically the determination of acid content and base content, as well as moisture. These methods have been designed to overcome some key limitations associated with the corresponding ASTM titrimetric procedures and expand the overall utility of FTIR instrumentation in centralized lubricant analysis laboratories. This paper provides an overview of FTIR CM, how it has been and can be further improved, and discusses the recent advances in the newer quantitative FTIR CM

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methods as well as the issues that need to be addressed to further enhance the utility of FTIR spectroscopy in relation to lubricant analysis.

KEYWORDS: lubricant analysis, FTIR spectroscopy, acid number, base number, moisture content, spectral reconstruction

Introduction

Lubricants undergo complex chemical changes when in-service as a result of thermal, physical, and oxidative stresses imposed on them as they lubricate moving surfaces. Whether they perform their task adequately depends on their formulation and their "condition" as a function of time. Although the term condition is rather nebulous, it implies a measure of changes in the quality of the lubricant relative to its original formulation and its ability to perform the lubrication task for which it was originally designed. Depending on the application, the loss of lubricant performance may range from problematic to catastrophic—in the case of a bicycle chain, lubricant failure may make pedaling harder, whereas a bearing malfunction due to poor lubrication in a jet engine clearly has more serious consequences. Lubricant condition monitoring (CM) tends to focus on factors known to affect equipment performance and reliability and aims to enable informed decisions to be made as to whether there is a need to replace the lubricant or in some instances to re-additize [1]. Although time- or mileage-based criteria can be and often are used to schedule lubricant changes, CM affords the possibility of optimizing lubricant use by avoiding unnecessary changes while simultaneously safeguarding against premature lubrication degradation and consequent machinery failure. Typical examples of sectors where CM is a cost-effective and useful diagnostic tool include the military, the transportation sector, and various industrial sectors operating heavy equipment that is expensive and requires organized ongoing service and maintenance.

Lubricant CM can involve a wide variety of measures, including wear metals, magnetic metal debris, oil particle counts, viscosity, acid number (AN) or base number (BN), soot, levels of specific additives (e.g., anti-wear, antioxidants) and specific contaminants (e.g., moisture and glycol), as well as the lubricant's overall oxidative status [2,3]. Fourier transform infrared (FTIR) spectroscopy has emerged as a CM tool of choice because it provides a simple means of screening lubricants for a wide range of important lubricant quality parameters, yielding a wealth of information in a matter of minutes [4,5]. Because most oil constituents and contaminants have fairly distinct infrared (IR) signatures, FTIR spectroscopy can provide much of the basic oil condition information required to make a determination as to whether the oil is potentially problematic, either by comparing its FTIR spectrum with that of a reference oil or by trending the in-service oil over time without using a reference oil. Beyond this essentially *qualitative* approach, FTIR spectroscopy has also been developed as a *quantitative* analytical tool that can be applied to determine acid content (AC), base content (BC), and moisture content in lubricants [6]. These quantitative FTIR methods provide laboratories with rapid, automated, and high-throughput alternatives to conventional ASTM titrimetric procedures. In this paper, we examine the benefits of FTIR CM, its limitations and enhancements, as well as the newer quantitative methods and their more recent enhancements.

IR Spectroscopy

All organic molecules absorb IR energy at specific wavelengths that match the frequencies of their molecular vibrations. Indeed, a pure compound has a unique IR spectrum that represents a "fingerprint" from which it can be definitively identified. Similarly, a well-defined mixture of compounds, such as a new lubricant, has its own characteristic spectrum, representing the overlap of the spectral contributions of all the individual components making up the mixture (Fig. 1).

One of the strengths of IR spectroscopy derives from the fact that specific functional groups, such as CH_3, CH_2, OH, COOH, NH_2, etc., each absorb in defined wavelength regions, allowing one to ascertain their presence in a sample and, to some degree, the relative proportions of each. This capability to provide functional-group information is one of the underlying principles of FTIR CM analysis, in that many of the chemical changes occurring in in-service oils over time are associated with the formation or loss of particular functional groups. Similarly, the quantitative lubricant analysis methods described in this paper have all been designed around the concept of functional-group absorptions and their adherence to Beer's law (i.e., the direct proportionality between the concentration of the functional group in the sample and the intensity of its absorption in the sample's IR spectrum).

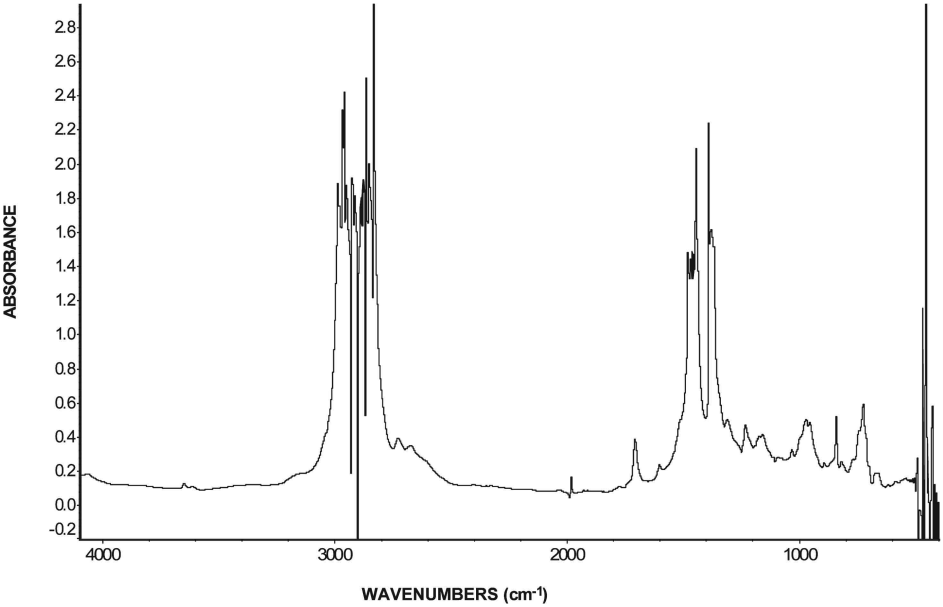

FIG. 1—*Spectrum of a typical new mineral oil based commercial motor oil formulation.*

Qualitative FTIR CM

Owing to the variability and the complexity of the IR spectra of lubricants, FTIR CM of in-service lubricants is usually a comparative trending procedure. The basic premise is to track spectral changes over time and associate these changes with well-defined and known quality changes or objective measures thereof. Although it was recognized early on that IR spectroscopy had potential as a screening tool for assessing in-service lubricant quality [3,5], the concept was not developed in a structured manner until the backing of the U.S. military establishment was placed behind the effort. In the mid-1990s, FTIR CM protocols were elaborated for the Joint Oil Analysis Program (JOAP) of the U.S. military, with much of this fundamental development work being carried out by Toms [7–10] at the JOAP Technology Support Center. The basic premise underlying the JOAP initiative was that routine FTIR examination of in-service oils could be employed to monitor their condition so as to minimize mechanical failures in the field and lead to improved use of lubricant resources. The development work conducted by Toms involved an extensive study of the typical spectral changes taking place in in-service mil-spec oils over time, supplemented by experiments in which components were spiked into the oils to simulate various types/levels of contamination. This seminal work focused on relating specific spectral changes to well-known deteriorative processes, such as the loss of antiwear additives, ingress of moisture, oxidation, soot accumulation, and glycol contamination, amongst others, which will be referred to in this paper as CM parameters, and ultimately led to the development of ASTM E2412-10, Standard Practice for Condition Monitoring of Used Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry [11]. The rapid and centralized analysis of in-service lubricants was also facilitated by the commercialization of automated FTIR analyzers for lubricant CM [12,13], which CM laboratories commonly link to a laboratory information management system (LIMS) to produce concise reports on lubricant quality specific to the equipment from which the oil was sampled [13]. If trend lines derived from the FTIR CM parameters as a function of time/mileage have been developed for the equipment in question and correlated with oil quality, FTIR CM analysis can serve to trigger an objective “treat oil” or “change oil” action, as discussed in ASTM E2412-10. Although maximizing the effectiveness of FTIR CM in this manner can be a fairly sophisticated undertaking, it has real benefits, in terms of both lowering lubricant costs and reducing mechanical failures, for organizations that have large equipment inventories or fleets. However, even in the absence of such an experiential database, equipment operators can still benefit from general assessments made by experienced commercial laboratories based on FTIR CM data.

Current FTIR CM Procedures

Typically, representative oil samples are taken periodically from the equipment of interest and sent to a laboratory for FTIR analysis. As in all types of CM, sampling intervals are scheduled on the basis of the type of equipment, its operating conditions, the type of oil, and available historical data. At the laboratory end, samples are typically loaded into an auto-sampler tray, one of the strengths of FTIR spectroscopy being its ability to automatically analyze an oil sample "as is." The sample is pumped from the sample bottle, usually via a peristaltic or syringe-pump, into a 100 μm ZnSe transmission cell, and its IR spectrum is then collected in <1 min. Under software control, the specific absorbance or peak area measurements that have been defined for the various CM parameters [11] are then made, and the resulting information is exported to LIMS for reporting. The cell is subsequently rinsed with an oil-miscible solvent and re-loaded with the next oil sample and the process is repeated.

It should be noted that FTIR CM results are not necessarily comparable from one laboratory to another when ASTM Standard Practice E2412-10 is followed, as the latter allows for a certain amount of latitude in the instrument settings. For this reason, ASTM Sub-committee D02.96-03 has developed a new Standard Practice (ASTM D7418–07, Standard Practice for Setup and Operation of Fourier Transform Infrared (FTIR) Spectrometers for In-Service Oil Condition Monitoring [14]), which standardizes the instrument configuration and data collection protocol so that the spectral information collected for the same sample in different laboratories should be comparable. In addition, depending on the instrument manufacturer and its software, the algorithms (regions and baselines) used to measure the various FTIR CM parameters are not necessarily identical, and thus Sub-committee D02.96-03 has developed Refs 15–18 and is continuing to develop test methods associated with the practice to standardize these individual measures.

Analytical Issues

A major complicating factor associated with FTIR CM analyses is that data interpretation can be heavily dependent on the oil formulation because IR absorption bands of the oil may interfere with measurement of the spectral changes that are to be monitored in in-service oils over time. An illustrative example is provided by ASTM D7414-09, Standard Test Method for Condition Monitoring of Oxidation in In-Service Petroleum and Hydrocarbon Based Lubricants by Trend Analysis [16]. This method makes measurements in the carbonyl absorption region of the spectrum where organic esters and acids absorb. In terms of monitoring oxidation, such a measurement is valid in the spectra of purely hydrocarbon-based oils, but meaningless in the spectra of ester-based oils because the carbonyl absorption band of the ester linkage will swamp that of any organic acids and esters produced by oxidation. It is for this reason that the scope of the above ASTM method is restricted to "petroleum and hydrocarbon-based lubricants," as its title indicates. For similar reasons, ASTM E2412-10 addresses three lubricant categories individually [i.e., petroleum-based (crankcase) lubricants, extreme-pressure (EP) fluids, and polyol ester fluids] in relation to which CM parameters are meaningful and how spectral measurements (region and baseline) are to be made for these parameters. However, within any such category, there will be a wide range of proprietary formulations marketed by various lubricant suppliers as well as unique formulations designed for specialized applications, and these formulation differences will manifest themselves in the FTIR spectra of the oils, again complicating the interpretation of FTIR CM data. Within this context, two FTIR CM analytical approaches have evolved: Direct trend analysis and differential trend analysis, both of which have merits and limitations. Direct trend analysis works on the assumption that spectral changes over time that are superimposed on the spectrum characteristic of the oil in question can be tracked to develop an oil degradation profile and intervention criteria. Differential trend analysis is a more rigorous procedure that involves collecting the spectrum of an in-service oil sample and subtracting from it the spectrum of the corresponding "fresh" oil or other appropriate reference oil, such that the net spectral changes that have taken place are isolated in the difference spectrum [19]. This approach works well if the oil is well-defined and one can be assured that it has not been topped up with some other oil. Direct trend analysis is more practical, but less rigorous, good for the routine monitoring of, for example, oils obtained from a truck fleet, where the oils employed are generally similar. Differential trend analysis is more exacting and is more attuned to examining gear or compressor oils, where the oil formulation is quite specific, the same oil is expected to stay in use for long periods of time, and changes in moisture, oxidative status, and additives are important degradation indicators. However, this approach requires that the spectrum of the appropriate reference oil be available in a spectral database or recorded as part of the sample analysis protocol. Ultimately, neither the direct nor the differential approach is entirely satisfactory, the first from the standpoint of formulation dependence and the second from the standpoint of practicality. It should be noted that both these approaches are encompassed within ASTM E2412-10 [11] and ASTM D7418–07 [14] as well as in the associated test methods [15–18].

Broadening the Analytical Scope of CM

In light of the issues discussed above, it would be useful to devise a general spectral classification system for oils so that the selection of appropriate CM parameters and associated spectral measurements can be made solely on the basis of the spectral characteristics of the oil formulation. Although spectral searching and matching routines are addressed in general terms by ASTM E2310-04 [20], several challenges will have to be addressed in developing this concept. One of the major challenges is to achieve correct classification of in-service oils that have undergone chemical changes (e.g., oxidation, additive depletion, soot buildup, etc.), given that the corresponding spectral changes represent confounding factors for the classification. A potential means of addressing this challenge investigated by the present authors involves the use of region selection algorithms to optimize classifiers at each branch of a "decision tree;" however, further research will be required to validate this approach. If successful, oils could not only be grouped on the basis of their spectral similarities and categorized accordingly, but appropriate FTIR CM analytical protocols could be developed for each oil "category" and validated in terms of spectral regions where measurements could best be made for the appropriate parameters of interest. As noted above, ASTM E2412-10 specifies FTIR CM measurement parameters for only three classes of lubricants (petroleum lubricants, EP petroleum lubricants, and polyol esters), and although the possibility of encompassing other lubricant types as data becomes available is mentioned in ASTM E2412-10, it has yet to come to fruition. If a generalized oil classification system were to be devised for existing commercial formulations and updated as new ones come on stream, efforts to broaden the scope of FTIR CM to encompass a wider range of oils would be facilitated. Although it will be difficult to demonstrate the universal reliability of this approach to FTIR CM, given the very large number of lubricant products currently on the market, the use of a spectrally based classification system should provide safeguards against sample misclassification that are lacking in present-day FTIR CM. In addition, such a system could overcome the general confusion associated with trade names and "end-use" lubricant designations (e.g., ester versus mineral versus synthetic versus EP fluid versus compressor oil, etc.). Trade names and end-use nomenclature are used interchangeably in the industry but the products do not necessarily have a common denominator in terms of the base oil or additive package, which often leads to confusion as well as analytical anomalies. A spectral classification system encompassing a wide range of oil formulations from mineral to ester-based, and combinations thereof, as well as other oil types (e.g., phosphate esters, polyalkylene glycol oils, etc.) could ultimately provide a simplified cross-referenced chemical/functional categorization scheme for thousands of brand-name lubricants, providing lubricant purchasers with a useful means of identifying and selecting among alternative products for a given application.

CM Improvements

The comments above indicate that FTIR CM, although used extensively and cost effective, is still a technology that has significant room for improvement to make it a more utilitarian analytical tool. Aside from these considerations, a recent advance in FTIR CM has been the implementation of *spectral reconstitution*, a technique that has a major impact on sample throughput and solvent use. With this technique, the viscosity of the oil sample is reduced by diluting it with low-cost kerosene or odorless mineral spirits (OMS) containing a unique spectral marker, thus allowing the sample to be pumped more rapidly into the IR transmission cell and eliminating the need for a solvent rinse between samples [21]. The presence of the spectral marker in the diluent then allows the spectrum of the original neat oil sample to be obtained via spectral reconstitution by quantitatively stripping out the spectrum of the diluent; Figure 2 presents a schematic diagram of how spectral reconstitution works.

The spectral reconstitution technique has been implemented on Thermal-Lube's continuous oil analysis and treatment (COAT) System, an integrated auto-sampler and FTIR analyzer (Fig. 3) that uses a single low-dead-volume pump to load diluted samples into the IR transmission cell, with each sample being rinsed out by flowing the next sample through the cell. The fact that a spectral marker is present also allows the dilution to be approximate, the marker measure determining the precise dilution. Aside from the substantial

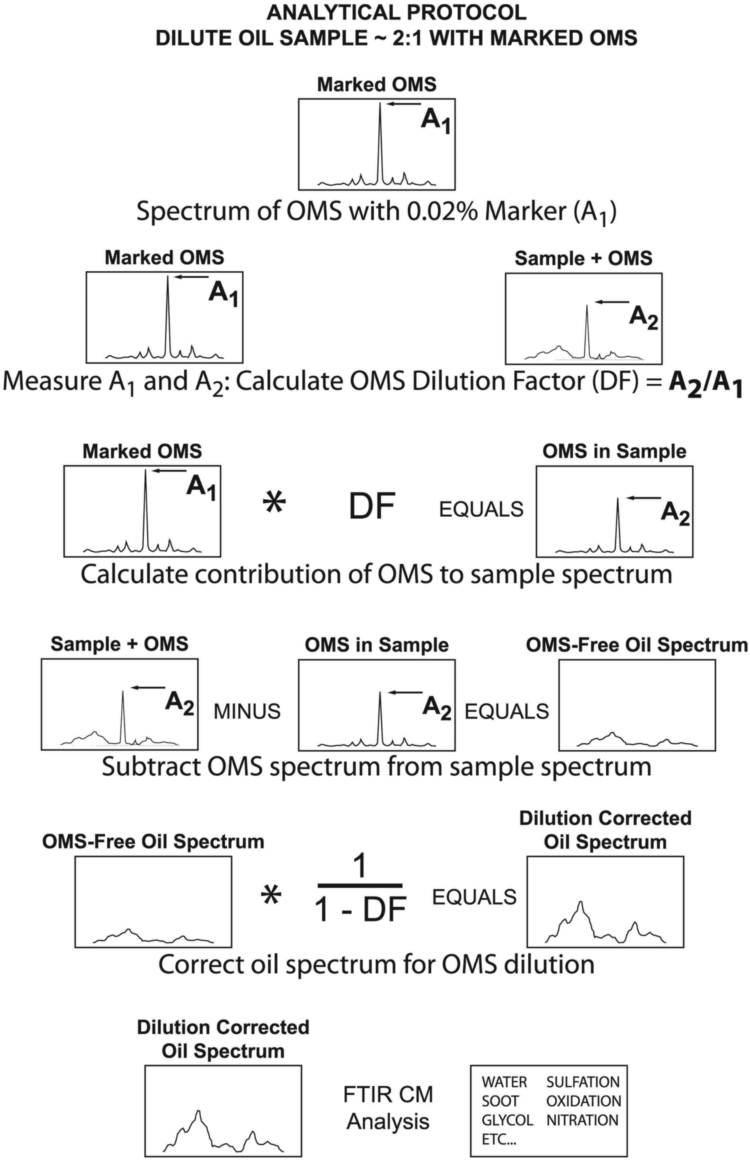

FIG. 2—*Schematic diagram showing the spectral manipulations required so that an oil sample that has been diluted with OMS or kerosene containing a unique spectral marker is mathematically transformed back into the spectrum of the original neat sample.*

analytical speed benefits conferred (up to 180 samples/h versus 20–30 samples/h with conventional peristaltic- or syringe-pump-based FTIR auto-sampler systems [22]), spectral reconstitution also minimizes cell wear, allows the use of low-cost KCl cell windows (versus ZnSe), and requires less sample and solvent,

FIG. 3—*The COAT system, an integrated auto-sampler and FTIR analyzer designed for lubricant analysis, capable of carrying out qualitative FTIR CM as well as analyzing oils quantitatively for AN, BN, or H_2O at throughput rates of up to 180 samples/h.*

thus minimizing the environmental impact associated with the oil and solvent waste generated. All the procedural elements of FTIR CM in accordance with ASTM D7418–07 are followed except that the neat oil spectrum is not measured directly but is instead reconstituted from the spectrum of a diluted sample. Extensive comparisons of CM data obtained under standardized conditions by the spectral reconstitution technique and by conventional FTIR analysis of the corresponding neat oil samples indicate that the two procedures effectively provide identical data [21].

Quantitative FTIR Analysis

As noted earlier, FTIR spectroscopy is not restricted to providing CM trending data but is also capable of delivering quantitative analytical data for key oil quality parameters, specifically AN, BN, and moisture (H_2O). For in-service oils, these three parameters are all important oil quality indicators that are routinely analyzed for and quantified, usually after CM screening procedures point to a potential problem with the oil. Depending on the application, lubricants in most non-combustion-related applications tend to acidify largely as a result of oxidative processes producing relatively weak organic acids while in most combustion applications, the acidity is contributed by both oxidation and blow-by gases, the latter producing strong acids (nitric, sulfuric). AN analysis tends to be restricted to low-ash oils, which do not contain acid-neutralizing base additive packages (e.g., compressor oils) as they slowly accumulate acidity by oxidation. BN analysis is associated with high-ash oils containing base additive packages (commonly known as detergents) to counteract more rapidly accumulating stronger acids. Thus, the formation of acids in these types of oils does not result in acid accumulation but rather in the loss of the reserve alkalinity, and it is this loss that is measured in BN analysis. Closely related to both of these measures is moisture, which, being both a reactant and a reaction medium, is closely linked to acidity-related corrosion as well as a variety of other detrimental changes that can take place in oils when it is present.

The ASTM methods for AN and BN and for moisture determinations, utilizing acid-base and Karl Fischer titration, respectively, are generally acknowledged to be problematic, of limited accuracy and reproducibility, and relatively expensive to carry out from the standpoint of equipment and reagents. For these reasons, substantial efforts have been employed to develop automated FTIR methods for these analyses that are simpler, less costly, and more reliable than the ASTM titrimetric procedures. As described more fully in the following paragraphs, this methodology development work has been an iterative process, aimed at simplifying the workable methods originally developed [23,24] as well as increasing sample throughput rates. In addition, measures of acidity and reserve alkalinity obtained by the FTIR procedures have been differentiated from the traditional AN and BN measures by being recast as AC (AC_{pK_a}) and BC (BC_{pK_a}), respectively, where the subscript represents the pK_a of the spectrally active base or acid employed in the FTIR determinations. AC and BC data are expressed in units of mEq acid/g oil and mEq base/g oil, respectively, but can be readily converted to the units of AN and BN (mg KOH/g oil); however, the values obtained will generally not match those obtained by ASTM titrimetric methods owing to the differences in the pK_a values of the "titrants" employed in the FTIR and ASTM methods

Acid Content

The ASTM methods for the determination of AN, such as the widely employed ASTM D664-09a [25], are designed to measure acidity accumulation in a range of oil and lubricant products, such as the accumulation of acidic combustion by-products and acids formed by oxidation, or to measure corrosive naphthenic acids commonly found in crude oils [26]. For FTIR AN determinations, the original method [23] was based on the stoichiometric reaction of a spectrally active base, potassium phthalimide, dispersed in oil-miscible 1-propanol, with acids present in the oil. The calibration was developed by reacting the potassium phthalimide reagent solution with 4-nitrophenol and measuring the absorbance of the product (neutral phthalimide), which exhibits an intense band at

1727 cm^{-1}. In the analysis of oils, any interfering oil absorptions were accounted for by preparing two samples: One with the active reagent (potassium phthalimide in *n*-propanol) and one with a blank reagent (1-propanol). Although workable, this dual-sample procedure is clearly cumbersome, and consequently subsequent work focused on eliminating the requirement to prepare sample blanks. This objective was met by selecting sodium hydrogen cyanamide (NaHNCN) as the spectrally active base [27]. Because NaHNCN, unlike potassium phthalimide, could be solubilized in a solvent (ethanol) that is immiscible with most oils, the need to prepare a sample blank to compensate for spectral interferences associated with the oil matrix was eliminated for most types of samples. NaHNCN (pK_a of ~ 9.5) is basic enough to react fully with all organic (carboxylic) acids and stronger acids that may be present in oil samples. As a result of the acid-base reaction, the $\nu\text{C} \equiv \text{N}$ band of NaHNCN, which is observed at 2109 cm^{-1}, is lost in proportion to the total amount of acid present. Any organic acids present are converted into their carboxylate salts (COO^-) and measured separately at 1570 cm^{-1}. Accordingly, this FTIR method not only measures acidity but also differentiates between the types of acidity present, providing additional information that may be useful in assessing the corrosion potential associated with the acidity in the oil. Oleic acid, a carboxylic acid and hence considered representative of the weakest acids found in in-service oils that are of significance from a quality standpoint, is used for calibration, standards being prepared by direct addition of this acid to the NaHNCN/ethanol reagent solution. The calibration equations are formulated in terms of unit acidity (mEq acid/mL of reagent solution) to make them universally applicable (i.e., independent of the sample preparation protocol); in the analysis of oils, the values of total and organic unit acidity obtained from the FTIR analysis are each multiplied by the volume of reagent solution added to the oil sample and divided by the weight of the oil sample to yield total AC and organic acid content (OAC) in units of mEq acid/g oil, with any difference between AC and OAC being considered to be the inorganic acid content.

Implementation of the FTIR AC method on an auto-sampler-equipped spectrometer allowed for the automated analysis (after sample preparation) of up to 180 samples/h, representing a significant increase in analytical throughput relative to autotitrators. A comparison between the FTIR results (multiplied by the molecular weight of KOH to express them in the same units as AN) determined for 32 in-service oils and their AN values, determined by ASTM D664-09a, is shown in Fig. 4 and yielded the following linear regression relationship:

$$(AC_{9.5} \cdot MW_{\text{KOH}}) = 0.06 + 0.72 \cdot AN_{\text{titration}} \quad SD = 0.14 \quad R = 0.985$$

These results indicate that although the FTIR method produces lower values than KOH titration, which is attributable to its use of the weaker base NaHNCN, there is a good correlation between the FTIR and titrimetric results.

A somewhat similar FTIR AC method is being developed specifically for phosphate esters, commonly used as fire-retardant hydraulic fluids in power-plant compressor hydraulic speed control systems [28] and other high-temperature applications. These esters develop acidity predominantly via

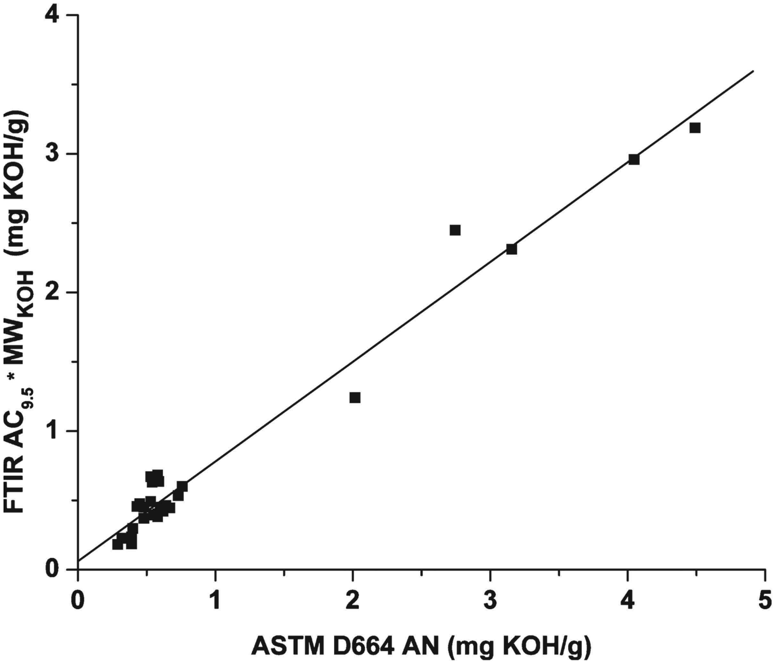

FIG. 4—*Plot of FTIR AC results for 32 in-service oils versus AN data obtained by ASTM D664-09a potentiometric titration. A plot of the same data was previously published in Ref 27 (note: In the latter publication, FTIR AC values were reported in units of mg KOH/g rather than mEq acid/g).*

hydrolysis. There is great interest in differentiating between the more weakly acidic partial hydrolysis products and the stronger and more corrosive phosphoric acid. Acid formation is the primary contamination parameter for this type of fluid. By using carefully selected IR-active bases of known pK_a, the FTIR AC method is capable of differentiating between these hydrolysis products (F. R. van de Voort and T. Yuan, manuscript in preparation [29]). Thus, in general terms, FTIR AC methodologies are not only rapid and automated procedures but are also capable of providing more specific information in terms of the nature of the acidity.

Base Content

As noted earlier, BN measurements are designed to measure the reserve alkalinity in lubricants. Overbased detergents are common additives in oil formulations destined for combustion engines, with the amount of base (usually metal carbonates) incorporated being a function of the acidity the lubricant is expected to encounter. Thus, marine engines, burning high-sulfur bunker C crude, require high levels of base formulated in their crankcase oils to counter the acidity generated as the fuel is burned, while for truck diesel engines burning low-sulfur diesel, the base requirement is much more modest. In addition to detergents, dispersants also tend to have basic characteristics and these may also contribute to the measured BN of an oil, depending on the BN method used. ASTM D2896-07a [30], which uses perchloric acid, is considered to measure all basic materials present whereas ASTM D4739-08e1 [31], which uses HCl, is considered to predominantly measure the metal carbonates in the detergents, but little, if any, of the dispersant contribution. This distinction is not necessarily adhered to, such that in-service oils are often analyzed by either method, leading to some confusion in relation to inter-laboratory results obtained for the same sample analyzed by different methods. For the FTIR analysis, trifluoroacetic acid (TFA) in 1-propanol has been selected as the spectrally active acid employed to react with the bases present in oils [23]. Because TFA has a pK_a of $\sim$0.5, it is strong enough to react with metal carbonates (e.g., $CaCO_3$), but not with weaker basic dispersants. Like the FTIR AN method, the original FTIR BN method [23] required the preparation of two samples to obtain a single BN measure. This method has recently been upgraded to a single-sample procedure (S. Ehsan, J. Sedman, F. R. van de Voort, E. Akochi-Koblé. T. Yuan, and D. Takouk, manuscript submitted for publication [32]) with its measurement being recast as BC to differentiate it from the traditional BN measure. The base employed for calibration has been changed from dodecylamine to 1-methylimidazole, which is a liquid and therefore easier to handle. As in the original method, the reagent solution employed is TFA in *n*-propanol. The use of a more polar solvent such as ethanol caused detergents present in the fluid to coalesce and form a gel, preventing the acid-base reaction from going to completion. Even with the 1-propanol reagent solution, this problem was highly prevalent with new high-BN oils and additives but was overcome by diluting the oil sample with heptane. In contrast to the AC method, in which matrix effects are minimized owing to the immiscibility of most oil samples with the reagent solution, the BC method relies on the use of second derivative spectra computed using a gap-segment algorithm to minimize spectral interferences. The latter algorithm, which has been extensively employed in near-IR spectroscopy [33], incorporates smoothing into the derivative calculation and therefore provides second derivative spectra with superior signal-to-noise ratios compared to conventional second derivative spectra (computed by the simple first-difference approach). In addition, because quantitation of BC is based on a universal calibration in terms of unit basicity (mEq base/mL of reagent solution), the spectra collected for samples are mathematically corrected to compensate for oil miscibility with the reagent solution by employing a spectrally determined dilution factor. Validation studies of this single-sample BC method conducted to date have indicated that the use of a universal calibration in combination with a spectral dilution correction and a gap-segment second derivative is a viable means of obtaining quantitative results, without necessarily having to resort to the use of an oil-immiscible solvent to aid in minimizing oil spectral interferences. Figure 5 presents typical results of these ongoing validation studies, yielding the following relationship between the FTIR $BC_{0.5}$ results (multiplied by the molecular weight of KOH) and BN values obtained by ASTM D2896-07a (perchloric acid titration) for a set of 18 in-service oils

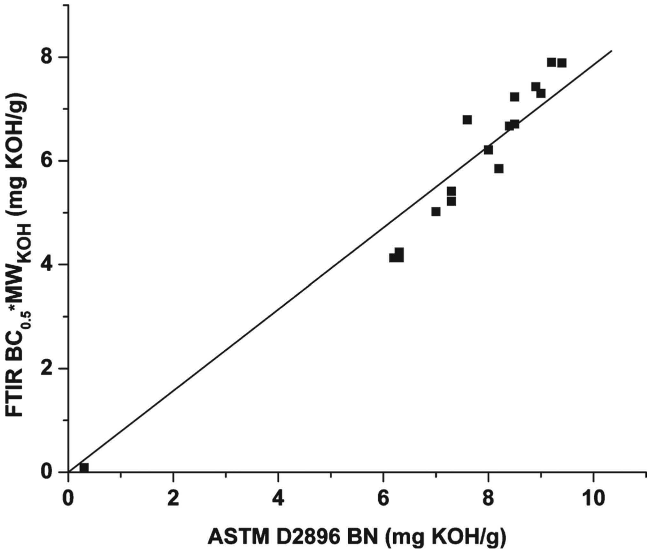

FIG. 5—*Plot of FTIR $BC_{0.5}$ results for 18 in-service oils versus BN data obtained by ASTM D2896-07a potentiometric titration.*

$$(BC_{0.5} \cdot MW_{\mathrm{KOH}}) = 0.79 \cdot BN_{\mathrm{D2896}}; \quad SD = 0.52; \quad R = 0.978$$

Given the use of a much weaker acid in the FTIR method, the fact that the slope of the regression line (forced through the origin) is <1 is to be expected for samples that contain weak bases, while the correlation between the FTIR and titrimetric results is surprisingly good. In this regard, it may be noted that the correlation coefficient is only reduced slightly (to 0.966) when the two samples falling below the BN range of 6–10 mg KOH/g are excluded from the regression.

Moisture

The third FTIR method of consequence is the measurement of moisture in lubricants based on a simple acetonitrile extraction procedure [34]. The premise is that most hydrophobic lubricants are largely immiscible with polar acetonitrile. The water can be extracted from the oil and readily be measured spectrally from a differential spectrum obtained by subtracting the spectrum of dry acetonitrile from that of the acetonitrile extract obtained from the oil. It was been found that this simple extraction procedure tracks Karl Fischer moisture determinations very well and is reproducible and adequately sensitive. Like AN and BN analyses, this procedure, in its automated form and implemented on the COAT system, can attain FTIR throughput rates of up to 180 samples/h after sample preparation. A moisture calibration is developed simply by adding defined amounts of water to dry acetonitrile. Since its development, this methodology has been generalized and improved significantly, in particular from the standpoint of the determination of the absolute moisture content of acetonitrile through the addition of D_2O. The use of D_2O is workable because acetonitrile is an aprotic solvent and hence does not undergo any H–D exchange. Thus, any H_2O present in the acetonitrile will fully exchange with an excess of added D_2O to produce HOD, quantitatively, thereby eliminating the 1630 cm^{-1} band arising from the H–O–H bending vibration. Thus, measurement of this band in a differential spectrum obtained by subtracting the spectrum of the D_2O-treated acetonitrile from that of the acetonitrile prior to D_2O addition provides an absolute measure of the moisture present in the acetonitrile. Not only does this provide knowledge of the moisture content of the extracting solvent, but this concept can be taken further to provide absolute moisture contents in oils. Further generalization of the method includes the ability to work with samples that are partially miscible with acetonitrile, using measurements of the intensity of an acetonitrile overtone band to compensate for density and/or dilution effects and, as in the BC method, employing the gap-segment second derivative to minimize spectral interferences.

Principles of Quantitation

There are some common denominators associated with how quantitation is achieved in the FTIR methods for the determination of AC, BC, and moisture, albeit with some differences in the details, which will not be elaborated on here.

One of the key principles is that the calibrations are all simply based on adding various known amounts of the component of interest [i.e., a representative acid (oleic acid), a representative base (1-methylimidazole), or H_2O] to a reagent solution (AC and BC calibrations) or a pure solvent (moisture calibration) and obtaining a linear regression equation relating the parameter in question (AC, BC, or moisture) to a spectral response at a single wavelength in the absorbance or second derivative spectra of these calibration solutions. Such calibrations are very easy to develop and understand and independent of sample characteristics. Critical to the application of these calibrations in the analysis of oil samples is the capability to correct for any dilution or change in density of the reagent solution/solvent, the extent of which will depend on the degree of miscibility of the sample in question, or particular components therein, with the solvent employed. Thus, while the correction for a predominantly immiscible sample would be negligible, a miscible sample would displace a very significant portion of the solvent and hence a substantial correction must be made to compensate. This correction is accomplished by employing a weak solvent overtone band as a measure of the “amount” of reagent solution/solvent in the cell and determining the correction factor (spectral multiplier) that will make the intensity of this band equal to its intensity in the spectrum of the reagent solution/solvent employed in the calibration procedure; when this correction factor is then applied to the spectrum, one is effectively “filling” the IR cell with the reagent solution/solvent. This procedure facilitates the use of these simple universal calibrations, devoid of sample spectral contributions. Of course, any miscibility brings with it some potential spectral contributions from the sample, especially if the sample is highly miscible; however, even in the latter case, the sample spectral contributions can be minimized through the use of a gap-segment second derivative rather than relying on the conventional differential spectrum used in the dual-sample FTIR methods. In general terms, one can be confident in the results of the rapid single-sample procedures for AC, BC, and moisture content when the miscibility of the sample with the reagent solution or extraction solvent is low, which is generally the case, and employ the dual-sample procedures when very precise data are required for samples that are significantly miscible with the reagent solution or extraction solvent, which can make spectral interferences problematic.

Methodology Summary

A unifying principle associated with the FTIR AC, BC, and moisture content methods is sample viscosity reduction, either through sample dilution or by partitioning the component of interest into a low-viscosity solvent. This facilitates rapid sample throughput without the need for cell rinsing between samples. All methods are based on simple spectral measurements directly related to changes associated with the component(s) in question. UMPIRE-PRO (Universal Method Platform for Infra Red Evaluation) software executes all spectral data manipulation, analyzes the data, and exports the results in various compatible formats. The use of universal calibrations renders all the quantitative methods straightforward to calibrate and implement, drawing on simple Beer's Law principles rather than often poorly understood black-box inputs and outputs associated with more advanced chemometric procedures. In the case of spectral reconstitution procedures used for CM, the accurate assessment of the diluent and spectral marker concentration allows one to obtain a facsimile of the neat oil spectrum without having to pump a viscous sample through the IR cell, therefore facilitating higher speeds and minimizing the wear and tear on the IR cell windows. In addition, all these novel FTIR methods confer the benefits of automation and high sample throughput, while minimizing reagent use and oil waste.

Role of ASTM

As a voluntary organization, the ASTM is driven by the mutual interests of the parties involved to have available peer-reviewed methods that are reliable and workable in the hands of the users. FTIR CM analysis of lubricants, pioneered by the JOAP, is still in the process of being standardized and validated by the D02.96-03 IR and FTIR sub-committee. Long-standing ASTM wet-chemical methods for the determination of AN, BN, and H_2O have seen only minor changes over the past 30 years. Thus, the FTIR methods discussed in this paper represent a significant innovation, and bringing them to fruition by formulating them as standardized ASTM methods is the next challenge. These methods must then undergo inter-laboratory testing to determine whether consistent and valid results can be obtained and to ensure that they will perform reliably in the hands of many users and under a wide range of conditions. Given that FTIR spectrometers are ubiquitous in modern CM laboratories worldwide, the infrastructure is available for the implementation and testing of the methods in independent laboratories following simple, rigorous, and manual protocols. Once the methods have been tested and proven, automation can be implemented to attain the higher throughputs already available on systems like Thermal-Lube's COAT System, which has been designed using the concepts presented here.

Conclusion

FTIR spectroscopy is an important automated qualitative CM screening tool for in-service lubricant analysis. This screening methodology has been advanced in terms of sample throughput through the use of spectral reconstitution, allowing up to 180 pre-prepared samples to be analyzed per hour. Although CM is presently limited largely to mineral based oils, it is foreseen that direct trending can be broadened to a much wider range of basestocks and formulated oils by bringing to bear new oil identification/classification capabilities and adjusting the measurement regions and parameters accordingly. More importantly, the most common quantitative follow-up analyses, AN, BN, and moisture content, have also been developed into viable automatable FTIR methods, significantly extending the utility and analytical role of FTIR spectroscopy in relation to lubricant analysis. AN and moisture analyses, specifically, may well have wider application beyond lubricants per se, and will likely prove to be applicable to a wide range of hydrophobic materials, including fuels (diesel, gasoline, and biodiesel), biodiesel feedstocks, and crude oils. Whether FTIR quantitative methodology will be accepted by the broader lubricant analytical community and the ASTM will depend to a large degree on the desire of commercial CM laboratories to take advantage of the economies of scale offered by this technology.

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Particle Characterization and Sizing: SEM Utilizing Automated Electron Beam and AFA Software for Particle Counting and Particle Characterization

ABSTRACT: In recent decades oil analysis laboratories have used automatic particle counters to determine the number and size of particles in many oil wetted systems. Once determined, there has been the question of "what are they?" Logically, the next step is to investigate the make-up of the particles by extracting the particles and analyzing using emission spectroscopy and/or a filter membrane for view them under magnification. Characterizing the make-up of the particles is very useful in determining the source. A cost-effective action plan can be undertaken to rectify the identified problem once the particle type is determined. The problem with these two approaches is; emission spectroscopy analysis is often unreliable in identifying larger particles and; under optical magnification it is not possible to tell for sure the makeup of the particles. In some cases scanning electron microscopy (SEM) analysis is undertaken to further identify the particles. This proves to be time-consuming for the SEM operator and is left to the operator to judge which areas of the sample to investigate. This study illustrates the performance and innovative approach to resolving the aforementioned limitations on sizing and identifying particles using an "Automated Electron Beam Particle Analyzer" equipped with "Automated Feature Analysis" (AFA) software to characterize complex matrix of particles extracted onto a membrane filter from oil samples.

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KEYWORDS: particle count, wear metals contamination, electron microscope

Introduction

In recent decades oil analysis laboratories have used automatic laser particle counters and pore blockage counters to determine the number and size of particles in many oil lubricated systems. A point of interest, once the particles are counted, is their composition and morphology. Logically, the next step is to extract the particles and analyze them using emission spectroscopy and/or a filter membrane to review under magnification. Characterizing particles by shape and elemental composition is very useful in determining the source. A cost-effective action plan can be undertaken to rectify the identified problem once the particle type is determined.

Two problems with this approach are:

(1) The emission spectroscopy is often unreliable in identifying larger particles; and

(2) Under optical magnification it is not possible to tell for sure the elemental composition of the particles.

In some cases SEM analysis is undertaken to further identify the particles. This proves to be time consuming for the SEM operator and is left to the operator to judge which areas of the sample to investigate.

This report illustrates the performance and innovative approach to resolving the aforementioned limitations on sizing and identifying particles using an “Automated Electron Beam Particle Analyzer” equipped with “Automated Feature Analysis” (AFA) software [1] to characterize complex matrix of particles extracted onto a membrane filter from oil samples.

Methods

The scanning electron microscope and its automated feature analysis function combine the high magnification capabilities of an electron microscope and simultaneously supported energy dispersive X-ray measurement system with the capability of automating the searching and recording of data for future reference. The process is done in four steps: (1) Sample preparation, (2) automated data acquisition, (3) analysis and configuration of the data, and (4) processing the results into a useful report.

(1) Preparation is accomplished by taking a representative amount of the thoroughly homogenized sample and diluting it in a suitable solvent. Some prior knowledge or screening of the sample should be done to determine the best volume of sample to use (usually 1 to 3 mL) to optimize the capability of the automated functions. The diluted sample is drawn through a low contrast filter of appropriate pore size, such as a 0.8 μm polycarbonate filters (Fig. 1). Extra care is taken in the handling, indexing and storing of the filter so that the specimen can be reloaded at a later date, if desired, and the same core automated data can be used to search out and further analyze particles.

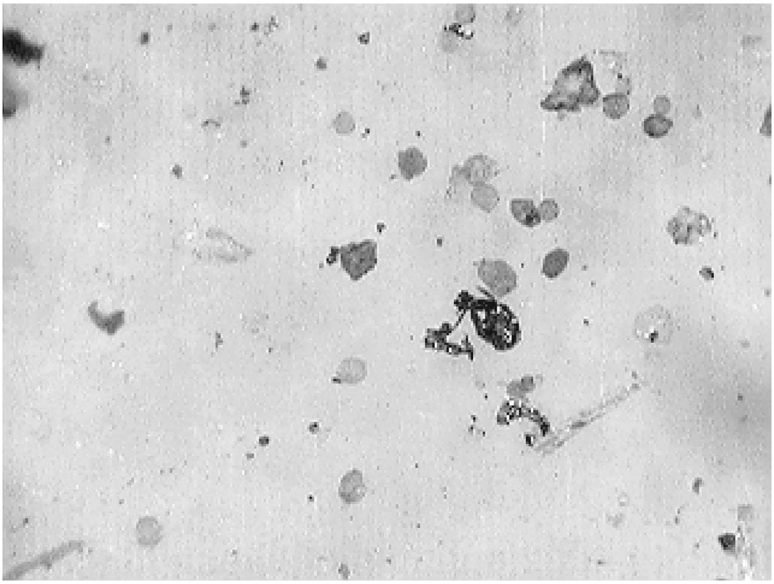

FIG. 1—*Sample prepared on filter for SEM/AFA (~30× optical magnification).*

(2) The automated data acquisition system records the spatial distribution of the back-scattered electron intensity from the sample. Back-scatter electrons are electrons which are scattered elastically (i.e., no change in their kinetic energy) from the atomic nuclei into the backward hemisphere. At any given primary electron beam energy, the coefficient for back-scatter efficiency depends solely on the atomic number, thus providing a contrast mechanism for differentiating between light and heavy elements. Electron microscopes image items by detecting contrast in signal intensity between adjacent pixels in a raster image. Unlike a digital camera, SEM's operate sequentially, scanning rows (Fig. 2 left

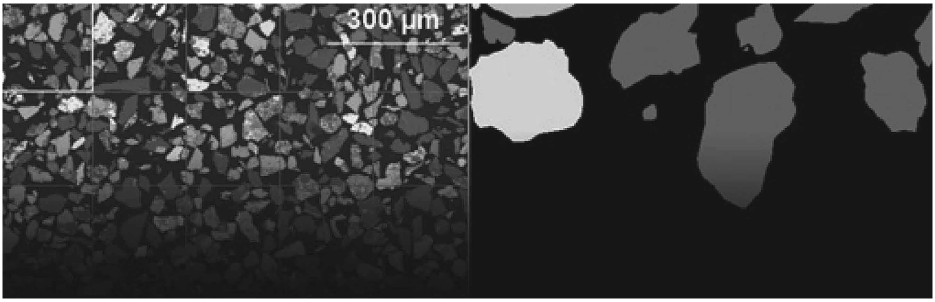

FIG. 2—*Computer program raster's across filter (left image). The computer stops at each particle, increases magnification, then measures and analyzes each particle (right image). Source: ASPEX Corporation.*

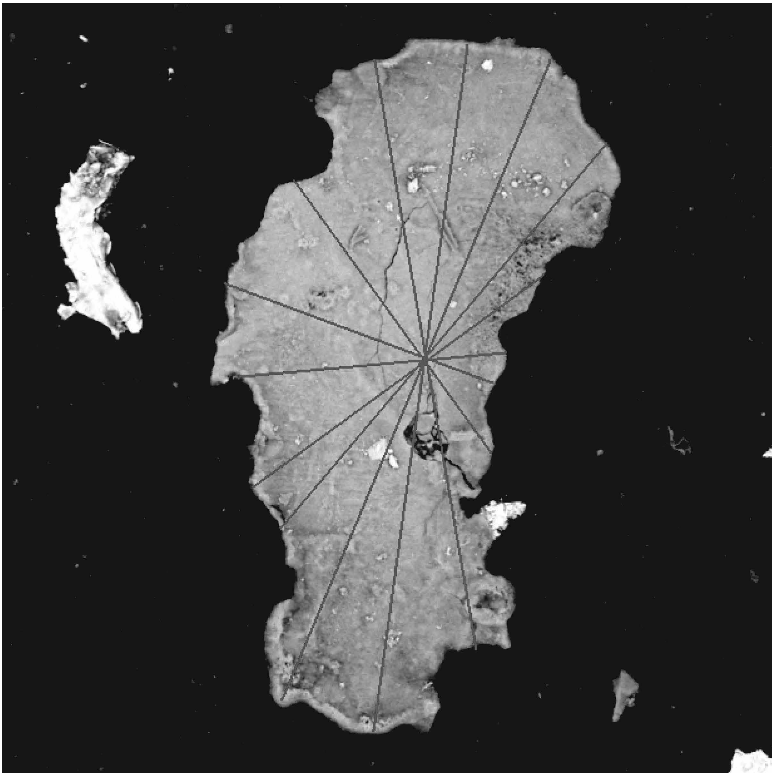

FIG. 3—*Rotating chord analysis. 16 chord illustration of how RCA determines the size and shape of a particle. Only 1/2 of cords are shown in this figure for illustrative purpose. The actual particle size is* ~100 *μm wide and* 200 *μm long.*

image) of pixels and stop at each pixel to acquire a signal (Fig. 2 right image). The intensity of that signal increases or decreases as the scan encounters elements of different atomic number. The system automatically scans the area of the filter we select at a rapid rate and pause at each particle it detects and shifts to its highest resolution to perform a "rotating chord algorithm" using 16 straight line scans (Fig. 3), each rotate approximately 11° from the last. The lines drawn (cords) on the images of the particles are for illustration only. The algorithm yields a plethora of information about the shape and size of each particle (Table 1 and Figs. 3–7). The search portion of the process can be set to a dimension that will find all the smallest particles of importance without spending extra time searching for smaller particles. (For example, it can be set to find all particles of 4 μm or greater without necessarily looking for all the 1 μm particles.) The search and measure portion of the automated process is extremely fast, up to 33 000 particles per hour.The process can be limited to measuring particle morphology and generalized

TABLE 1—*Definitions (41 categories are available; source—ASPEX Corp.).*

Name	Unit	Description
Dave	μm	The average length of the sixteen chords through the feature centroid
Dmax	μm	The length of the longest of the sixteen chords through the feature centroid (image #1)
Dmin	μm	The length of the shortest of the 16 chords through the feature centroid (image #2)
Dperp	μm	The length of the chord perpendicular to the longest chord (image #3)
ASPECT RATIO	1	The ratio of DMAX/DPERP
AREA	μm^2	The area of the feature
PERIMETER	μm	The perimeter of the feature as measured from one chord end to the next (image #4)
ORIENTATION	Degrees	The orientation of the longest chord. Zero is as noon and the angle increases clockwise.

Note: Dave, Aspect Ratio, Area, and Orientation are not illustrated in this paper.

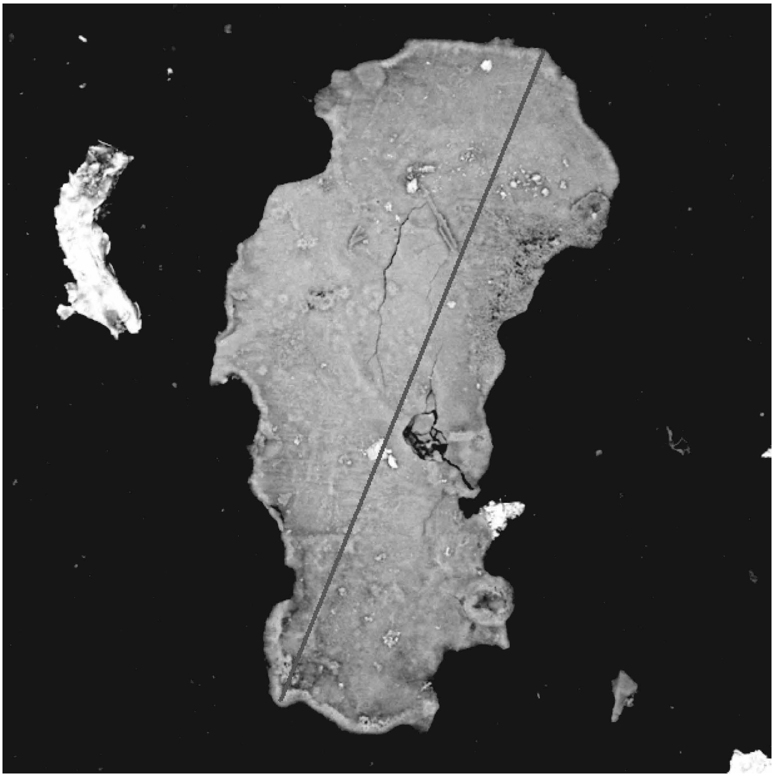

FIG. 4—*DMAX—the length of the longest of the 16 cords through the features centeroid.*

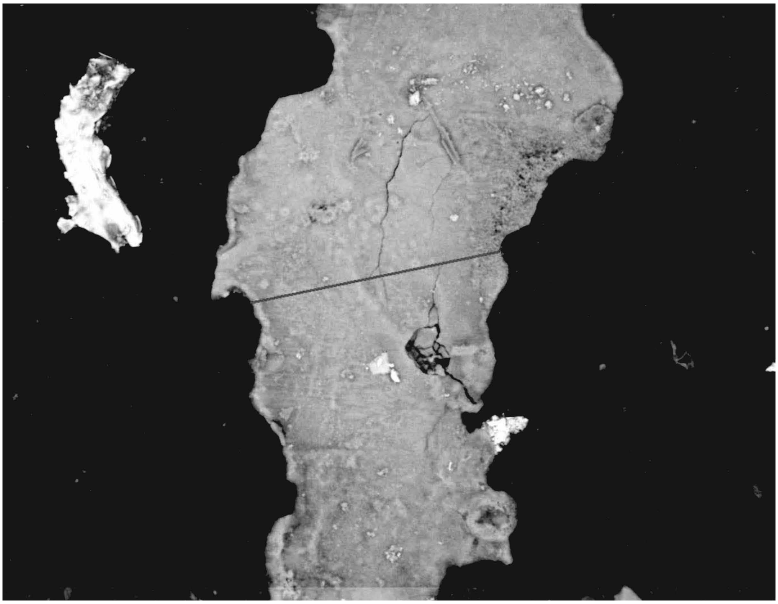

FIG. 5—*DMIN—length of the shortest of the sixteen chords through the features centeroid.*

criteria can be used to differentiate the elemental composition of the particles by simply segregating the various levels of signal intensity, the video level. This will answer the general question of heavier/more dense compounds vs. the lighter ones, however it does not provide a definitive identification of the elements involved. A huge gain is made in the quality of the information by combining the use of X-ray spectroscopy with the electron imaging. The X-ray signal generated from the target particle specifically identifies the elemental composition of the particle, nevertheless it takes more time to acquire the data from each particle. Whereas a typical 10 μm round particle takes the system 10ms to measure, the analysis with X-ray data collection averages around 4 s per particle. It takes approximately 10 min to collect all the particle data without X-ray analysis for a sample with 1 000 000 particles per 100 mL. It would take more than 11 h to analyze 100 % of the same sample with X-ray data. The X-ray data's absolute identification of chemical composition is a hugely important addition to the quality of the information; a compromise is generally achieved to correlate a percentage of the sample data to the total. If 10 % of the specimen is analyzed in a random manner it can be completed in about 1 h and 40 min and the data extrapolated to 100 %. An additional benefit of slowing the process down is that it can also record an image of every identified particle at the slower speed.

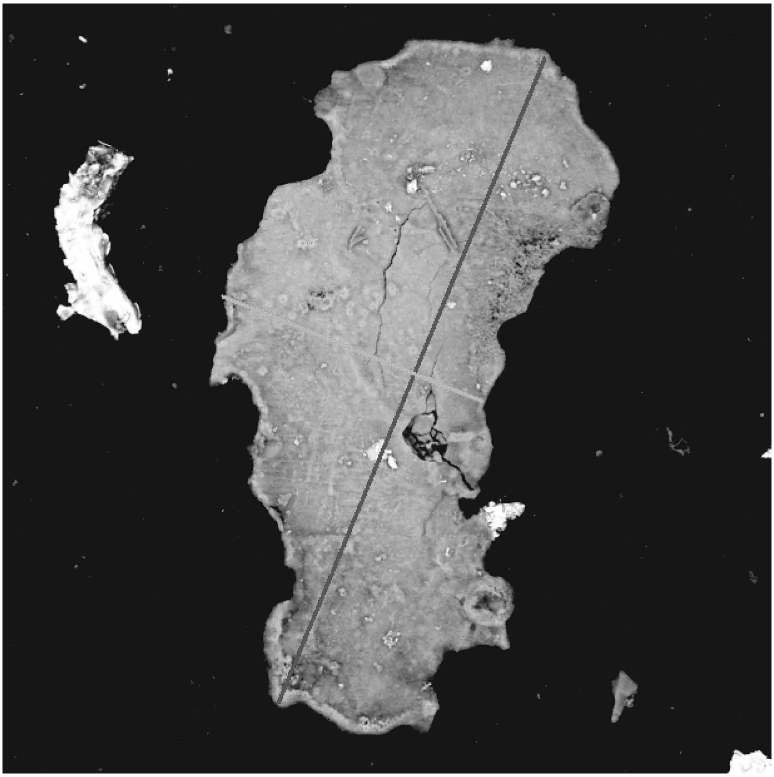

FIG. 6—*DPERP—the length of the cord perpendicular to the longest chord.*

(3) At this point the data is ready for review and assortment into classifications that describe the dominant characteristics of the specimen. These classifications can be based on morphology, chemical composition, and combinations of both. Four software programs are operated in conjunction with each other to view all the collected data, establish classification "rules," apply the rules to the data to organize it into an interpretable structure, and view specific particles for more detailed imaging and analysis. This portion of the process is done manually with the analyst applying specific knowledge of the specimen and customer requirements to arrive at the most appropriately descriptive and useful results. Experience in Tribological systems is of considerable assistance in formulating classification rules to optimize the process. This expertise is applied to this analytical process. The rules and structure applied to this analysis are archived and can be revisited later if revisions to the methodology are desired.

Rules for Particle Characterization

As an example we can use Case Study 1 (rules are shown in Table 2). After scanning the filter it is determined that the majority of the particles are in these

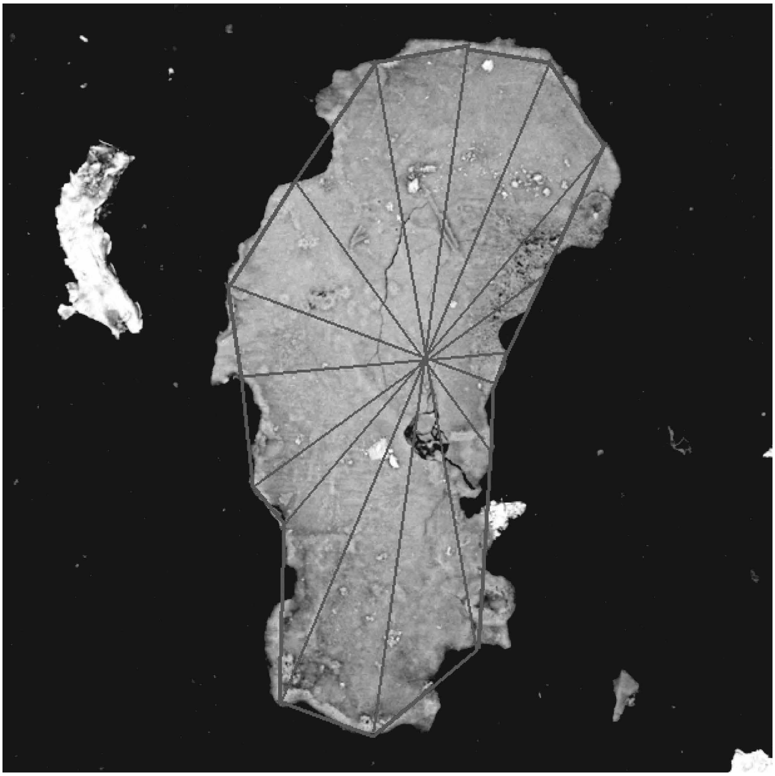

FIG. 7—*PERIMETER—the perimeter of the feature as measured from one chord end to another.*

classifications. As a result, the rule is made requesting both number and size of the particle containing the elemental proportions in the Rule.

The final step in the process involves taking the finished analysis and applying various publishing software to present an understandable and useful report.

TABLE 2—*Rules particle classification case study 1.*

Classification	Rule	% Content
Stainless steel	Fe > 30% and Cr > 5%	1.7
Iron	Fe > 30%	33.9
Tin	Sn > 30%	49.0
Silicates	Si > 5%	1.4
Brass	Cu + Zn + Sn > 35%	6.6
Sodium	Na > 10%	2.6
Miscellaneous	All remaining particles	4.8

TABLE 3—*Guaranteed performance characteristics (performance as measured using Performance Grading Software^{TM} for features 1 to* 100 *µm); source—ASPEX Corp.*

Particle detection efficiency	Greater than 99 %
Particle sizing precision	0.25 µm or better
Particle sizing accuracy	0.50 µm or better
Occurrence of false positives	Less than 1 per mm^2
Particles sized per hour	Up to 33000
Particles characterized per hour	Up to 1800

The data is presented in graphs, tables, electron images and X-ray spectra, which represent useful tools for the customer. The report can be standardized for use in ongoing trend analysis and in comparison to data from other similar equipment. Portions of the data also can be presented in spreadsheet format for the customer to insert in his or her ongoing data analysis system. Table 3 provides “Guaranteed Performance Characteristics.”

Case Study 1

Customer: Nuclear Power Plant

Description of Sample: Oil collected from Containment Spray #1-#1 Motor Inboard journal bearing, ID: 3485 mm, 6:31:23 a.m. MRSR# DB-2612

Background: Due to the critical nature of the application, the client collects samples from the Containment Spray Motors about every month. The standard oil test package includes metals analysis, particle count and physical properties. The particle count for this motor bearing oil is consistently higher than all of the other motors of the same make and model (ISO Code 4406 = 21/18/14 versus 16/14/10). There is no indication of silicon or wear metals by the emission spectroscopy. It is very important for the root cause investigation to determine what these particles are along with their sizes. The decision is made to employ the SEM/AFA to resolve any doubt in the analysis.

SEM Observing conditions: The specimen is observed under reduced vacuum at 15keV of beam energy to perform AFA on a non-conductive substrate. Working distance is approximately 16 mm to facilitate X-ray spectroscopy.

General observations: Approximately 9.5 % of the surface area of the filter is analyzed with the automated process. A total of 5618 particles are identified and classified by elemental composition and size. This extrapolates to 1 182 740 total particles per 100 mL.

Most debris in the sample is tin or iron (Table 2): The dominant tin material displays as a range from nearly pure tin to various brass-like alloys with copper, zinc, and lead. The iron-dominant particles are mainly a low chromium alloy or oxides, however about 1.7 % of the iron particles contain greater than 5 % chromium.

TABLE 4—*Number and percent of particles.*

Class	Total	
Fe, Cr	335	16.08 %
Iron rich	1270	60.97 %
Al	120	5.76 %
Si, Mg	15	0.72 %
Si Rich	37	1.78 %
Silicates	85	4.08 %
Na	58	2.78 %
Misc.	163	7.83 %
Total	2083	100 %

Summary of Results: This data from the nuclear power plant shows the journal bearing is wearing, producing large babbitt and iron wear particles. The data above suggests the bearing may be damaged due to too thin an oil film.

Case Study 2

Customer: Coal Fired Power Generation

Sample description: Gear oil from Pulverizer Gear Box, ISO VG 150

Sample preparation: 20 mL of thoroughly mixed oil is diluted with heptane and drawn through a 0.45 μm nitrocellulose filter, followed by thorough rinsing with heptane.

Analysis parameters: 100 % of the exposed filter area is searched with a criteria to find all particles of 4 μm and greater in dimension. 15 keV beam energy and reduced vacuum is used with a backscatter element detector.

Summary of findings: A total of 2083 particles are identified on the filter. Eight general categories of particles are identified based on their elemental composition. The criteria are selected based on the dominant patterns of chemical distribution in the 2083 particles (Table 4).

Mining the Data

The table below (Table 5) shows key parameters for a typical analysis. This table illustrates that one can easily sort by parameters of interest. In this case, we sorted on Dmax (descending). It is clear the particles are:

(a) Fairly oblong or rectangular due to their ASPECT of less ~2 on average; (Table 5)
(b) The particles are primarily iron and silicates; (Table 4)

Summary of Results

The concentration of silicon and silicates (6.58 %) leads to the conclusion the gears were wearing due to three body abrasion by contaminating abrasives.

TABLE 5—*Morphology analysis of data in Case Study 2.*

Particle Number	Dave	Dmax	Dmin	Dprep	ASPECT	AREA	PERIMETER	Primary Element	2nd Element	% of 1st	% of 2nd
156	108.3	134.4	90.05	102.4	1.313	9243	368.4	Fe		100	0
191	78.49	1.06.3	54.72	66.87	1.59	4962	321.2	Si	Mg	60	40
180	75.34	96.96	54.01	72.23	1.342	4491	315.4	Ca		100	0
158	54.22	93.47	38.3	40.71	2.296	2552	243.5	Fe		100	0
207	61.94	93.11	43.7	45.87	2.03	3120	245.3	Si	Mg	60.8	39.2
57	43.74	76.42	23.56	34.95	2.187	1556	209.4	Si		100	0
273	59.1	75.36	49.92	64.64	1.166	2738	239	Si	Mg	62.3	37.7
277	38.84	66.72	5.16	14.08	4.737	1351	308.3	Fe		100	0
64	44.45	66.61	31.65	43.6	1.528	1566	183.2	Fe		100	0
218	47.55	66.02	33.91	37.03	1.783	1799	198.8	Si		100	0
240	36.24	65.46	16.13	25.26	2.592	1127	234.9	Fe		100	0

Conclusion

The SEM/AFA analysis offers the analyst an innovative method compared to conventional particle analysis methods for determining particle size and elemental composition In oil samples. The method described can save industry money and end speculation about the source of the particles.

Acknowledgments

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Stuart Lunt[1]

Recent Developments in Online Oil Condition Monitoring Sensors and Alignment with ASTM Methods and Practices

ABSTRACT: Oil condition monitoring is a vital part of integrated asset health management. With an increasing impetus toward real-time decision making, delays incurred in offline laboratory oil analysis are becoming less acceptable. At present, several oil quality parameters can be monitored by commercially available sensors, and active research and development programmes are being pursued by both academic and industrial researchers to develop robust, cost effective sensors for the remaining key parameters. Published (active) ASTM methods or practices do not yet cover the sensor technologies employed or under development, although work is in progress to address this deficit. This paper presents an overview of currently available oil condition sensors and looks at some recent developments, particularly in the following three areas: contamination by metallic wear debris, measurement of total water content, and determination of in-service oil viscosity. In each case, quite different technological solutions have been adopted. Where applicable, alignment and overlap with existing ASTM methods and practices will be reviewed and future directions indicated. Recent improvements in the sensitivity of inductive particle counters have enabled the detection of individual ferrous particles down to the sub-100 μm diameter regime and close to the 100 μm diameter mark for non-ferrous metals. Experiences of particle counters in wind turbine applications have shown the potential for enormous benefits in failure prevention. One standard practice covering the installation, operation, and requirements of such devices is published and a second is currently in draft mode. Online sensors utilising infrared transmission

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measurements recently have been developed by two independent companies. The systems are targeted primarily at marine diesel engine installations, although the method is not restricted solely to these applications. Maximum water content measurable depends on both the optical path length and the cleanliness of the oil. For marine applications a practical upper measurement limit of 1 % by volume has been adopted. In the system described here, a correction methodology, correlating to an accepted Deutsches Institut für Normung-Fourier transform infrared standard, has been adopted to cope with oils contaminated by soot. Soot loading increases the opacity of the oil, causing a concomitant reduction in the maximum water content measurable. The correction procedure increases the accuracy of the water content measurement and additionally provides a determination of the soot content. Commercially available viscosity sensors include both the oscillating piston type and high frequency oscillating crystal designs; however, a cost effective device employing a low amplitude, mid-frequency vibrating sensor element has been developed recently. Key features include accurate measurements over a very wide viscosity range and an operating range that covers combustion engine oil temperatures and pressures. Correlation with existing ASTM methods and practices is presently limited to calibration aspects only.

KEYWORDS: oil condition, sensors, wear debris, viscosity, water contamination

Introduction

Oil condition based maintenance (CBM) in which active monitoring of key oil parameters is used to monitor machine health and determine variable service intervals is an emerging maintenance philosophy. CBM provides the potential for reductions in operational costs, increases in performance, and improvements in machine safety. Offline laboratory oil analysis at regular defined sampling intervals has traditionally provided the data for CBM programs. However the case for online, real-time, monitoring of oil condition is easily made. In situ sensors address concerns of sampling issues, time lags from sampling to reporting, and analysis errors. It is not surprising; therefore, that considerable activity is in progress to develop simple, robust, cost effective sensors and systems for monitoring important oil condition parameters.

Oil analysis laboratories employ a suite of test methods and instruments for lubricant testing. Not all of these methods are directly transferable to online sensing without encompassing significant complexity and therefore expense. For example, in the mass automotive market, where cost is a significant driver, only two technologies have seen any uptake thus far:

(1) Capacitative dielectric measurements for oil quality trending.
(2) Solid state oscillating crystal devices for viscosity sensing.

Capacitance based sensors, e.g., the TEMIC QLT-sensor [1], have been fitted to high end passenger cars by several manufacturers since the mid-1990s. The dielectric properties of lubricating oils are determined by many factors, e.g., oxidation, fuel dilution, soot loading, additive depletion, and water or coolant ingress, and it is not possible to readily deconvolute individual influences from the observed changes. Nevertheless, in combination with a few other engine parameters, adequate prediction of the remaining useful life of in-service oils can be made with these sensors, resulting in flexible servicing intervals and optimised operational costs. Viscosity sensors, employing flexural or surface wave oscillators [2,3], are beginning to make inroads into automotive applications, although they have yet to be adopted in any significant numbers. The oscillating piston type of viscosity sensor has been used for several years in more critical or higher value engine applications, e.g., military vehicles [4], but has yet to be adopted en masse.

In other markets where capital investment costs are considerable, such as the marine and shipping worlds, and where the need for real-time monitoring of critical equipment is even more relevant, the uptake of online oil condition monitoring sensors is, surprisingly; still at a somewhat early stage.

Commercially available oil sensors for high ticket marine engines and machinery include capacitance, humidity (dissolved water), wear debris (including ferrous content and particle counting), and viscosity. Of these the so-called “oil quality” capacitance sensors seem to be the most established notwithstanding their inability to diagnose precise oil issues, as noted previously. Humidity sensors, discussed later, also have some limitations in their use and are rarely used in engine applications, where significant water levels are often encountered. However, total ferrous content wear debris sensors are routinely employed with great effect in the optimisation of cylinder oil feed rates in two stroke marine diesel engines [5].

Wear debris particle sizing and counting sensors are largely confined, at present, to aero-engine and gearbox applications [6,7], although considerable interest in monitoring marine maneuvering thruster gearboxes has been shown of late.

Process-style viscosity sensors are an essential part of heavy fuel oil control systems but, as yet, online viscosity sensors have found sparse use in marine markets.

Combining different types of sensors into one integrated package to provide a complete, application specific, picture of oil health is, of course, an obvious and desirable goal. Several companies are actively pursuing such projects; for example, a combination of wear debris and oil quality sensors is available for helicopter gearboxes [8] and an integrated multi-component microelectromechanical systems (MEMS) sensor is described in Duchowski and Mannebach [9].

Oil Condition Parameters

As outlined in the Introduction, only a few oil condition parameters can be monitored with commercially available online sensors. They include:

- acid number (AN),
- base number (BN),
- soot loading,
- metallic wear debris,
- water contamination, and
- viscosity.

AN, BN, Soot Loading

Changes in AN, previously referred to as total acid number, are largely due to oxidation processes associated with time and/or operating temperatures. It should also be noted that anti-oxidant and extreme pressure additives often impart significant acidic characteristics to fresh oil and depletion of these will tend to drive the AN in the opposite direction from that due to oxidation. AN changes can be trended with dielectric sensors—providing no other parameters are varying—after the establishment of a baseline AN response for the new (unused) oil.

BN, previously referred to as total base number, can also be correlated with dielectric measurements in a similar fashion to AN. BN monitoring is particularly important in diesel engine lubricating oil applications where a reduction in BN is observed as the alkaline reserve in the oil additive package is depleted by acidic combustion products. BN changes are often interlinked with changes in AN and it may not be straightforward to separate the two with a single dielectric measurement.

Soot loading—an increase in the insoluble content level—stems mainly from combustion products in internal combustion engines and is most pronounced in diesel engines. Increasing soot levels lead to increased oil viscosity, subsequent wear of bearing and running surfaces, blocking of oil ways and filters, and fouling of piston clearances. Soot loading can be sensed either by trending viscosity or determining dielectric changes. The same caveat applies in the latter case so that no other oil parameters should contribute to the observed variation.

ASTM methods for AN and BN determination are based on potentiometric titration (ASTM D664-09 [10], ASTM D2896-07 [11], and ASTM D4739-08e1 [12]), a chemical reaction, and can quite clearly separate the acidic and alkaline nature of the oil sample under test. Simple dielectric sensors which measure a single capacitance value do not have this capability, although frequency dependent dielectric measurements can provide information on individual quality parameters through a multivariate modelling approach [13].

ASTM methods and practices for determining soot loading include thermogravimetric analysis (ASTM D5967 [14]) and infrared (IR) attenuation (ASTM E2412 [15]). For the latter a standard practice for Fourier transform infrared (FTIR) trending is published, although it recommends reporting soot levels in absorbance units rather than concentration units. However, a published German standard (Deutsches Institut für Normung (DIN) 51452 [16]) recommends a method of calculating the soot content in mineral diesel oil from the measured absorbance levels. Finally, in preparation is an ASTM standard test method using an offline fixed-filter IR attenuated total reflection instrument for determining soot contents up to 12 %, following calibration by samples verified by D5967.

Metallic Wear Debris

Metallic wear debris particle counters employing inductive technologies [17] have been commercially available for some time. Using a combination of

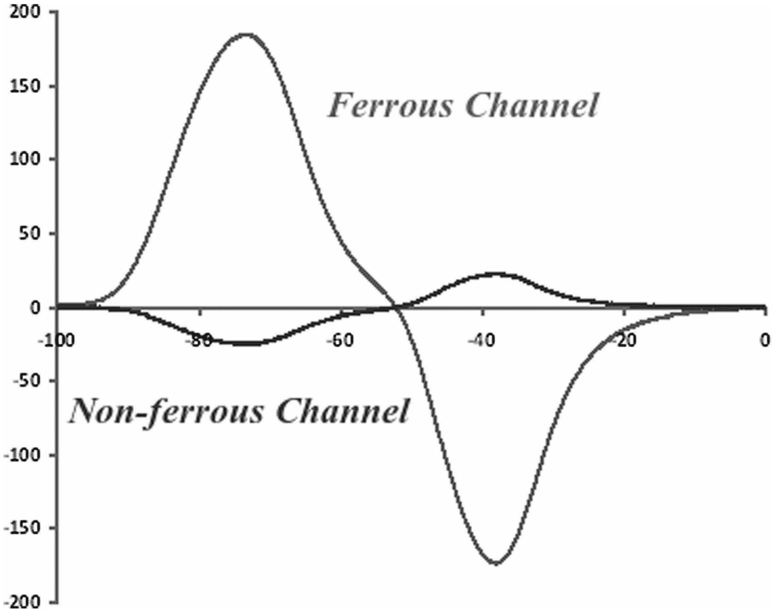

FIG. 1—*Inductive sensor waveforms.*

balanced field coils and sensing coils, these devices are able to determine particle sizes and differentiate between ferrous and non-ferrous metals. The sensitivity to ferrous particles is greater than non-ferrous because of the difference in mechanisms by which these two materials interact with the magnetic field. Classification into the two material types is straightforward via the reversal shown in the two signal phases (Fig. 1). In addition, sensitivity to both materials increases with reducing bore size. For practical oil line diameters around the 10 mm mark, the smallest particle sizes have previously been limited to 120 μm equivalent spherical diameter (ESD) for ferrous materials and around 300 μm ESD for non-ferrous materials. Significant improvements in signal conditioning circuitry have improved these figures to 40 and 135 μm ESD, respectively, dramatically increasing the chance of catching the early onset of wear before significant damage can occur. Wear modes in machinery take many forms, e.g., abrasion, spalling, pitting, cutting, and scuffing, and give rise to a large variety of shapes and sizes of debris. Nevertheless, the more sensitive the detector, the easier it should be to distinguish between normal and abnormal wear patterns.

Typical applications of wear debris sensors are in turbine, gearbox, or drive train installations, with an increasing interest in wind turbine gearbox monitoring which exhibits variable loading, due, in part, to the somewhat erratic nature of the primary power source. Some recent data from a wind energy field trial are shown in the section entitled "Wear Debris Sensor." One ASTM standard practice has been published for the use of wear debris sensors in aero-derived turbine engine applications [7] and a second is being developed for gearbox applications.

Water Contamination

Water contamination of lubricating oil has serious consequences and can lead to catastrophic failures, e.g., bearing lifetimes can be reduced to 25 % of their normal expected level by as little as 0.1 % (1000 ppm) of water [18]. It is not surprising; therefore, that water content measurements are often at the top of laboratory oil analysis reports. For online applications, several manufacturers offer relative humidity (RH) sensors adapted for oil use—often referred to as moisture sensors. These make use of capacitance measurements to monitor the amount of dissolved water in the oil. A porous polymeric material in contact with the oil functions as the dielectric medium between the measurement electrodes. Migration of water molecules into this polymer—driven by the chemical energy, or activity, of the oil/water solution—causes a change in the capacitance between the electrodes, which can be measured. Moisture sensors respond to increasing water levels but plateau at the saturation limit; at this point any additional water will exist in the free, undissolved state and will not contribute to the signal recorded. The saturation level depends on the oil type and state; in particular, age and operating temperature. Saturation can occur as low as a few hundred parts per million (ppm), which is easily exceeded in combustion engine operations. Consequently, moisture sensors are more appropriate for monitoring very low water levels; for example, in oils used as insulating media in electrical transformers. In addition, correlation between sensor output, usually reported as equivalent relative humidity (ERH), and water content in ppm, follows a logarithmic, Arrhenius-like behaviour. For fresh, new oils saturation versus temperature curves can be readily determined for a wide range of oils. In service; however, the situation becomes more complicated as oil aging causes shifts in saturation levels. This is of most concern in engines where aging is more rapid and pronounced, and less so; for example, in electrical or hydraulic applications. Note that ERH is defined as the RH of a sample of moist air which would give the same capacitance value as that measured in the sample oil at the same temperature.

Finally, ingress of water into oil will affect its overall dielectric properties and again—providing no other competing factors are present—can be monitored by simple capacitative dielectric sensors. Several companies offer these commercially for specific applications, e.g., water contamination in vegetable oil production facilities [19], where any response is unequivocal.

ASTM methods and practices include Karl Fisher titration (ASTM D6304 [20]) for determining water content and FTIR (ASTM E2412 [15]) measurements for trend analysis.

There remains; therefore, a clear need for an online water-in-oil sensor that can operate and report, with good accuracy, over a wide ppm range from zero to well beyond the saturation limit. Recent developments, see the section entitled “Total Water Content Sensor,” have focused on a sensor design using IR transmission measurements to measure total water-in-oil levels, i.e., levels irrespective of water state.

Viscosity

Viscosity is a key physical property of a lubricating oil; engine and machine builders specify viscosity grades after rigorous tests in order to minimise wear and optimise equipment lifetimes. Significant changes in in-service oil viscosity can indicate potential issues and should be investigated. Despite this, few online oil viscosity sensor technologies are currently available, although there is considerable academic interest in developing miniaturised devices [21]. Sensors of the oscillating piston type have been available for some time and are used extensively in the process industry. They are found in high value engine monitoring and critical vehicle applications [4]. However, the narrow clearance between piston and bore is a concern in diesel engine use where soot loading can be considerable, leading to deposition and subsequent jammed pistons.

Recent published research has focused on the use of micro-acoustic resonators operating at ultrasonic frequencies. In a typical device, a crystal oscillating in thickness shear mode is in contact with the oil along one face. Either the change in resonant frequency or the increase in driving power required to maintain the oscillating amplitude can be monitored, as both are a function of the viscous loading of the crystal. However, the penetration or sensing depth of such a device is inversely proportional to the square root of the oscillation frequency and deviation from expected behaviour can be seen in the case of oils containing long-chain viscosity modifiers [22]. Here, the macroscopic character of the oil clearly exceeds the microscopic sensing scale of the crystal. Nevertheless, such sensors are commercially available and include systems that monitor both oil viscosity and dielectric properties [23] simultaneously.

In order to circumvent some of the difficulties encountered in both approaches, recent developments have concentrated on sensor designs employing lower frequency vibrating elements of an open construction. These are described in the section entitled “Viscosity Sensor.”

There are many ASTM methods for oil viscosity measurements; probably the most widely used is ASTM D445 [24] for the determination of kinematic viscosity using a capilliary instrument. There are, as yet, no ASTM methods or practices for online viscosity sensors.

Sensor Developments

Wear Debris Sensor

As indicated earlier, improvements in signal conditioning circuitry have enabled significant increases in sensitivity for inductive particle sensors. We now have the capability to determine ferrous and non-ferrous wear debris down to diameters of 40 and 120 μm, respectively, in oil pipes of 10 mm bore with flow rates up to 21 l/min. This enables much earlier detection of early onset of wear, allowing timely remedial action before repair costs escalate.

A typical application for wear debris sensors is the monitoring of wind turbine gearbox oil (Fig. 2). The nature and variabilty of wind power is graphically demonstrated in Fig. 3, which shows a rapid rise in the total counts in all size ranges and both material classifications during a grid power trip—essentially a crash stop of the turbine.

Typical longer term data are shown in Figs. 4 and 5 for the same turbine over the 5 month period, May to October 2010. A gradual increase, with

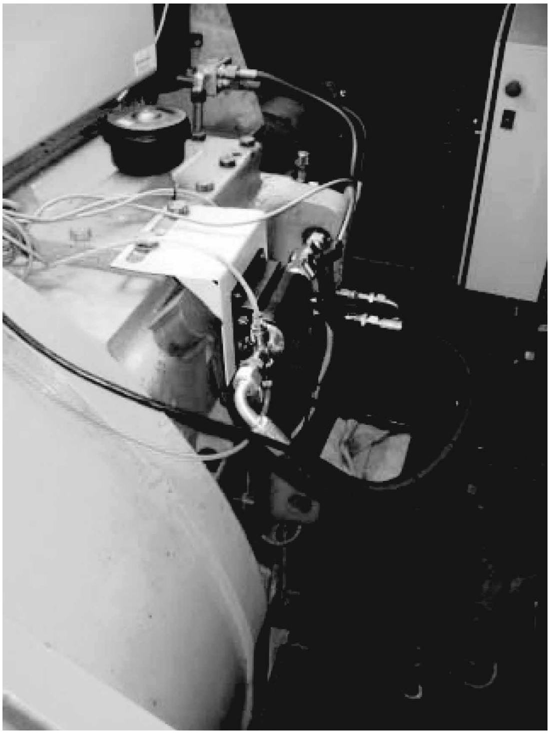

FIG. 2—*Wear debris sensor installation on a wind turbine gearbox.*

occasional step changes, in the counts accumulated is evident in the smaller particle size bins. This is consistent with sporadic high load events, similar to Fig. 3, causing the early onset wear revealed during a subsequent boroscopic investigation, Figs. 6 and 7.

Total Water Content Sensor

A fixed filter IR transmission sensor has been developed to measure water-in-oil levels up to the 1 % (10 000 ppm) mark. A schematic is shown in Fig. 8. IR radiation from a broadband source is focussed through an inline flow cell fitted with IR transparent windows and the transmitted radiation is recorded by two solid state detectors, each fitted with a narrow bandpass optical filter. The centre wavelength of one of these filters has been chosen to coincide with the absorption of the IR by water (actually the O–H bond vibration in water) and the other to a wavelength showing no water absorption (reference channel). In clean oils, the difference in the transmission seen by these two channels is related to the absorption of IR by the presence of water—the width of the water filter allows for any slight shift in the peak of absorption due to the different water states within the sample (free, hydrogen bonded, etc.). In used oils, additional attenuation may be seen in both channels due to scatter from particulates, most notably

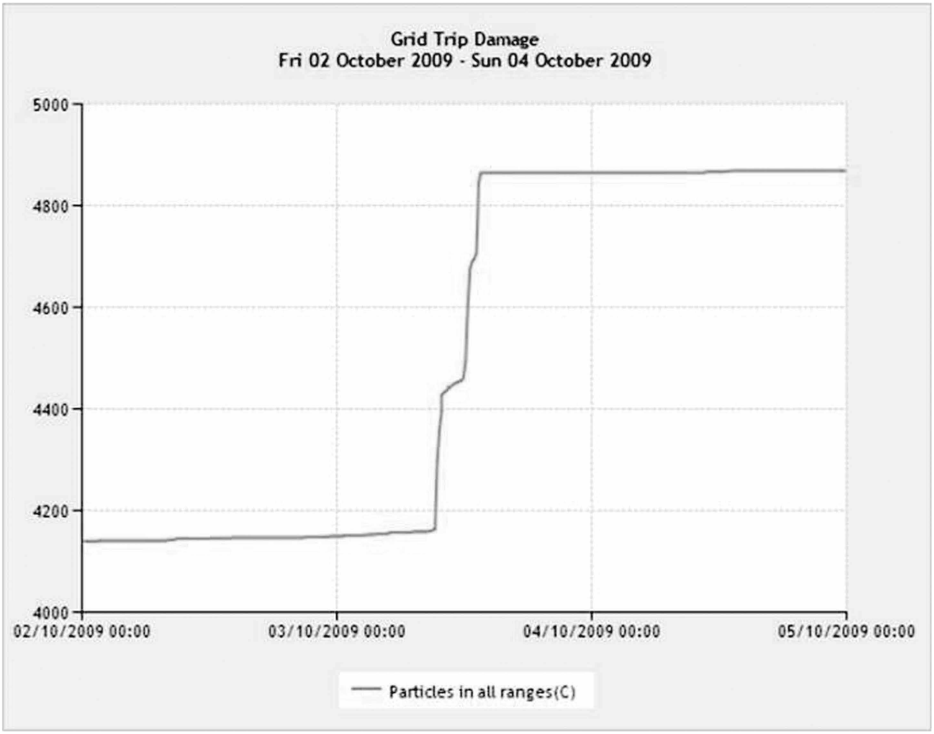

FIG. 3—*Rapid particle count rate increase during a grid power trip.*

soot in the case of diesel engines. During operation, an estimate of the amount of soot can be made by comparing the transmission, in the reference channel, of the dirty oil to a previously determined value in clean oil. A published protocol, DIN 51452 [16], is used to calculate the soot content from the observed reduction in transmitted radiation. IR attenuation due to scatter by soot has a wavelength dependence [16] and an appropriate factor due to the different filter centre wavelength of the water channel filter must be used to correct for soot attenuation.

Calibration of the instrument response is accomplished by measuring the IR transmission as a function of water spiked oil samples. Figure 9 shows absorbance data for a typical unused mineral diesel lubricating oil accurately dosed with water from 0 to around 14000 ppm. The absorbance is measured as the logarithm of the ratio of the IR transmission measured for the empty flow cell, within the bandpass defined by the optical filter, to that of the spiked sample. The slope of the calibration curve thus defines the response of the sensor to changes in water content and the offset (intercept) is due to any inherent absorbance in the oil or in its additives or contaminants (e.g., glycol and carboxylic acids contain O–H bonds) together with that due to any trace water level in the new, as supplied, oil. The latter value can be independently measured by, e.g., Karl Fisher titration, to determine the overall (total) water content and

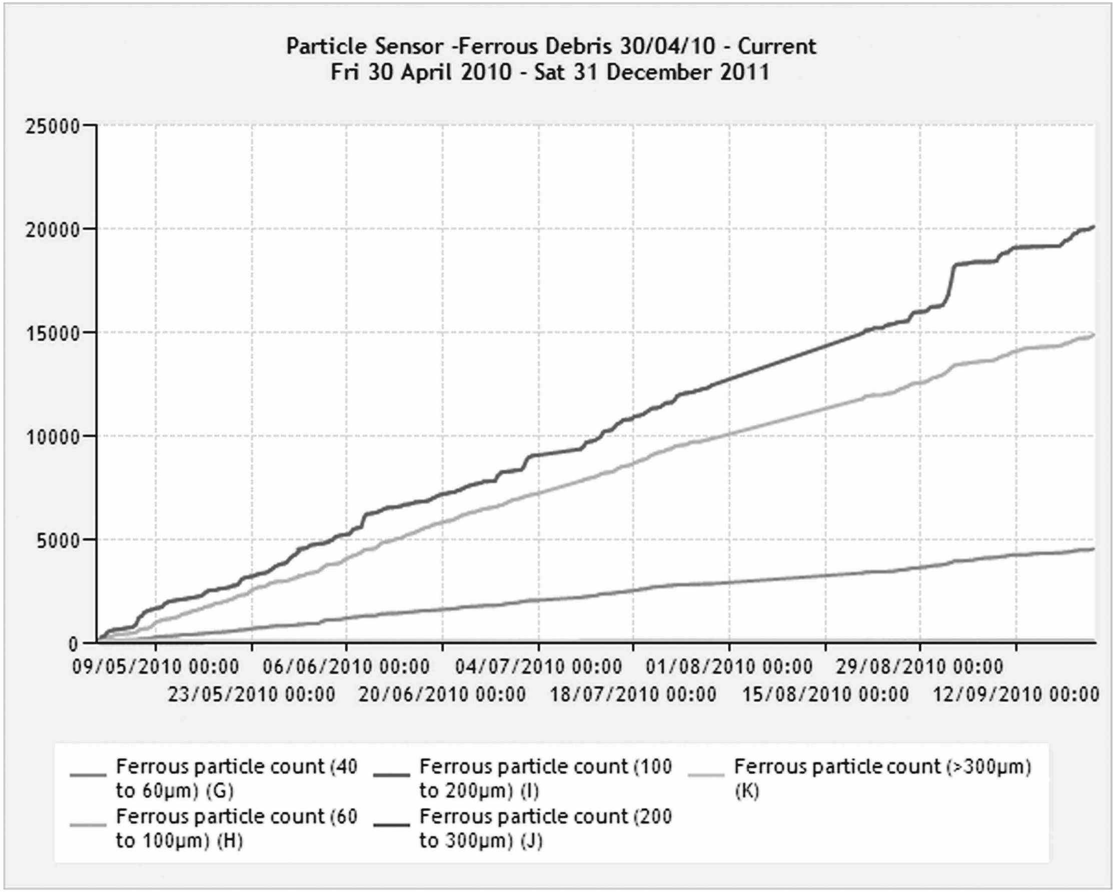

FIG. 4—*Accumulated ferrous debris counts in five size ranges.*

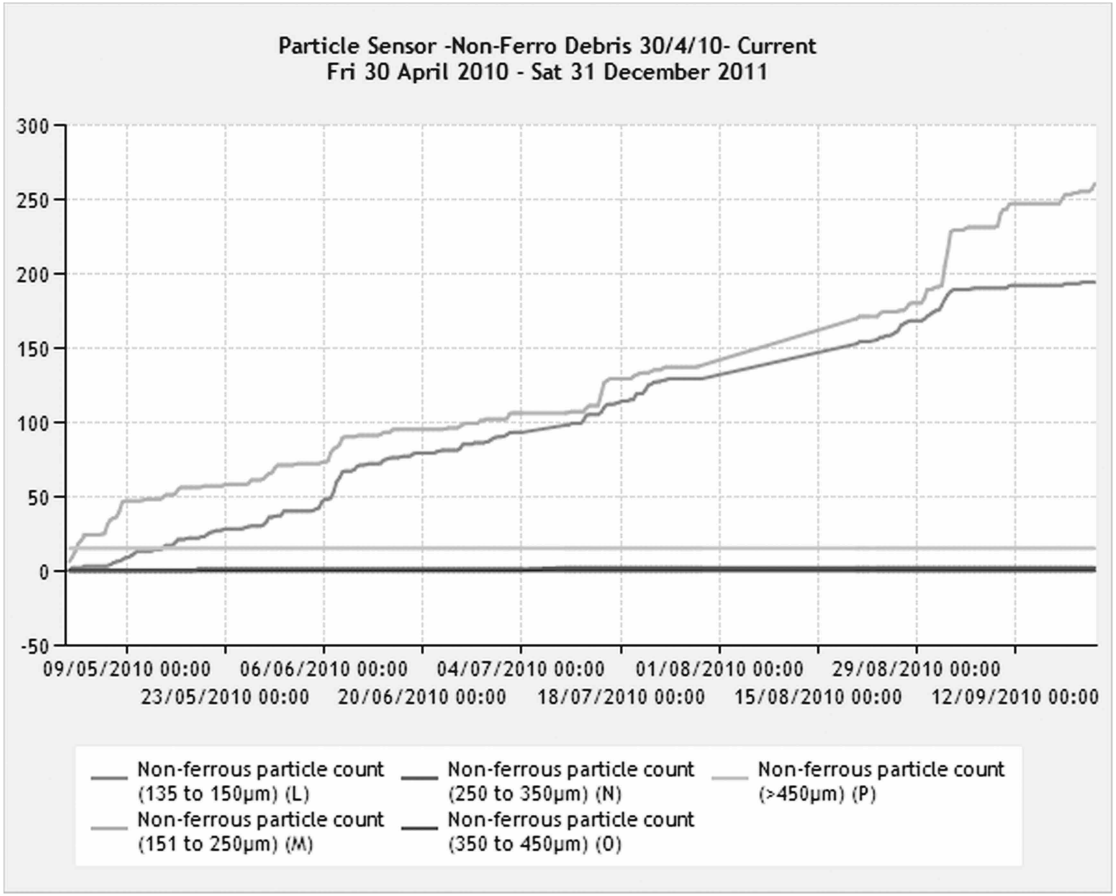

FIG. 5—*Accumulated non-ferrous debris counts, also in five size ranges.*

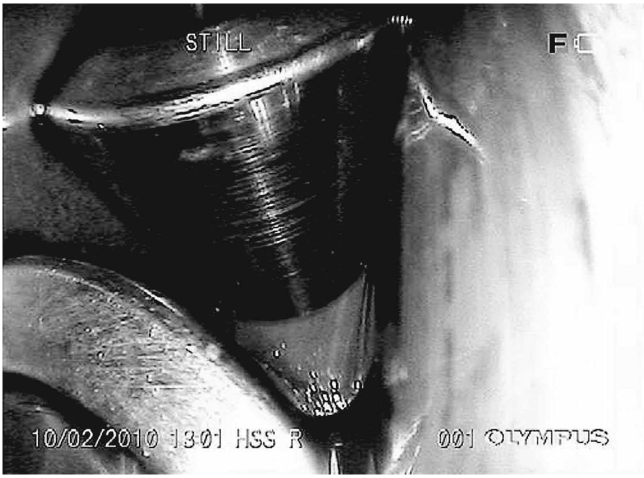

FIG. 6—*Visible scoring on a bearing roller.*

therefore it is possible to completely define the calibration curve for any particular oil type.

Figure 10 shows the correlation of DIN 51452 soot measurements for oil samples prepared with varying soot and water contents (1000–10000 ppm). For each soot content data set there is some slight scatter within the group of spiked water contents, but nevertheless the correlation shown is still very good. A further set of data, Fig. 11, shows the water absorbance for samples of the same type of oil as that in Fig. 9, all of which contain a similar amount of soot (0.5 %)

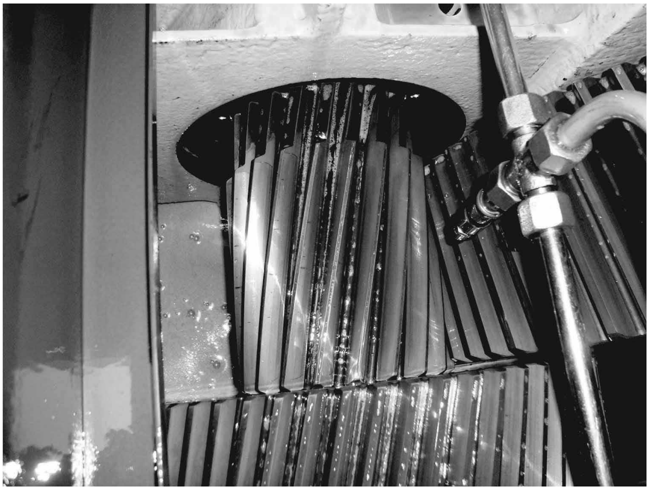

FIG. 7—*Visible scuffing on shaft gears.*

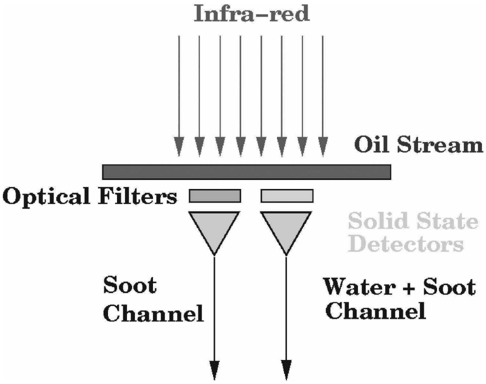

FIG. 8—*Schematic of IR total water sensor.*

and after correcting the response for the attenuation due to the soot. The important point to make is that the slope of this calibration line (0.487) is in excellent agreement with that of the clean oil (0.492).

Four prototypes of the water sensor are currently undergoing sea trials in independent marine main engine installations. Figure 12 shows a picture of one of the installations monitoring the system oil of a nine cylinder Sulzer RTA-96C slow speed crosshead diesel. Figure 13 shows some typical trend data from this sensor. Very little soot content is recorded due to the clean nature of this engine

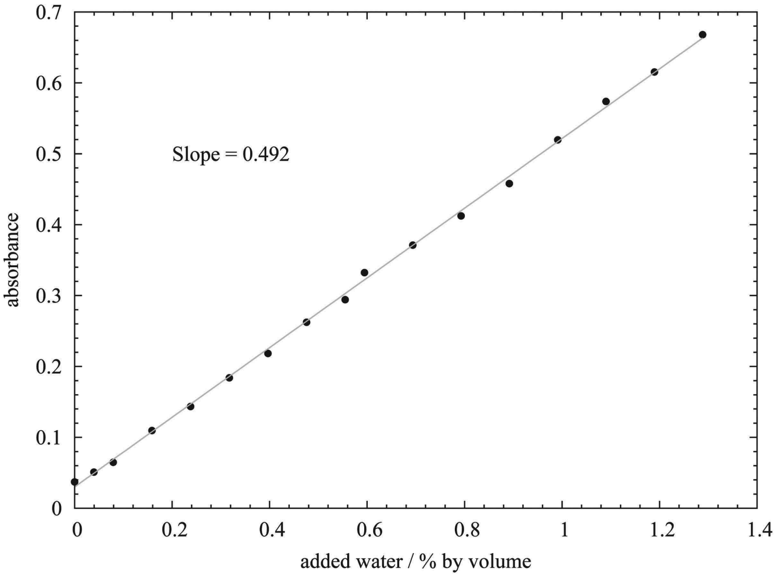

FIG. 9—*Calibration line for clean diesel mineral oil.*

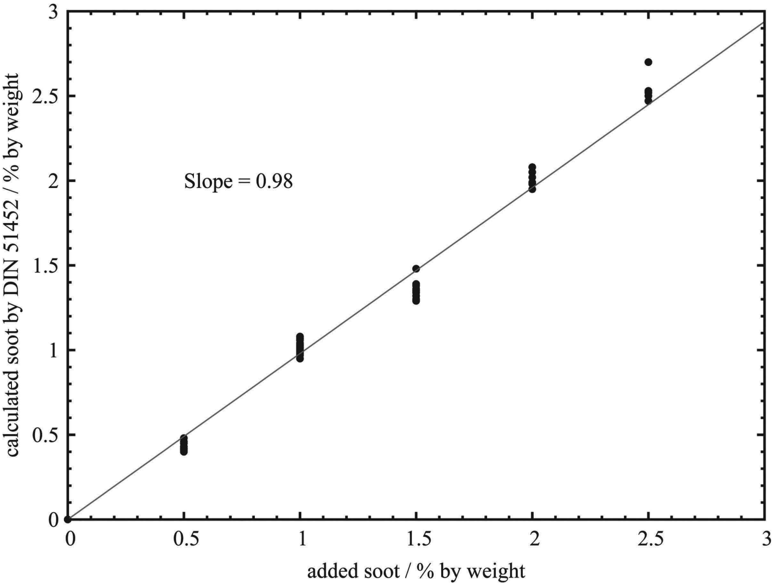

FIG. 10—*Correlation of measured soot levels with actual levels.*

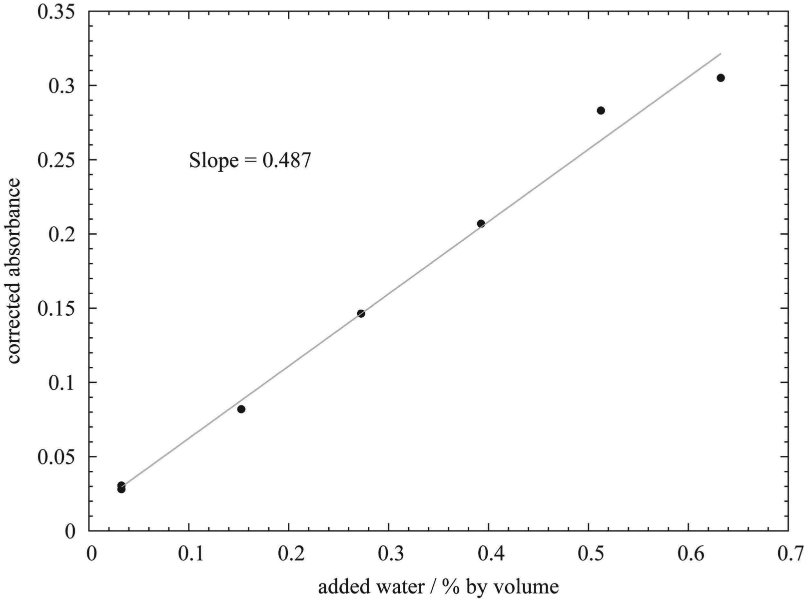

FIG. 11—*Soot corrected water calibration line.*

FIG. 12—*Slow speed diesel engine installation.*

design, with the system oil well separated from the combustion chamber. Figure 14, on the other hand, shows data from an eight cylinder Sulzer ZA40S medium speed diesel installation where the soot content is noticeably higher. The data from these two installations has been verified by offline periodic sampling and testing in an independent analytical laboratory using ASTM certified test methods.

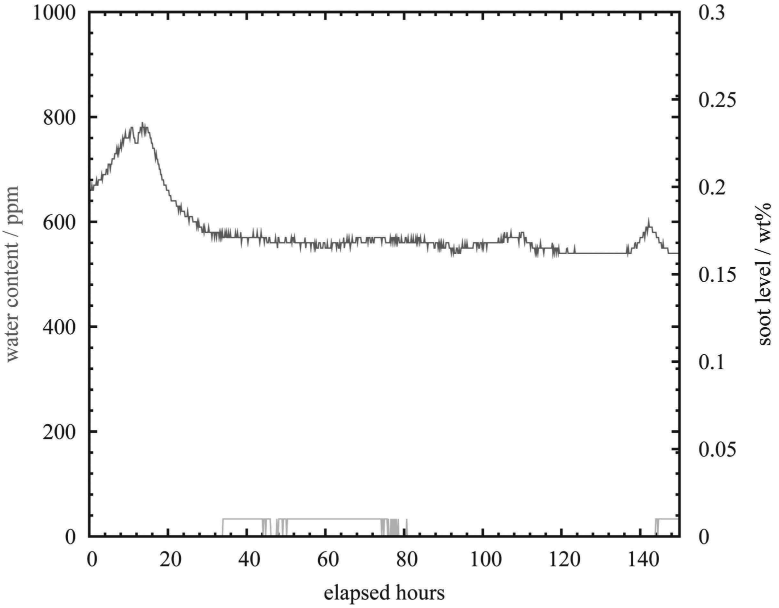

FIG. 13—*Slow speed diesel engine data.*

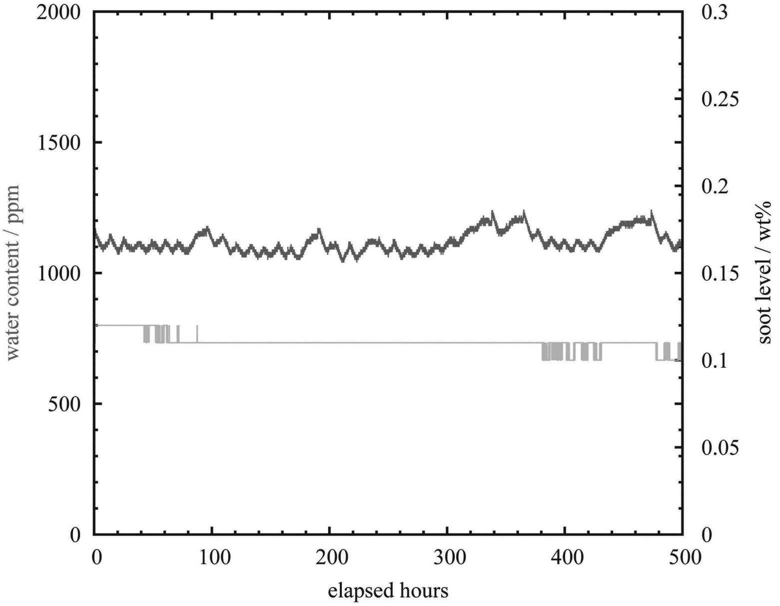

FIG. 14—*Medium speed diesel engine data.*

Viscosity Sensor

A vibrating element viscosity sensor based on an earlier device [25] has been developed. In this sensor, a thin metal strip is excited into a resonant longitudinal vibrational mode in a pulsewise fashion. The fundamental longitudinal vibrational mode of a strip has a node at the mid-point along the length of the strip that can be used as a position for a suitable "bulkhead" to separate the oil side from the air side. A concept sketch of the sensor is shown in Fig. 15, where the excitation and sensing make use of the magnetostrictive properties of a

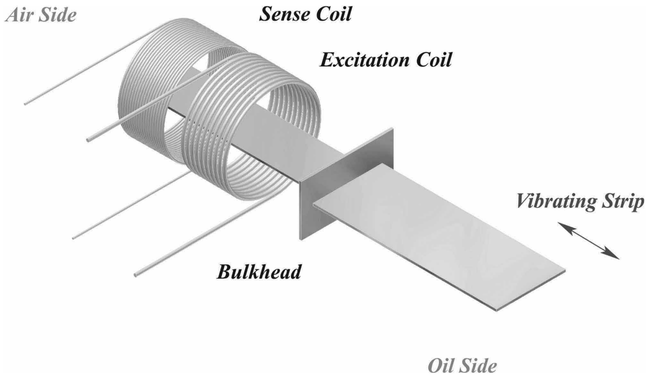

FIG. 15—*Viscosity sensor concept.*

ferromagnetic strip. Small dimensional changes, of the order of a few ppm, occur in ferromagnetic materials when they are subjected to magnetic fields (Joule effect). By applying a short magnetic pulse, a magnetostrictive strip can be given a mechanical impulse which will induce vibration. The converse effect, known as the Villari effect, in which a change in the mechanical stress of a magnetostrictive material gives rise to a change in the magnetic susceptibility of the material can be used to follow the resulting decay pattern.

Vibrating viscosity sensors, of both the tuning fork and torsional oscillation design, are prevalent in the process industry with the vast majority employing piezoelectric excitation and sensing. Careful design in these viscometers is required to ensure that the input mechanical energy is not dissipated into additional unwanted vibrational modes or heavily attenuated in the support structure, and also that the mode damped by the process fluid can likewise be detected with sufficient sensitivity. In the sensor presented here, contact-free axial magnetic pulsing leads to efficient excitation of the fundamental longitudinal mode, resulting in the required shear motion of the transverse metal strip surfaces with respect to the oil.

Figure 16 shows typical decay profiles for a strip vibrating in air (red-- decaying to zero by 14 milliseconds) and in an oil of moderate viscosity (green). The increase in the rate of decay of the individual oscillations, shown expanded in the insert, is quite marked between air and the oil. This additional decay is due to the viscous damping effect of the oil on the strip and follows a simple exponential form characterised by a single decay constant

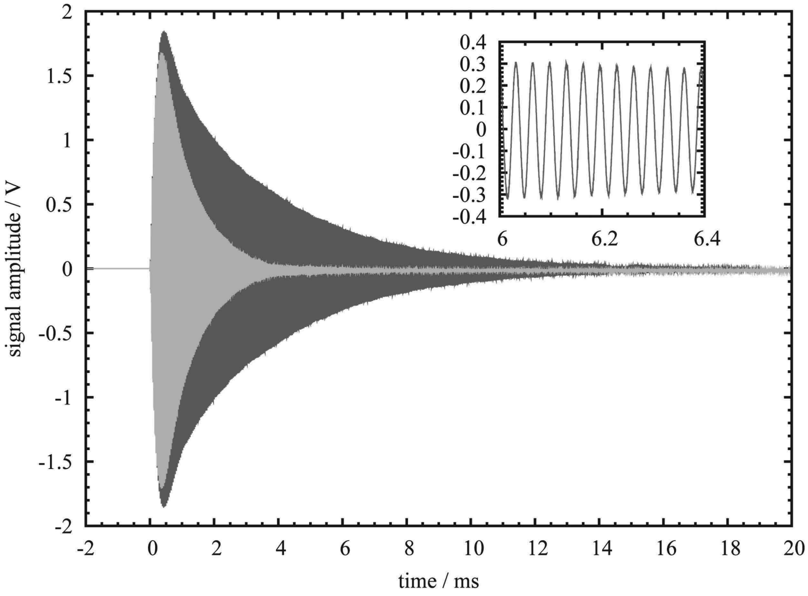

FIG. 16—*Typical decay profiles.*

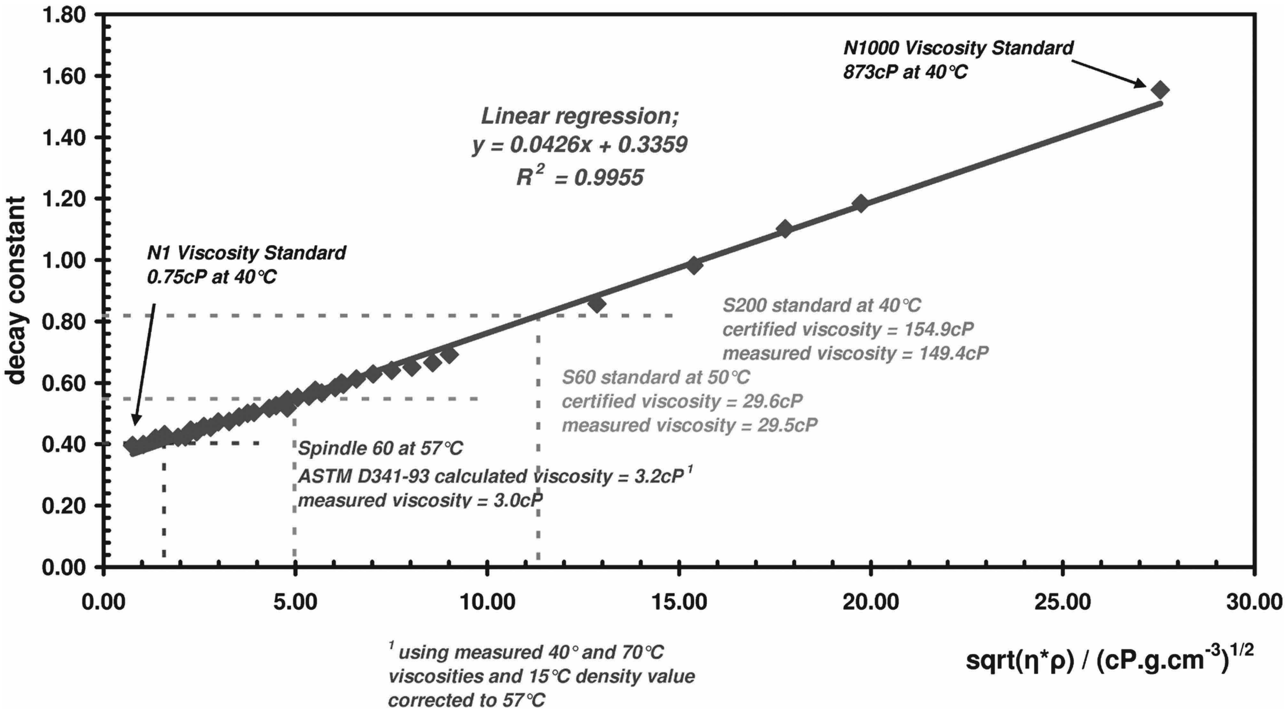

FIG. 17—*Calibration with known viscosity standards.*

$$S(t) = ae^{-b.t} \tag{1}$$

where:

$S(t)$ = observed signal after time t,

a = initial amplitude at $t = 0$, and

b = decay constant.

The rate of decay can be shown to be a linear function of the square root of the viscosity-density product [26] of the oil. Figure 17 shows measured decay rates as a function of the square root of the product of certified viscosities and densities, in units of centipoise (cP) and grams per cubic centimeter (g/cm^3), for various commercially available viscosity standards at different temperatures between 25°C and 70°C. For each measurement the oil was held to a temperature stability of ±0.1°C and between 50 and 100 waveforms were captured and analysed. The time required to record and process a single waveform is of the order of 20 ms (Fig. 16), which allows signal averaging techniques to increase the statistics even over periods as short as 1 s for live measurements.

Also shown in Fig. 17 are comparisons of the viscosities of three test samples, determined from the calibration line, with the known viscosities of the samples.

The range of viscosities used to prepare Fig. 17 was from 0.7 to 900 cP and a statistical analysis of this calibration data leads to reporting accuracy limits of less than 1 % for a 99 % confidence level. A sensor based on this concept is currently at the prototyping stage (Fig. 18) and will shortly be undergoing field trials.

FIG. 18—*Prototype viscosity sensor.*

Concluding Remarks

This paper illustrates the increasing pace of development of online sensors and provides a view of their future potential in the key role of managing and maintaining operational efficiency of engines and associated machinery. The uptake in the use of online sensors for oil condition monitoring in machine applications has been relatively slow to date. In part this may have been due to a mismatch between those oil properties currently obtainable for a reasonable investment cost and the requirements of condition based monitoring programs; in particular, the key parameters of viscosity and total water content. The sensor developments described in this paper address these shortcomings and provide cost effective solutions to monitoring such important oil properties.

At present, there is one published ASTM practice for an online oil condition sensor, namely wear debris sensors in aero-derived turbine engine [7] and work is in progress to develop a second practice for similar sensors in gearbox applications. Existing offline methods can, and should, continue to be used to verify or calibrate online sensor responses and suitable methods have been indicated in the text where appropriate.

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Thomas M. Canty[1]

In-Line Monitoring of Particulate, Color, and Water Content in Lubricating Oils to Facilitate Predictive Maintenance, Reduce Wear, and Provide Real Time Alarming

ABSTRACT: Imaging techniques for characterizing particle shapes and sizes in lubricating oils can be a valuable tool toward improving the life span and reliability of mechanical equipment. ASTM currently specifies several tests to monitor the condition of lube oil; however, most of these are off-line, lab based tests. Vision based systems are well designed to perform the examination of the lube oil, either in line or off line, as they are the only instrument systems that can determine particle shape, which is a critical parameter in determining the source of wear in a mechanical system. The visual capability allows the user to input his or her knowledge of the process to work in making meaningful analyses. Imaging filters can be applied to the analysis to sort through noise and focus on the important particle information that must be monitored. This paper describes a vision based system used for particle sizing that provides a true two-dimensional size and shape analysis. The paper also discusses the fundamentals of a vision system that allow for accurate and repeatable analysis of a wide range of processes.

KEYWORDS: lube, particle, shape, visual, fused, glass

Nomenclature

CCD = charge coupled device; camera chip which converts light to a digital image signal

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Lube oil = oil circulated through a mechanical system to lubricate moving parts and reduce friction and wear
Vision system = visual based configuration of illumination, image capture, and associated product handling hardware

Introduction

ASTM describes several lab based methods for assessing wear and contamination of a lube oil sample. Most require human inspection in some form to determine particle shapes which are critical in determining the source of wear in the mechanical system. For instance, particles that exhibit a crescent shape are classified as being caused by fatigue of the parent metal [1]. Water droplets appear as a dark ring with a mostly transparent center. Vision technology can detect these features, classify the particles, and give real time feedback of the condition of the lube oil. This paper is based, in part, on several unpublished lab and field studies of particle detection in lube oil conducted by J.M. Canty Inc.

Background

Vision System Fundamentals

The building blocks of a vision system include a constant and bright illumination source, a quality lens and camera to gather true images, and a method of controlling the flow of the process through the measurement zone in a way that provides repeatable and accurate analysis results. These requirements hold true for both wet and dry process systems.

Illumination, transmitted through the particle stream, allows the camera to see the particles. As with the human eye, objects or particles can appear to be of varying size due to changing light conditions. In very poor light conditions an object's edge may be difficult to see and thus its size may be difficult to determine. The same is true for a vision based instrument in that it detects the size of a particle by determining what its boundaries are by separating the object from its background. In the simple case of a backlit system, bright lighting conditions will reveal definite boundaries of particles, while dim lighting will reveal less definite boundaries. Under these two lighting conditions it is likely the same particle will be sized differently and that likelihood is the reason constant, bright illumination is a critical component of a good vision system.

The camera/lens combination dictates the resolution with which the particles will be viewed. Proper magnification and chip array combinations are required to accurately resolve the true size of the particle captured. The lens system must also be capable of conducting a true image to the camera chip. Distortions due to lens effects can insert error into the analysis. Current technology standards include Ethernet camera systems which can be viewed and controlled over network communication lines. This enables the best quality video transmission as well as remote access throughout a network.

Lastly, the presentation of the particles to be measured to the camera is as important as the first two requirements. A typical layout is included in Fig. 1.

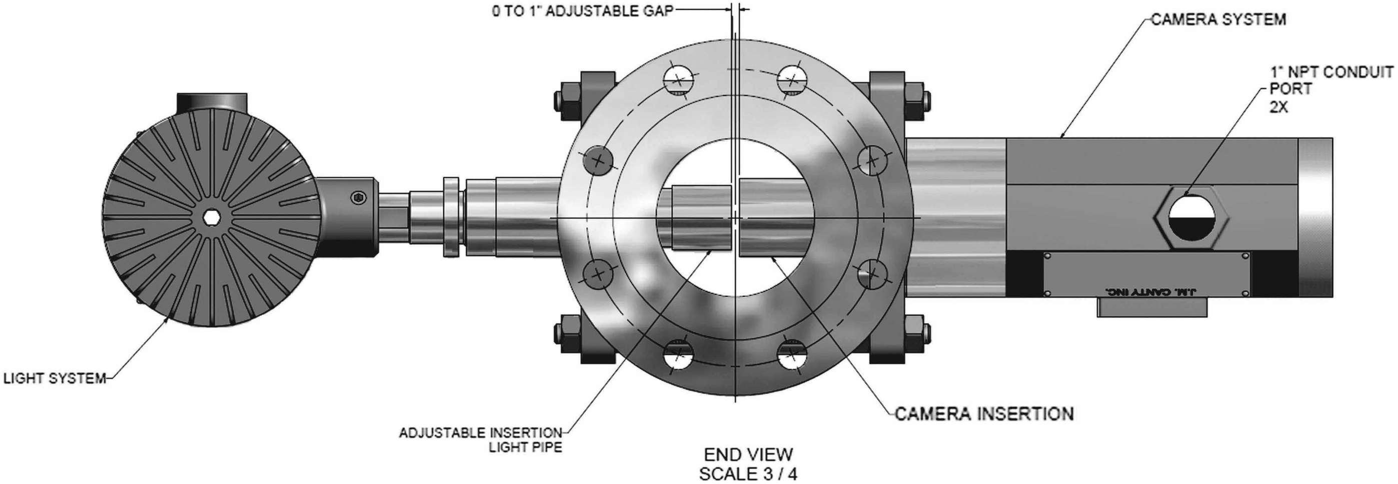


FIG. 1—*Typical in-line particle size instrument.*

Illumination is provided from one side of the flow chamber while the camera is on the opposite side. Particles are backlit and captured on video where they are further analyzed by the software. The critical part of the presentation of the process is that the vision system can handle the process conditions (pressure, temperature. etc.), but also have the capability to control the flow gap and maintain a clean view into the process. Both of the latter capabilities may seem obvious; however, many in situ instruments are not capable of achieving these conditions, which leads to degradation of the measurement quality.

At this point an explanation of fused glass to metal technology is important in understanding how the process view can remain clean and unobstructed. A fused glass to metal window is constructed by melting glass into a metal ring. The glass fuses to the metal and as the window is cooled the glass accepts greater compression until ambient temperatures are again reached. This manufacturing process places the glass under several thousand pounds of compression, which changes the apparent characteristics of the glass from fragile to rugged and from poor under pressure load to very good under pressure load. As a window is placed under pressure, the outer surfaces do not go into compression, rather they merely relieve compression and this process avoids the normal reaction glass would have, i.e., to break in a brittle way under low pressure.

The manufacturing process also provides another benefit to process viewing. In a normal, unfused window the glass is sealed to the process by a gasket. This type assembly creates contamination sites. The fused glass construction presents a perfectly joined metal–glass interface that acts as one material, and so the process only encounters a smooth, polished surface (see Fig. 2).

Hardware is not the only important piece to the puzzle. Software provides the real insight into the process when it is integrated with a properly designed hardware system. Typical backscatter devices (lasers) classify particles by the reflected light pattern; they do not directly measure particle features and only provide a size characterization of the particle [2].

These systems do not provide any insight into the particle shape, which can be of great interest for many manufacturers. Without knowing the particle shape it becomes impossible to classify particles, which is a requirement for lube oil analysis. For reference, particle shape classifications can be found in ASTM D7596 and are as follows:

(1) Non Metallic Particles—partially transparent.
(2) Cutting Wear Particles—elongated, crescent, or curly shape.
(3) Fatigue Wear Particles—mostly near unity aspect ratio with jagged edges.
(4) Sliding Wear Particles—elongated with straight edges.
(5) Fibers—extreme elongation and/or partial transparency.
(6) Air Bubbles—generally spherical with thick, dark edges and transparent centers.
(7) Water Bubbles—generally spherical with thin, dark edges and transparent centers.
(8) Soot—particles generally less than 1 μm that darken the base oil fluid.

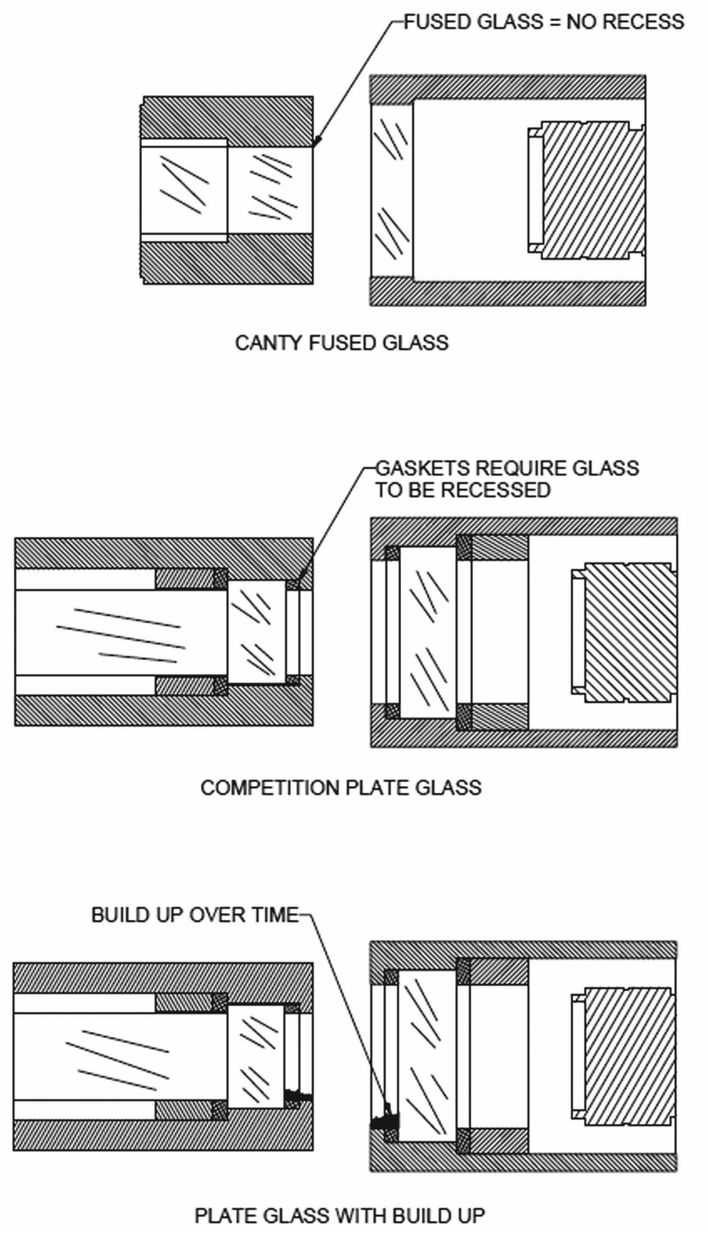


FIG. 2—*Fused glass versus gasketed glass.*

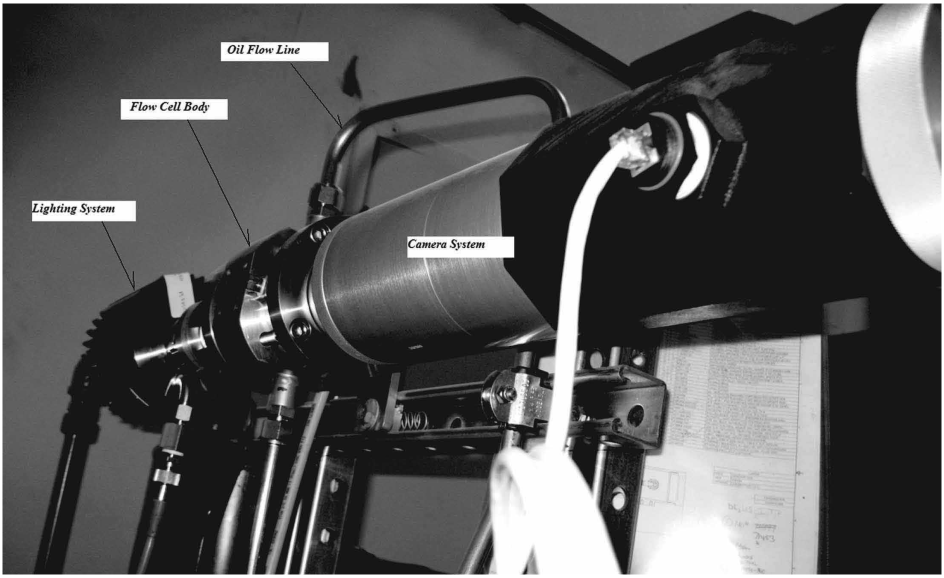


FIG. 3—*Vision system mounted in 1/2 in. oil line of compressor.*

Experimental

The capture and analyses of particles was taken from in-line vision systems installed in the lubrication flows of mechanical equipment. The image in Fig. 3 shows a vision system plumbed into the oil line of a compressor system as a typical example. For convenience, videos were recorded daily during oil circulation cycles and analyzed off line in order to maintain a record of the oil condition versus time.

Particle Analysis

The definition of a particle is determined by the user interacting with the software. This is done by viewing the particles as they move through the measurement zone and, with the aid of the software, determining how the digital particle is formed. As it is with the human eye, objects are distinguished from their backgrounds due to the way light reflects off different surfaces as well as variations in object color. The vision system can be set so it accurately digitizes a particle in the field of view (see Figs. 4 and 5). The scale of the images to follow is approximately 650 μm with a picture resolution of 0.4 μm per pixel. Lens systems are telecentric, meaning no change in apparent object size with distance from the lens, and are generally factory set and need no adjustment in the field. With no lens adjustment necessary the calibration remains fixed with no drift. Shutter speed and light intensity are by adjustment through the software, and also remain constant after initial setting. Once all operating parameters are input, the system is ready to run in a constant measuring state indefinitely. For

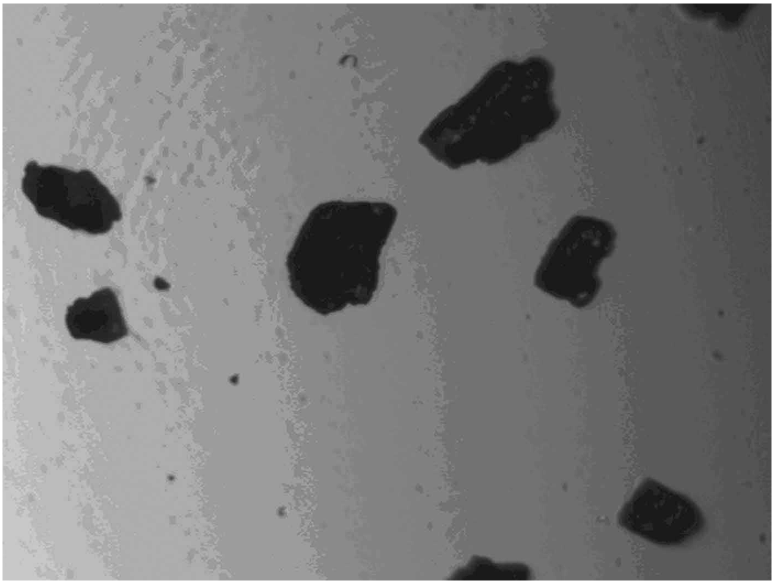


FIG. 4—*Live image of particles.*

FIG. 5—*Digital image of particles.*

more reproducible results (system to system) the instrument can be set up off line using particle spheres of known size. Each instrument should agree on size of known standards, although differences in setups by different operators do not make for large errors. Experience shows that different, trained operators will get size results within 1%–2% of each other.

The particles that are digitized show a very close profile to their undigitized images. This is done by manipulating the target value of light intensity change from the background to the particle that determines where the software finds the edge. This value is often referred to as the threshold. Some particles do not show up in Fig. 4. They do not meet the focus requirements set to be counted as a particle. It is normal to analyze well focused particles only. Due to randomness, in a properly designed analyzer all particles will flow through the measurement zone with equal regularity and so they all have the same chance of being captured in focus. The exercise of setting the threshold is a visual confirmation that the instrument is properly determining how to measure the particles and this is a powerful tool when trying to gather accurate information.

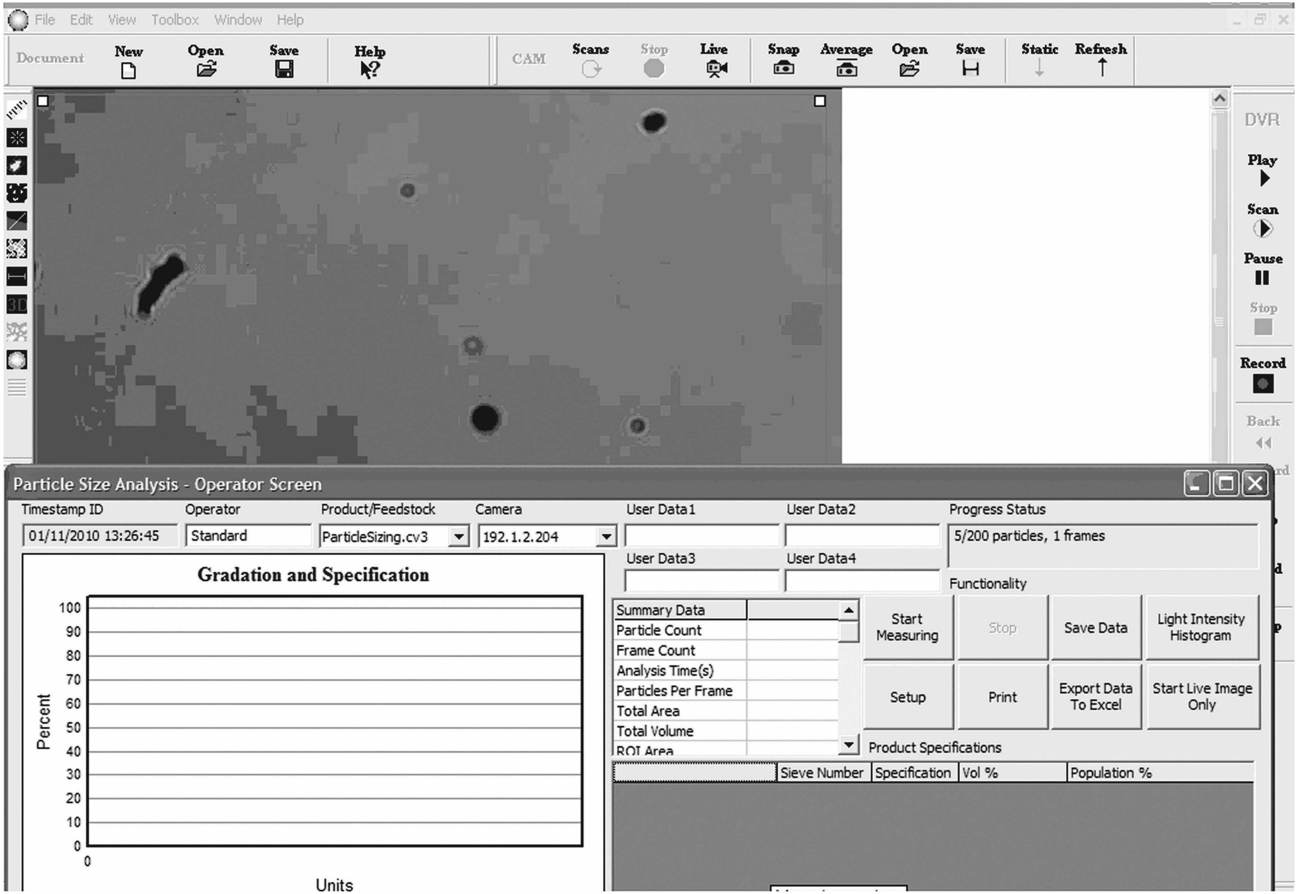
Results

To demonstrate some of these features, actual images and particle analyses will be presented and explained. The image in Fig. 6 shows a live image of a software interface as would be seen by the user. Figure 7 shows the image as digitized and analyzed by the software.

Figure 7 shows the scanned image of Fig. 6 and how it captures particles and what the particle size measurements are. Measurements are shown in millimeters, so transforming to the micrometer scale the elongated particle is 861×302 μm in size. Figure 8 shows the same image with additional information not shown in Fig. 7. These additional headings reveal color, intensity, and circularity information to name a few. As an example of how particles can be classified, look at the entries listed for particle circularity. Circularity is defined here as an area to perimeter ratio. Notice the value for the elongated particle on the left is much lower than either of the circular particles on the right side of the screen. Values near one (1) indicate a particle is close to circular in the 2D representation. An average circularity value of one (1) would indicate that the average particle is likely close to spherical, whereas a value well away from one tends more toward a rod shape.

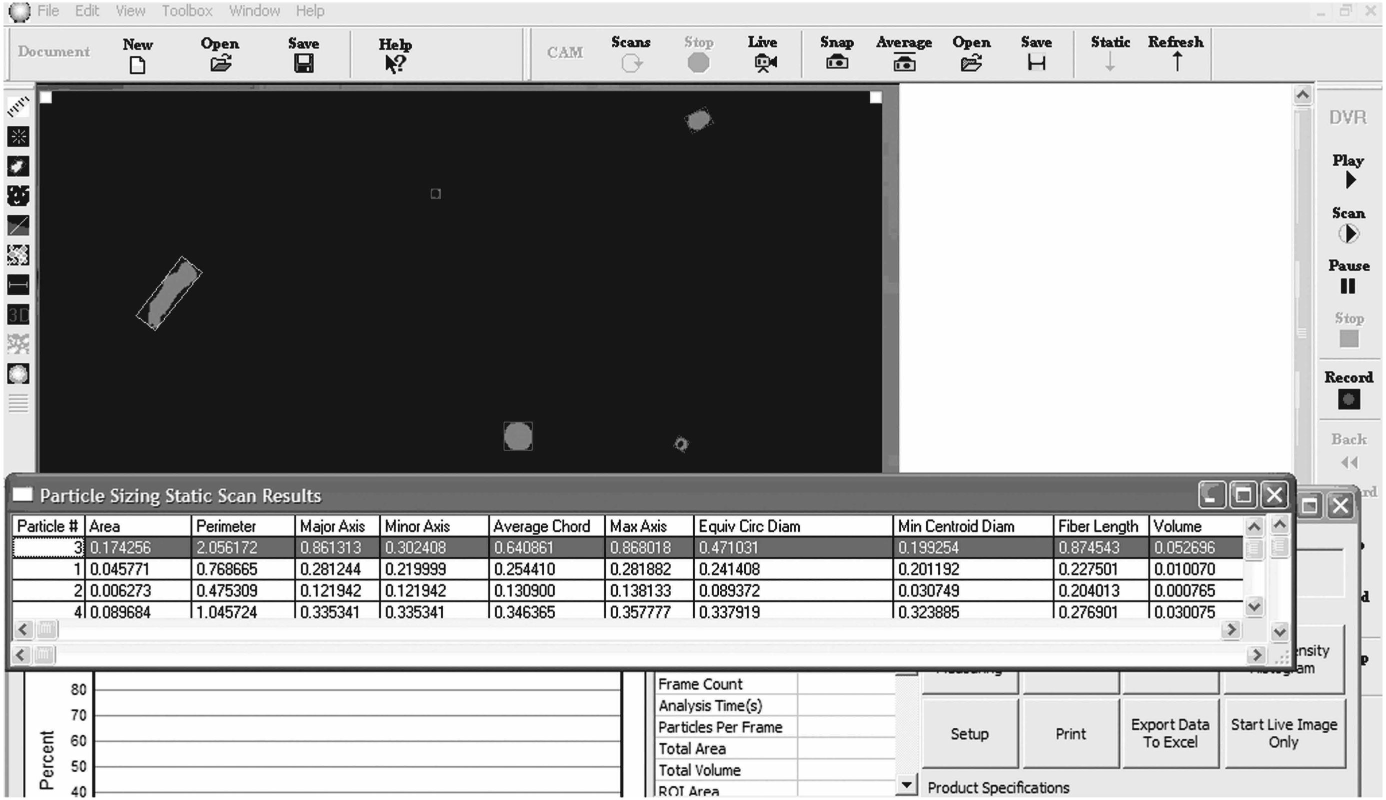
Discussion

ASTM D7596, as detailed previously, contains several categories of particle shape. These are general classifications and do not specifically identify particle material or source, but rather are based on experience of machinery wear and testing and provide a consistent logic with which to categorize detected particles. The following images better show how previously discussed particles of various classifications found in lube oil appear to the eye (Figs. 9 and 10).



Timestamp ID: 01/11/2010 13:26:45
Operator: Standard
Product/Feedstock: ParticleSizing.cv3
Camera: 192.1.2.204
Progress Status: 5/200 particles, 1 frames

FIG. 6—*Live image capture of particles in process.*

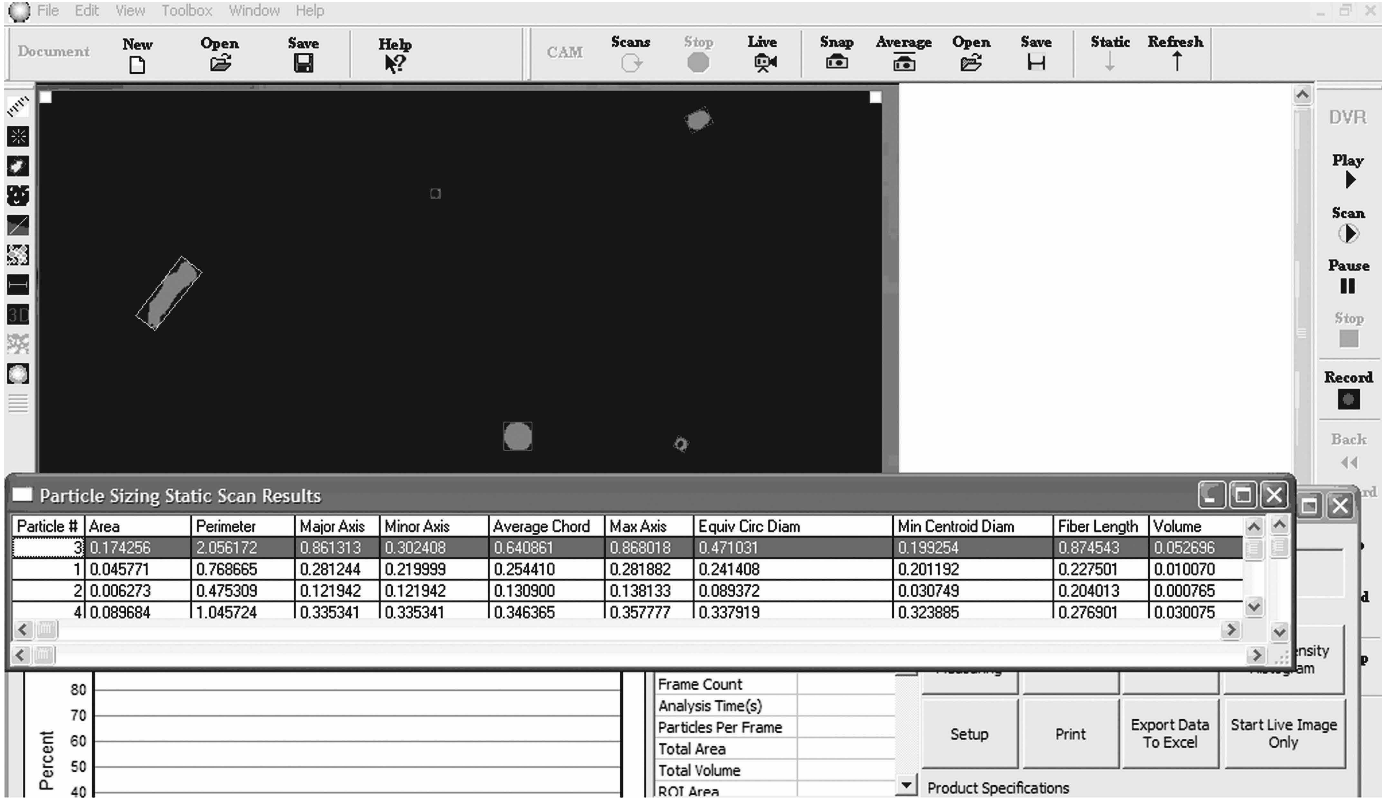


Particle Sizing Static Scan Results

Particle #	Area	Perimeter	Major Axis	Minor Axis	Average Chord	Max Axis	Equiv Circ Diam	Min Centroid Diam	Fiber Length	Volume
3	0.174256	2.056172	0.861313	0.302408	0.640861	0.868018	0.471031	0.199254	0.874543	0.052696
1	0.045771	0.768665	0.281244	0.219999	0.254410	0.281882	0.241408	0.201192	0.227501	0.010070
2	0.006273	0.475309	0.121942	0.121942	0.130900	0.138133	0.089372	0.030749	0.204013	0.000765
4	0.089684	1.045724	0.335341	0.335341	0.346365	0.357777	0.337919	0.323885	0.276901	0.030075

Frame Count
Analysis Time(s)
Particles Per Frame
Total Area
Total Volume
ROI Area

FIG. 7—*Scanned image of particles in process.*



Particle #	Area	Perimeter	Major Axis	Minor Axis	Average Chord	Max Axis	Equiv Circ Diam	Min Centroid Diam	Fiber Length	Volume
3	0.174256	2.056172	0.861313	0.302408	0.640861	0.868018	0.471031	0.199254	0.874543	0.052696
1	0.045771	0.768665	0.281244	0.219999	0.254410	0.281882	0.241408	0.201192	0.227501	0.010070
2	0.006273	0.475309	0.121942	0.121942	0.130900	0.138133	0.089372	0.030749	0.204013	0.000765
4	0.089684	1.045724	0.335341	0.335341	0.346365	0.357777	0.337919	0.323885	0.276901	0.030075

FIG. 8—*Additional data from Fig. 6.*

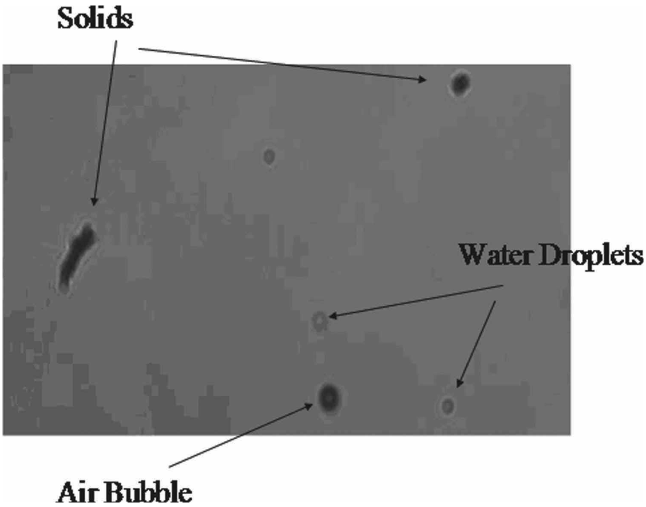


FIG. 9—*Live image of various particles.*

The ASTM specification is perhaps too general in that it does not distinguish separation points between particle types. For instance, what is the transition point between a sliding wear and cutting wear particle shape? How much curvature defines the cutting wear particle? Better definition by numerical dimensional ratios would improve repeatability of use of the ASTM standard. Additionally, the instrument does not control the orientation of the particle as it flows through the measurement zone, which can lead to uncertainty of the measurement. This may be statistically insignificant depending upon the relative particle count in a given oil.

In-line vision analysis provides several benefits over other methods that require sampling and human inspection of each sample. With a fixed set of parameters the vision system will yield repeatable, objective results. The in-line capability also makes visual records of oil status easily and automatically obtainable. Systems can notify by alarm when particle presence is trending

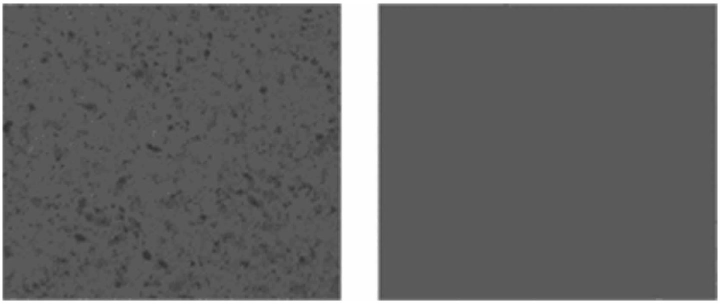


FIG. 10—*Soot in oil versus clean oil.*

worse, and the software can record detections as they occur to form a record for maintenance engineers to study and monitor on a daily basis. Ethernet capability allows data to be transmitted across networks and the Internet, which is beneficial in multiple ways. It is the case that some equipment, such as windmills, is not easily accessed for maintenance procedures as the engine is quite a distance off the ground. Still other equipment may be located in remote areas. In both cases having real time data can make maintenance decisions more efficient on a timing and cost basis. In addition, the capability to see the process from virtually anywhere can bring multiple skills together, i.e., engineering and maintenance, in order to optimize trouble shooting and decision making in the effort to extend the life of the equipment.

Conclusions

The adaptability of vision systems to processes along with their robust analysis capabilities make them ideal for applications such as lube oil monitoring. Operators "train" the system to pull out proper data from the live image, which enables real time analysis, and leads to more efficient maintenance of mechanical systems which can result in longer equipment life spans.

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David Turner[1]

Analysis of In-Service Lubricating Grease

ABSTRACT: The analysis of in-service lubricating fluids is a useful tool in determining the state of both the fluid and the equipment being lubricated. Many laboratories provide analysis services for in-service lubricating fluids. The results of the analyses performed are often tracked over time to allow for the observation of trends in the test results. The analysis of in-service lubricating grease is a more challenging task; since the amount of grease in many applications is quite small, the grease may be partially or totally replaced each time new grease is added, and access to the lubricated contact zone may be difficult. Many commercial in-service lubricant analysis laboratories lack the specialized test equipment required for grease analysis, so that type of analysis is performed by a limited number of laboratories, primarily those of the grease manufacturers. Various techniques are employed to determine both the macro and micro condition of in-service lubricating grease. The strengths and weaknesses of those techniques are discussed herein.

KEYWORDS: lubricating grease, in-service

Introduction

Lubricating grease is a mixture of a lubricating fluid, additives, and a thickener. The lubricating fluid may consist of mineral oil, vegetable oil, or synthetic fluids. Each type of fluid imparts unique properties to the grease, and in some situations, a blend of different types of base fluids is used to obtain the desired properties. Additives are used to enhance the performance of the grease. Common additives used in greases include oxidation and rust inhibitors, and antiwear and extreme pressure additives. Additives that are sometimes added to greases that are not typically found in fluid lubricants are solid lubricants (i.e., graphite, molybdenum disulfide, PTFE, etc.) and other solid fillers, and

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tackiness additives (high molecular weight polymers). The thickener is the component that makes a grease different from a fluid lubricant. The thickener gives the grease consistency (the property of resistance to flow not associated with the base fluid viscosity). Materials such as simple and complex metallic soaps of carboxylic acids (organic salts), polyurea (nonmetallic organic thickener), organophilic clays (bentonite or hectorite), fumed silica, and various other materials are used as grease thickeners. The majority of commercially-sold greases use lithium soap thickeners [1].

Two fundamental properties of lubricating greases are consistency and dropping point. The consistency of lubricating grease is measured with the ASTM D217 Cone Penetration test, based on the 60-stroke worked penetration value [2]. It measures the stiffness of the grease, which relates to its handling characteristics. Penetration is measured in units of mm/10. The ASTM D2265 Dropping Point [3] measures the temperature at which the thickener softens sufficiently to release the first drop of fluid, and is sometimes used to determine the maximum operating temperature at which the grease can be used.

The consistency of lubricating grease, as determined in ASTM D217 is classified according to the NLGI consistency grade classification system [4]. The consistency grades range from 000 (fluid) to 6 (block). Table 1 provides the complete listing of NLGI consistency grades.

National Lubricating Grease Institute (NLGI) 2 is the most commonly used grade, however, NLGI grades 3, 1, and 0 are also frequently encountered.

The dropping point of a grease may give an indication of the melting point of the thickener used in the grease. Table 2 shows the dropping point ranges of common grease thickeners.

Soap thickeners (both simple and complex) and polyurea thickeners have defined dropping points influenced by the softening response of the thickener and its melting point. The dropping point of greases based on inorganic thickeners (clay, silica) is undefined, since the thickener materials do not melt at temperatures within the operating temperature range of the equipment where grease is typically used.

TABLE 1—*NLGI grease consistency grades.*

NLGI Consistency Grade	Worked Penetration Range	Description
000	445–475	Fluid
00	400–430	Semi-Fluid
0	355–385	Very Soft
1	310–340	Soft
2	265–295	Medium
3	220–250	Medium-Hard
4	175–205	Hard
5	130–160	Very Hard
6	85–115	Block

TABLE 2—*Typical dropping point ranges.*

	°F	°C
Calcium	265–285	129–140
Lithium	380–400	193–204
Li Complex	450 +	232 +
Other Complexes	500 +	260 +
Polyurea	518 +	270 +
Clay	Undefined	Undefined

The common applications in which lubricating greases are used include bearings (both plain and rolling element types), gears (enclosed and open), sliding contacts, and grease-lubricated couplings. Grease is used in automobiles (including Formula 1 race cars), commercial aircraft, military aircraft, petrochemical plants, and other industries, such as cement; basically, anything with moving parts.

The amount of lubricating grease used in most applications is small, in some cases, just milligrams. Obtaining a representative sample, or enough grease to test, from some applications can be challenging.

Most grease tests require two ounces (50 g) or more of grease to perform them. Many grease tests require a significantly larger sample. Testing of in-service grease often requires modification of test methods or specialized test methods that require far smaller samples.

Commonly used grease tests include:

- Appearance.
- Water by crackle test.
- Fourier transform infrared (FTIR) analysis.
- Elemental analysis.
- Ferrography/ferrous density.
- Penetration ([1/2]- or [1/4]-scale).
- Grease rheometry.
- Pressure differential scanning calorimetry (PDSC).
- Remaining antioxidant by linear sweep voltammetry (RULER).
- SRV (EP).

Appearance is one of the most important of all tests performed on in-service lubricating grease, and sometimes is overlooked. The appearance of lubricating grease consists of the color, texture, and odor of the grease. The visual appearance of the in-service grease can be photographically documented.

For products that are familiar, the color of the in-service grease should be compared with that of the unused product. The color can indicate whether the proper product was used, and changes in color can indicate contamination or oxidation. The color of an unfamiliar grease, such as a competitive product, may help to determine the product identity. In-service grease samples with mixtures of colors often indicate the mixing of different products, or different levels of service severity within the equipment being lubricated.

The texture of grease can range from smooth and buttery to rough, stringy, mealy, fibrous, etc. The texture of in-service grease can indicate the presence of contaminants such as dirt/dust, wear debris, water, and oxidized grease. Changes in texture may indicate that the grease thickener or tackiness additive has broken down in service. Spreading the grease in a thin layer on a white ceramic tile can be very useful in determining the condition of in-service grease.

Although not a visual property, the odor of an in-service grease sample can tell a great deal about how the product was used. Some contaminants (especially solvents and other organic compounds) have characteristic odors that are easy to identify. Oxidation causes a distinctive odor, as does the degradation of certain additives. The odor of the in-service grease may help to indicate what further tests should be performed.

Photographic documentation of in-service grease or failed parts in the as-received condition can be very useful in reporting the results to the customer who submitted the materials for analysis. Photomicrographs can help to show the presence of minute contaminants in in-service grease.

Water is a common contaminant in in-service grease. Most fluid lubricants contain some dissolved water, however, because of the manufacturing process, most unused greases are water-free. Some greases purposefully contain some water. The crackle test is an easy qualitative test to run, and requires a very small portion of the sample. The grease is placed in a metal or glass dish and touched with a hot soldering iron or heated on a hot plate. Popping or crackling of the sample indicates the presence of water. Using the crackle test, water that is not visually indicated can be detected. A trained ear can differentiate between low and higher levels of water contamination. If an analytical value for water content of the in-service grease is necessary, ASTM D95 Water by Distillation may be used [5].

Fourier transform infrared (FTIR) analysis should be performed on every in-service grease sample that is submitted to the testing laboratory. The FTIR is most powerful when a reference spectrum of the unused product is available for comparison. FTIR provides useful information for the determination of base fluid characteristics, thickener type and concentration, additive types and concentrations and additive degradation products, and oxidation of the base fluid or thickener. FTIR also allows for the detection of water and other contaminants and mixtures of different grease types. The fluid and solid portions of the grease can be separated for more detailed analysis. If only one test can be performed on an in-service grease sample, FTIR should be that test. FTIR provides the most data with the smallest sample. It provides the in-service grease analyst the biggest ‘bang for the buck.’

Elemental analysis can be run by various test methods, including atomic absorption, inductively coupled plasma, spark emission spectroscopy, x-ray spectroscopy, and scanning electron microscopy with energy dispersive x-ray spectroscopy. Each test method has its own strengths and weaknesses, including particle size detection limitations, interferences between elements, etc. The proper test method for each sample must be selected based upon the condition of the sample (determined from the appearance or known service conditions). Elemental analysis is used to detect environmental contaminants, wear metals, additives, and thickeners. It can assist in confirming the identity of a grease, or help to determine the service conditions or mixture with another product. Some elements can come from multiple sources. Aluminum, for example, can be a thickener component, an environmental contaminant, or a wear metal.

Ferrography and ferrous density can be performed on an in-service grease sample to identify wear metal particle types and sizes. The test separates the particles from the lubricant and allows for their microscopic examination. Various wear modes and certain contaminant types can be determined using this technique. Ferrous density is a measure of the concentration of ferromagnetic metals in the sample, and can indicate the degree of wear that has occurred on metallic components. Analytical ferrography can be a powerful tool for determining wear particle types.

Small-scale penetration tests have been developed to allow for consistency measurement of smaller size samples. Both [1/2[-scale and [1/4]-scale tests are covered in ASTM D1403 [6]. Although less precise than the full-scale test (ASTM D217), the small-scale penetration tests can be used when the sample size is limited.

The rheometry of grease is a test that is just now being developed. It has the potential to be a powerful technique in the evaluation of in-service grease samples. The sample size required is very small (~1 mL), even smaller than the sample size required for the [1/4]-scale penetration test. Similar to the penetration test methods, the sample is evaluated under controlled conditions. ASTM D02.G0 is currently developing a test method based on a controlled strain sweep, possibly with more complex test methods to follow.

Pressure differential scanning calorimetry (PDSC), run according to ASTM D5483 [7], is used to measure the oxidation stability of lubricating grease. The oxidation induction time is measured under the conditions of elevated temperature and 500 psig (34.47 bar) pure oxygen. The value determined for an in-service grease sample can be compared to the value for the unused product to give an indication of the remaining oxidation life of the grease in service.

The remaining phenolic and aminic antioxidants in grease can be determined with ASTM D7527, along with the remaining antioxidant by linear sweep voltammetry, one of the newest ASTM grease test methods [8]. Known as the RULER test, the results are compared against those for the unused product, with the final result being the remaining levels of the antioxidant additives in the in-service grease. That result can then be used to estimate the remaining useful life of the in-service grease.

If the extreme pressure properties of an in-service or unused grease sample of limited size must be determined, the ASTM D5706 extreme pressure by SRV test can be useful [9]. It requires a very small sample size, and the test has a fast turnaround time. For in-service grease, the result should be compared to the value for an unused sample to determine whether the load carrying characteristics of the product have changed over time due to additive degradation or depletion.

In summary, in-service grease samples tend to be very small in size. It is often difficult to obtain a truly representative sample of grease that has been in the contact zone of a bearing or gear. Most tests for unused grease require relatively large samples, and are therefore not suited to the testing of in-service greases. Testing of in-service grease requires the use of specialized tests, or special adaptations of standard tests. In-service grease can be sufficiently well tested to provide meaningful information. In-service grease testing can be a powerful tool in troubleshooting problems in grease-lubricated systems.

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Lubricating Oils Evaluation of Dispersancy Capacity of Lubricating Oils and the Impact of Biofuels on Lubricant Dispersancy

ABSTRACT: One of the primary functions of a lubricant is to preserve the cleanliness of a combustion engine's mechanical parts. The cleanliness of the mechanical parts is facilitated by the introduction of "detergency" and "dispersancy" additives to the engine oil. This last property, dispersancy, is the property that allows the oil to suspend and carry away "pollutants" of diverse sources, such as soot from combustion, metallic particles from wear, corrosion of mechanical parts, and insoluble products resulting from the aging of the oil, etc. Recently, with the arrival of new fuels (e.g., biodiesel, ethanol blends, etc.) we notice that the existing or traditional lubricants present an important variability in terms of durability and resistance to pollutants, such as the fuel or the biofuel. Our studies show that the dispersancy capacity of certain types of lubricants is significantly degraded by the presence of specific pollutants, and in particular fatty acid methyl esters of the biofuels, which have a significant impact. It is therefore important to quantify the degradation of the oil in-service (engines or transmission oils) and to monitor the evolution of the oil's dispersancy properties during use to be able to determine steps and intervals for maintenance. Additionally, for the development of new lubricants it is necessary to define a criterion of acceptance of the oil by its dispersancy capacity. This new method will define and quantify this criterion. The writers will present the results of these studies with particular

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emphasis on the impact of biofuels on the dispersancy capacity of both gasoline and diesel engine oils. They will describe a method of analysis using an automated instrument providing precise, objective measurement of this important parameter. This method will therefore be able to estimate the dispersancy capacity of lubricants, as well as the implication of various pollutants on this parameter.

KEYWORDS: lubricant, dispersancy, blotter test, spot test, OCM, oil conditioning monitoring, thermal ageing

Problems—Impact of the Thermal Behavior and the Pollutants on the Lubricants

Dispersancy capacity of certain types of lubricants is significantly degraded by the presence of specific pollutants, soot, and in particular fatty acid methyl esters (FAME).

The impact of soots or insoluble matters on the in-service oil dispersancy power is known and is of course evaluated by the method, but we wanted to center our work mainly on the presence of organic ethanol fuels or diesel (EMHV), which poses two new major issues via the durability of oil:

- An acceleration of the oxidation of the lubricant under operation, which generates acidity, an increase in viscosity, and a possible formation of insoluble products.
- A reduction of viscosity by dilution, which involves a loss of lubricant flow

If this presence of fuel and thus of biofuel is not monitored, the main risk is, on one hand, the loss of pressure of lubricant and thus the break-in of the engine, and on the other hand, the formation of deposits and the coking of these deposits in the hot parts of the engines, such as pistons and segments, as well as the turbo compressors.

Problems with Diesel Engine Lubricant and Impact of Biofuel

The various systems of pollution reduction imposed by the European directives generate a presence of diesel fuel in the engine lubricant in service. For example, the use of diesel particulate filter (called DPF) has, as a consequence, the presence of fuel in the lubricant. This dilution by the fuel is due to post-injections generated to voluntarily facilitate the cleaning of the filter by increase in the temperatures.

Problems with Gasoline Engine Lubricant and Impact of Biofuel

For the gasoline engines, the new injection systems can also generate important dilutions by the fuel.

Review of the Existing Methods, Which Can Put Forward the Phenomenon

The effects on the durability of engine lubricant, due to several parameters, but more and more with the presence of biofuel, affect appreciably certain performances of lubricant. *One of these performances, essential for PSA (Peugeot Citroen Automobiles), is the impact of all these pollutants on the dispersancy capacity of lubricant and thus the cleanliness of the engine*. To date, no rigorous analytical method makes possible the measurement of the dispersancy capacity of the lubricant. The blotter spot method could give an answer to this need but the only method practiced to date is based on a visual evaluation. This visual interpretation; therefore subjective, is not rigorous and consequently limits the information that could be provided by the method.

Conclusions

Within sight of this report, the team undertook a work in three stages:

- Analyze existing technique for the measurement of dispersancy.
- Improvement of the blotter spot method.
- Application of the improved method to the problems met.

Methods to Analyze Lubricant Dispersancy

In lubricating oil technique, in-service oil conditioning monitoring and also during the monitoring of the state of the engines via the analysis of lubricant, it is very often a question of the dispersancy capacity. Nevertheless we do not think that a precise definition of dispersancy capacity was given, allowing for quantification. To date, the qualification of the detergency dispersancy of a lubricant is generally not carried out by measurement or calculation. We indirectly measure this performance based on the presence and the rating of carbon, soot, ashes in lubricant, or mud in the engine. However such a definition, even theoretically imperfect, associated very closely with the instantaneous demonstration with the detergency-dispersancy characteristic, would be extremely useful, in particular for the following:

- Lubricant qualification with engine tests, without disassembling and quotation of their parts.
- Oil conditioning monitoring.
- Engine monitoring during operation.

Dispersancy/Detergency Measurement by Engine Test (DV4TD - CEC-L-93-04)

Description: Peugeot DV4TD Medium Temperature Dispersivity test (CEC-L-93-04) is a part of ACEA 2004: A1/B1-04 A3/B3-04 A3/B4-04 A5/B5-04 C1-04 C2-04 C3-04 specifications.

The objective of this procedure is to evaluate the effect of combustion soot on engine oil viscosity increase and piston cleanliness. This procedure simulates high-speed highway service in a diesel-powered passenger car. The procedure fixture is an engine dynamometer procedure stand with a Peugeot DV4 TD/L4 four cylinder, in-line, common rail diesel engine installed. Pistons and rings are future rated for lacquer deposits and ring sticking. Kinematic viscosity at 100°C, soot content, and iron content in the used oil are evaluated at 24-h intervals during the procedure. The final oil drain is used in conjunction with the intermediate samples to interpolate the absolute viscosity increase at 6% soot.

Advantages and Disadvantages of This Method—This approach has the merit of exactly reproducing the behavior of lubricant under definite conditions of test. However, the evaluation methods on the engine are very long and expensive. In addition, the precision of this method cannot be at the level of a laboratory method.

Dispersancy Measurement with the Blotter Test Method

Various versions of this very old method exist in the industry. Many studies showed the value of this method as being rich in practical information on in-service lubricant, but it remains mainly manual and home-made. The interpretation of the spot to date remains subjective and not formalized by a universally recognized method (refer to Fig. 1).

To conduct the test, a small quantity of the homogenized sample is heated to 240°C (464°F) for 5 min. The purpose of this short period of intense heating is to stress any oil that is close to thermal or oxidative failure, so that the blotter spot shows a positive response. Any oil that is still in good shape will not be affected by such a short heating period, which will be reflected in the dispersion pattern of the blotter spot. Once the sample cools, an approximately 25 μl aliquot is dropped onto chromatography paper (or filter paper) and allowed to spread, or wick, for 1 h in an oven set at 80°C (176°F). The filter paper is then placed under a light source in order to locate the various rings. The calculation

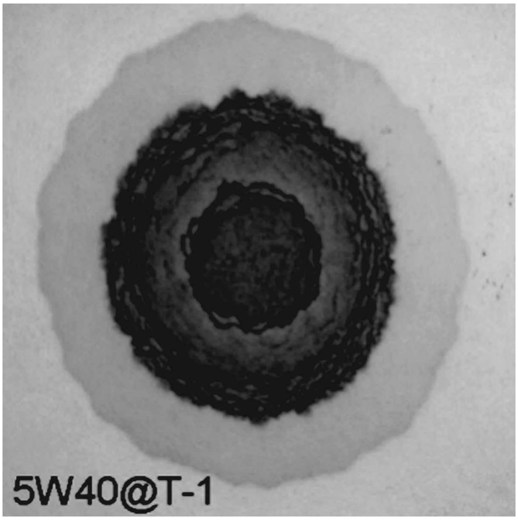


FIG. 1—*Blotter spot example.*

of an index of dispersancy is given by the measurement of the differences in size of the diffusion rings of the oil and the pollutants.

The Limits of the Method

Visual Rating—The current "blotter test" method by visual quotation remains subjective and not based on mathematical models (refer to Fig. 2).

Although the blotter test offers limited value measuring soot concentration, it provides an excellent assessment of the lubricant's dispersancy performance. An oil that is properly dispersing soot and other insolubles produces an evenly graduated blotter (see spot 1). A blotter indicating a high soot load, but even graduation, suggests the oil is still fit for service, but should be watched closely for degradation (see spot 4). When dispersancy begins to fail, the insolubles begin to form a dense ring on the exterior of the absorbing oil drop as seen in spot 7. Spot 9 indicates the characteristic dense black dot and sharp periphery that forms when the oil completely loses dispersancy performance. From a maintenance perspective, when the ring begins to form around on the exterior of the oil blotter, it is time to look at scheduling an oil change.

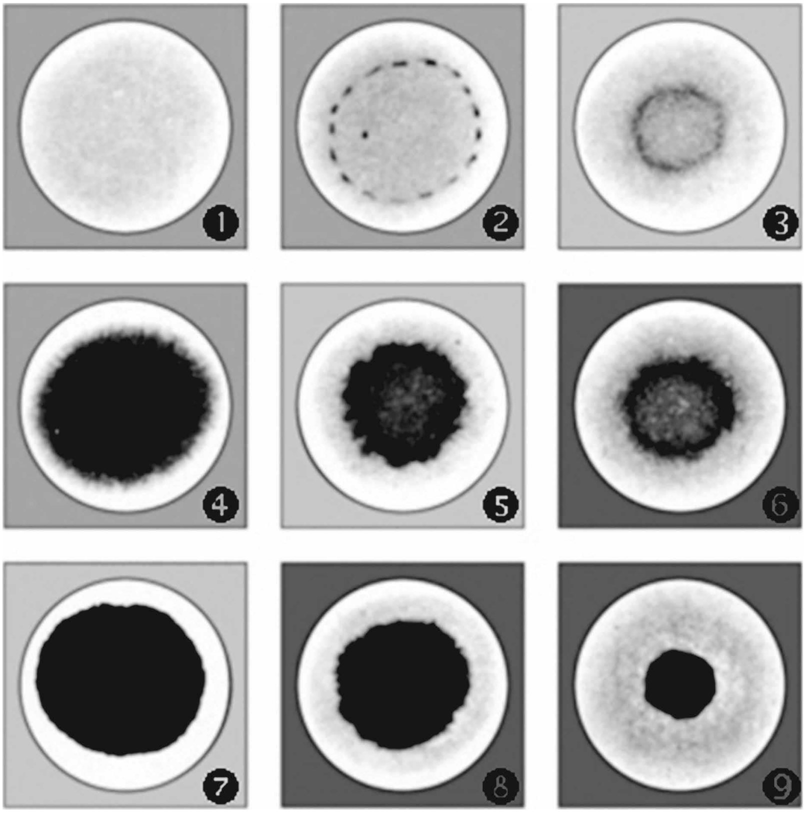


FIG. 2—*Lubricants at different level of wear.*

Certain laboratories have established their own quotation method of the spot by a measurement of the diameter of certain haloes (or rings) and calculations of diameter ratios. Because these methods are not published, it is almost impossible to date to compare the results obtained between various laboratories. Moreover, with the introduction of new oils and new fuels (e.g., biodiesel, ethanol) we note the appearance of multiple rings caused by various pollutants (carbon particles, EMHV, etc). *For all these reasons, the visual/manual interpretation of the various rings became very complicated and not easily exploitable.*

Automated Analysis—There exists an automated apparatus that facilitates the interpretation of the spot and eliminates the subjective aspect from the manual method. This instrument is equipped with a monochromic charge coupled device (CCD) camera and does not use the information color of the spot and does not differentiate each ring of the spot. This apparatus compares the diameter of the spot with a theoretical diameter and analyzes the opacity homogeneity of the spot. Of these two parameters, the apparatus calculates a dispersancy index that varies from 0 to 100 (100 = ideal dispersancy).

For our study, we decided to start from this instrument version and to improve it in order to be able to discriminate the rings that compose the spot.

The Filter Paper Importance—During our research, we noticed that many types of papers are currently used. The tests carried out during our study showed us an important discrepancy of results between papers that theoretically had the same characteristics but did not produce the same developments of spots. This phenomenon makes impossible any result comparison between laboratories. The team thus tested several types of filter paper in order to retain that which offered an optimal discrimination of the rings.

The New Approach

The goal of our study was not to reinvent the blotter test. We used a computer associated with a dedicated software that we specifically developed for recognition and analysis of color image. The contribution of this technology gives another dimension to this method. The work presented hereafter gathers a digest of many experiments and specific research. With the digital imaging analysis of the spot, in particular its opacity and its spreading out by means of a dedicated innovative algorithm and the choice of perfectly adapted filter paper, it becomes possible to evaluate with an objective and quantified way the residual power of a lubricant to disperse the insoluble matters.

Method Description

The general principle of the method for the preparation of the spot remains quasi unchanged. It consists of depositing a volume of 15 μl of oil on a specific filter paper and analyzing the rings of the spot, which are representative of the dispersion of the pollutants. For two reasons, the sample volume was decreased down to 15 μl instead of 20 μl. The first reason was to limit the size of the spot

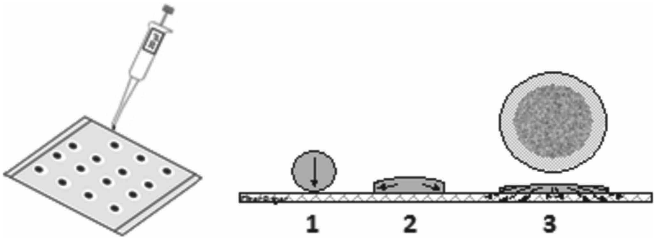


FIG. 3—*Oil spot preparation.*

and to make it compatible with the image analysis system and the second reason was to be able to analyze all types of lubricants (refer to Figs. 3 and 4).

The deposit of the oil on the filter paper is carried out at room temperature or, in certain cases, the sample is deposited at 200°C in order to free itself from the viscosity of the sample. The filter paper is then placed in a drying oven at 100°C for 24 h.

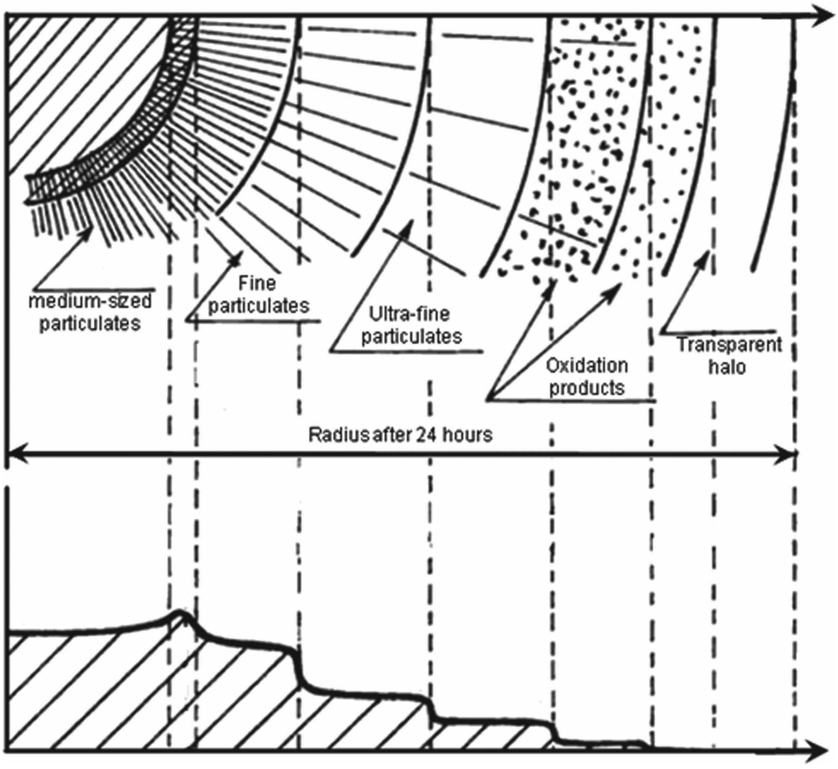


FIG. 4—*Diagrammatic representation of radial distribution of an oil spot (extracted from an abstract written by E. Sibenaler [1], published in 1971 in Technip Editions book [2].*

Note: In our study on the impact of biofuel on the lubricant, we chose to limit the heating temperature at 200°C to reduce the risk of projection related to a possible presence of fuel in the samples.

The Filter Paper

The precise and repeatable measurement of the opacity of the different rings requires a specific support that satisfactorily disperses all the pollutants. The tests carried out on different filter papers in terms of density, porosity and composition made it possible to choose a new homogeneous filter paper. Work was undertaken on oils resulting from field driving, from gasoline and Diesel engines, and after aging tests simulated in laboratory.

Instrument

Refer to Fig. 5.

Principle

The instrument is composed of the following elements:

- A light source positioned above the measurement table (direct light).
- A color CCD camera equipped with a high resolution.
- A dedicated software capable to monitor both the light source and the camera. The software memorizes the numeric color picture of the spot.

The instrument thus equipped makes it possible to take a digital color picture of the spot such as the human eye sees it, but with higher resolution. The

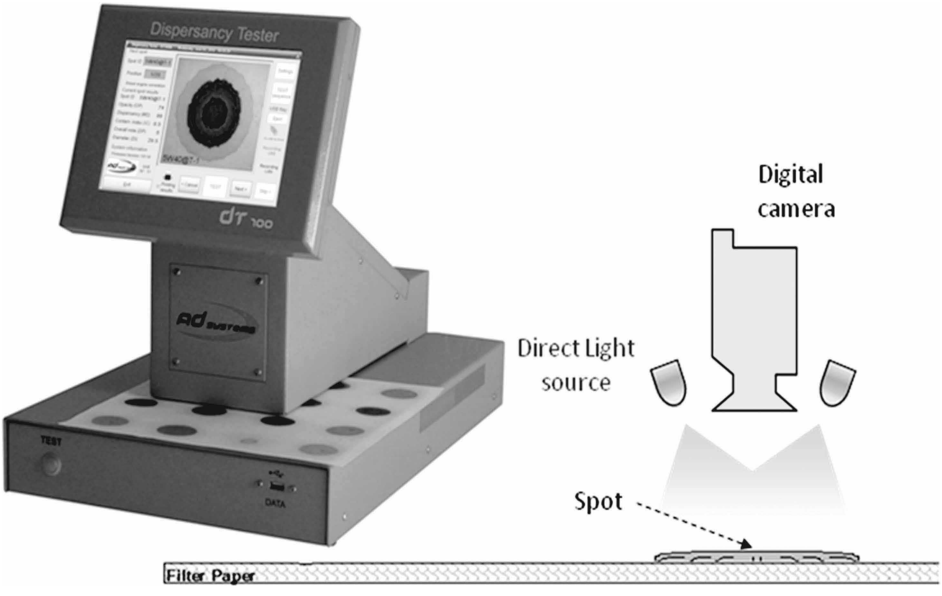


FIG. 5—*DT 200 Dipersancy Tester Instrument.*

image is memorized for the treatment. Moreover, this image is added to the test report and thus makes it possible to visually check the results reported by the software. The use of a color camera makes it possible to identify the various rings by obtaining chromatic information on the spot.

In order to always analyze the spot under the same conditions of lighting and thus of optimized reproducibility, the calibration of the device is carried out on a white sheet of filter paper, the one used for the tests.

Parameters Measured

The software reports the following images and data:

- The color image of the spot such as the human eye perceives it.
- The digital model in levels of gray associated each one with an opacity of the delimited ring by its real form.
- The number of rings present in the spot, ring 1 being the last external ring.
- The diameter of each ring.
- The surface (in mm^2) of each ring.
- The average opacity of each ring.

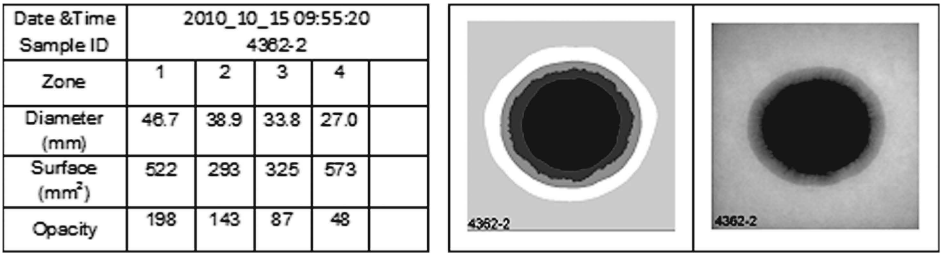
The software was designed to be able to analyze a series of spots coming from the same oil at various stages of degradation. We created this possibility in order to carry out a follow-up of each value measured during artificial life tests and also during the follow-up of engine (refer to Fig. 6).

Conclusions

As we have just seen, the instrument and the process we developed make it possible to obtain results of dispersancy analysis in a numeric format. With this technique, the detection of the rings is much more precise and repeatable. With this inherent, it is now possible for us to check that this method can be a powerful tool to work on the problems we are facing.

Result Examples

The work done to select the best, as well as the development of the dedicated software described previously, required many months. Because of this long study, the team has not yet had time to carry out all the tests on all the targeted directions.



Date &Time Sample ID	2010_10_15 09:55:20 4362-2				
Zone	1	2	3	4	
Diameter (mm)	46.7	38.9	33.8	27.0	
Surface (mm^2)	522	293	325	573	
Opacity	198	143	87	48	

FIG. 6—*Reporting example.*

The results presented hereafter are the preliminary results of the study and do not cover all the applications that this method could cover. These results are very encouraging and although we are at the starting point, they confirm all the potential of the method that we developed.

Thermal Qualification of an Engine Oil

During the performance evaluation of oils by artificial life tests, it is necessary to follow the evolution of their dispersancy properties for better knowledge of the limits of each oil in terms of durability. This gives us an idea of the dispersion of insoluble of oxidation and the resistance of the oil to the pollutants such as the fuels and the biofuels.

Before testing the method on lubricants contaminated by biofuel, we wanted to test two lubricants that we consider as our internal references:

- RH 2010, engine oil qualified as high level reference.
- RL 2010, engine oil qualified as low level reference; judged as "borderline."

PSA has its own aging method for the evaluation of thermal behavior of engine oil. The new and pure engine oil is stressed with an accelerated aging with a thermal stress (170 °C), *in the presence of oxygen and an oxidation proprietary catalyst*.

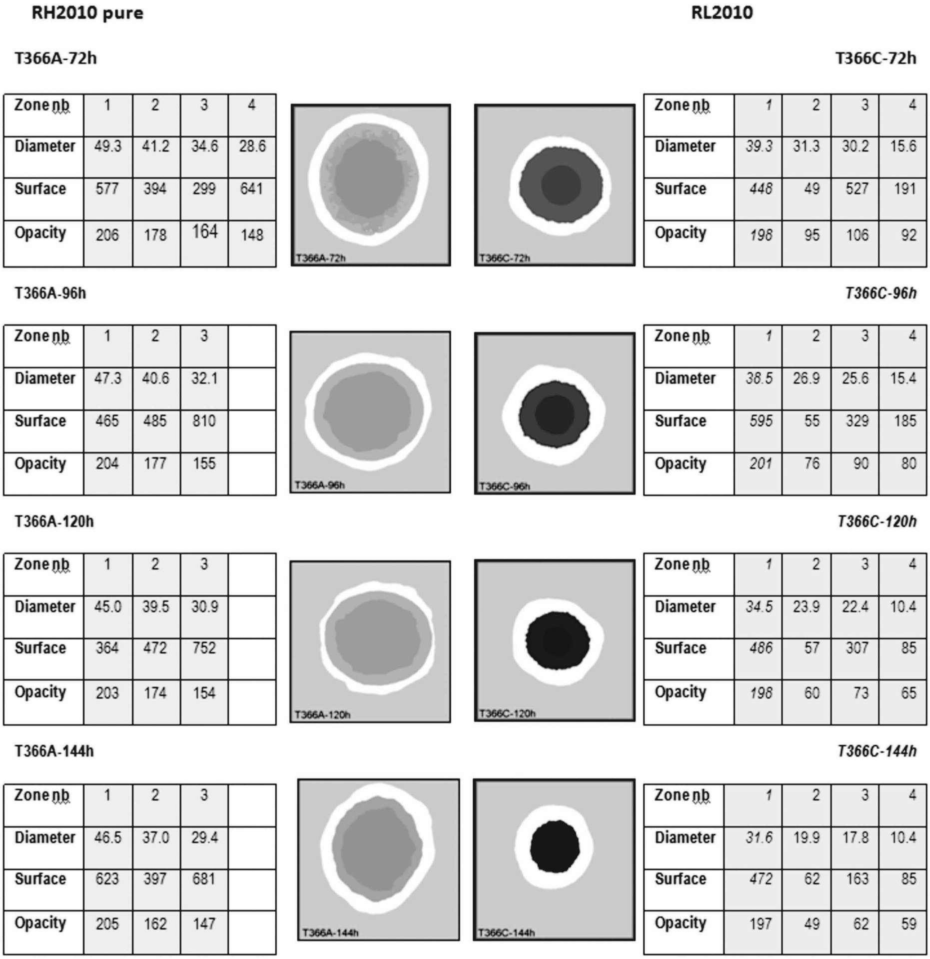
After 72, 96, 120, and 144 h, some samples are taken. Each sampling is then analyzed with the method and the tool we described earlier (refer to Fig. 7).

Observations: We observe a more important spreading out of high reference oil RH2010 compared to borderline oil RL2010. In addition, one can notice that opacities of the central rings are much darker. These tests carried out with the new blotter test method that we developed and described here, confirm if needed the respective level of quality of the two engine oils. In this particular case, we confirm their capacity to disperse insoluble oxidation matter.

Thermal Qualification of an Engine Oil in the Presence of Biofuel

We took again the same two qualified engine oils, the high reference and the borderline reference. We stressed these two oils with our aging method, but with the aim of simulating reality, generated by the use of oil standard European diesel B10, we added diesel B10 starting from 72 h of test. Then, the contamination level of diesel B10 was maintained to 10% during the remainder of the test, this method available from the GFC. (The Groupement Français de Coordination - GFC (www.gfc-tests.org) is a non-profit making association created in 1963 at the instigation of the Institut Français du Pétrole for the purpose of developing and fine tuning test methods that are essential to the oil industry, motorists and users.) (Refer to Fig. 8.)

Observations: We observe a reduction in spreading out and a more important opacity in the presence of biofuel GOPSA10LUB for the high reference oil. The RH2010 oil approaches the rupture limit at the end of the 144 h, but the



RH2010 pure

T366A-72h

Zone nb	1	2	3	4
Diameter	49.3	41.2	34.6	28.6
Surface	577	394	299	641
Opacity	206	178	164	148

T366A-96h

Zone nb	1	2	3	
Diameter	47.3	40.6	32.1	
Surface	465	485	810	
Opacity	204	177	155	

T366A-120h

Zone nb	1	2	3	
Diameter	45.0	39.5	30.9	
Surface	364	472	752	
Opacity	203	174	154	

T366A-144h

Zone nb	1	2	3	
Diameter	46.5	37.0	29.4	
Surface	623	397	681	
Opacity	205	162	147	

RL2010

T366C-72h

Zone nb	1	2	3	4
Diameter	39.3	31.3	30.2	15.6
Surface	448	49	527	191
Opacity	198	95	106	92

T366C-96h

Zone nb	1	2	3	4
Diameter	38.5	26.9	25.6	15.4
Surface	595	55	329	185
Opacity	201	76	90	80

T366C-120h

Zone nb	1	2	3	4
Diameter	34.5	23.9	22.4	10.4
Surface	486	57	307	85
Opacity	198	60	73	65

T366C-144h

Zone nb	1	2	3	4
Diameter	31.6	19.9	17.8	10.4
Surface	472	62	163	85
Opacity	197	49	62	59

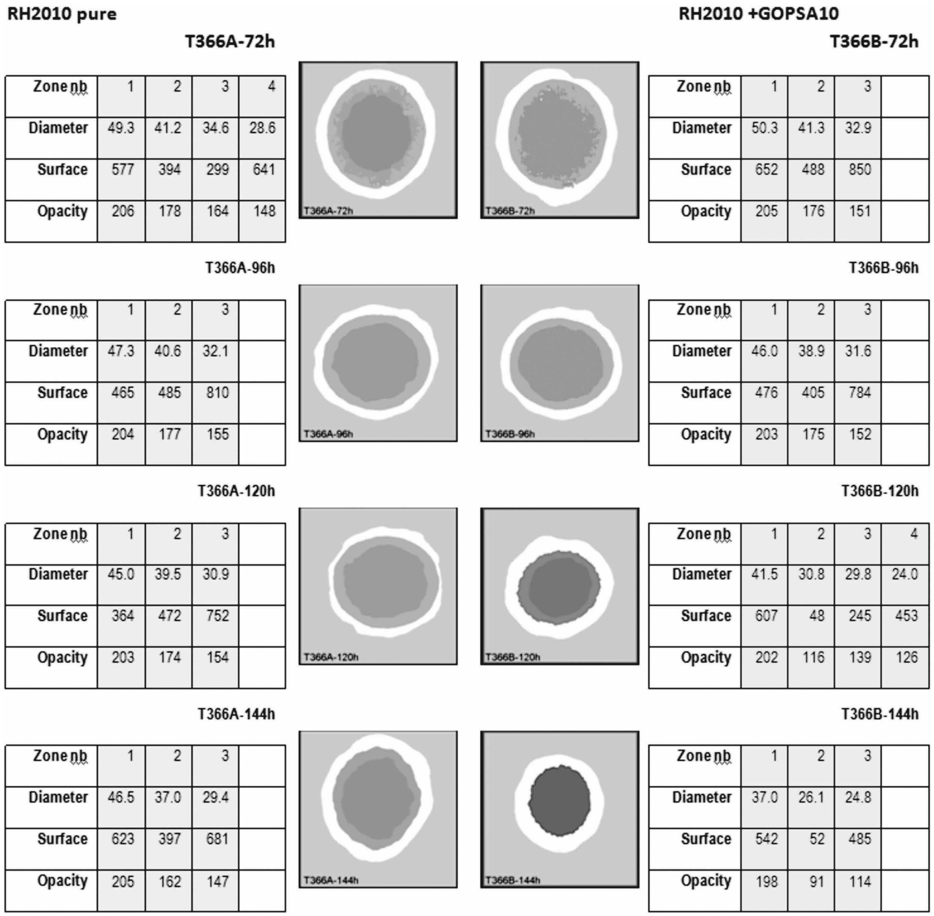
FIG. 7—*Test results with engine oils RH2010 and RL2010.*

total result according to our criteria remains satisfactory, although the bad dispersancy in the presence of a biofuel is highlighted (refer to Fig. 9).

Observations: With this engine oil evaluated as border line, we observe a reduction in spreading out in the presence of biofuel GOPSA10, this result becomes critical with respect to the acceptable requirements which are based over the duration of 120 h.

Conclusion

Although it remains up to us to develop a relevant mathematical model, we can affirm that this method will make it possible to determine the dispersancy of an oil by its capacity to disperse, in particular, the insoluble matters.



RH2010 pure

T366A-72h

Zone nb	1	2	3	4
Diameter	49.3	41.2	34.6	28.6
Surface	577	394	299	641
Opacity	206	178	164	148

T366A-96h

Zone nb	1	2	3	
Diameter	47.3	40.6	32.1	
Surface	465	485	810	
Opacity	204	177	155	

T366A-120h

Zone nb	1	2	3	
Diameter	45.0	39.5	30.9	
Surface	364	472	752	
Opacity	203	174	154	

T366A-144h

Zone nb	1	2	3	
Diameter	46.5	37.0	29.4	
Surface	623	397	681	
Opacity	205	162	147	

RH2010 +GOPSA10

T366B-72h

Zone nb	1	2	3	
Diameter	50.3	41.3	32.9	
Surface	652	488	850	
Opacity	205	176	151	

T366B-96h

Zone nb	1	2	3	
Diameter	46.0	38.9	31.6	
Surface	476	405	784	
Opacity	203	175	152	

T366B-120h

Zone nb	1	2	3	4
Diameter	41.5	30.8	29.8	24.0
Surface	607	48	245	453
Opacity	202	116	139	126

T366B-144h

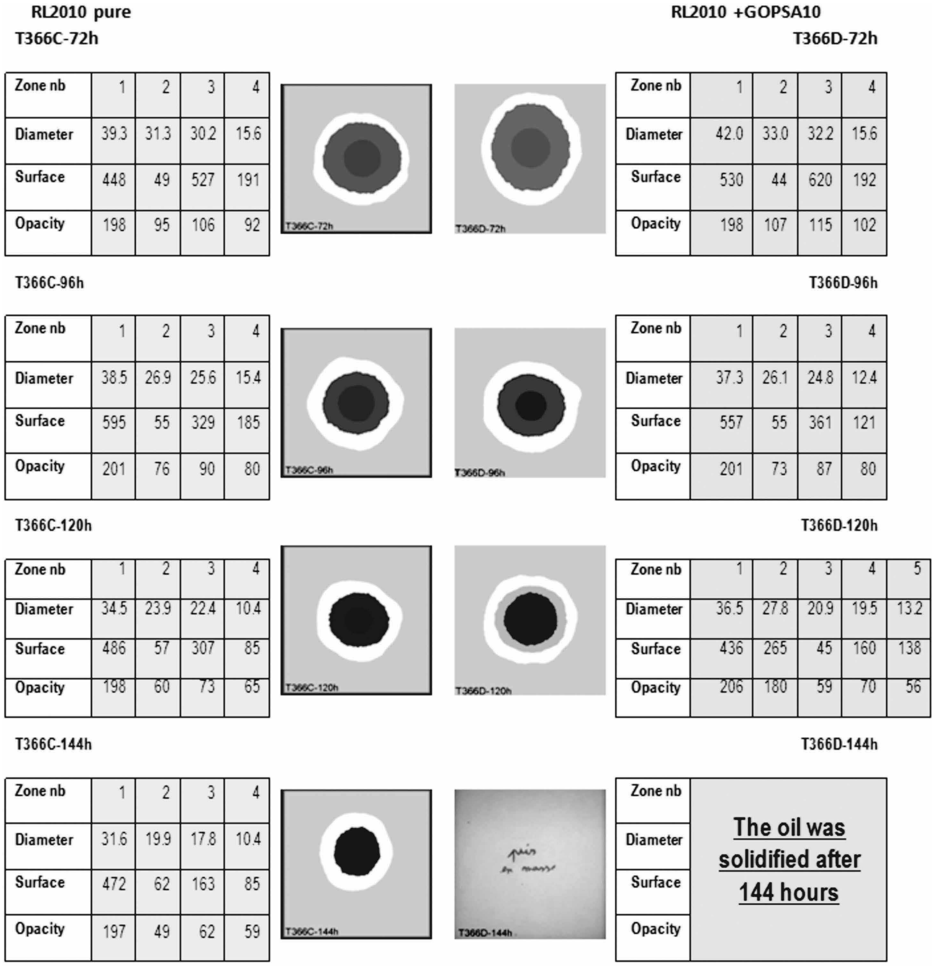
Zone nb	1	2	3	
Diameter	37.0	26.1	24.8	
Surface	542	52	485	
Opacity	198	91	114	

FIG. 8—*Test results with engine oils RH2010 in presence of biofuel (GOPSA10 = diesel B10).*

In technical terms, the developed method makes it possible to measure the dispersancy power in an objective way, which will be based on a mathematical model.

It will allow a precise follow-up of the evolution of dispersancy by a simple handling and will be able to precisely evaluate the resistance of a new oil to disperse the insoluble matters when submitted to oxidation test and/or thermal behavior test. Last, it will determine the impact of pollutants such as biofuel on the dispersancy capacity of oils thanks to the precise measurement of each ring.

In technical-economic terms, the invention improves considerably the processes known before by a precise follow-up of the dispersancy of the lubricant and its degradation; thus making it possible to control the cleanliness of specific



RL2010 pure

T366C-72h

Zone nb	1	2	3	4
Diameter	39.3	31.3	30.2	15.6
Surface	448	49	527	191
Opacity	198	95	106	92

T366C-96h

Zone nb	1	2	3	4
Diameter	38.5	26.9	25.6	15.4
Surface	595	55	329	185
Opacity	201	76	90	80

T366C-120h

Zone nb	1	2	3	4
Diameter	34.5	23.9	22.4	10.4
Surface	486	57	307	85
Opacity	198	60	73	65

T366C-144h

Zone nb	1	2	3	4
Diameter	31.6	19.9	17.8	10.4
Surface	472	62	163	85
Opacity	197	49	62	59

RL2010 +GOPSA10

T366D-72h

Zone nb	1	2	3	4
Diameter	42.0	33.0	32.2	15.6
Surface	530	44	620	192
Opacity	198	107	115	102

T366D-96h

Zone nb	1	2	3	4
Diameter	37.3	26.1	24.8	12.4
Surface	557	55	361	121
Opacity	201	73	87	80

T366D-120h

Zone nb	1	2	3	4	5
Diameter	36.5	27.8	20.9	19.5	13.2
Surface	436	265	45	160	138
Opacity	206	180	59	70	56

T366D-144h

Zone nb	**The oil was solidified after 144 hours**
Diameter	
Surface	
Opacity	

FIG. 9—*Test results with engine oils RL2010 in presence of biofuel (GOPSA10 = diesel B10).*

elements of a motor vehicle, such as the engine or the gear box by the follow-up of the impact on the dispersancy additives.

The process and the device of the invention are usable in laboratory and on engine benches or rolling vehicles, for any mechanical parts lubricated with an oil, such as, for example, a marine engine or a wind turbine, for many types of oils including industrial oils, the cutting oils, and others.

We can also define specific criteria of calculation for oils resulting from rolling bench or in-service vehicle

By analyzing the measured parameters in each ring, it should be possible to determine the types of pollutants present in oil and their implication on dispersancy. Thus, it becomes possible to have an indication on the cleanliness of the bodies and to quantify in a precise way the pollutants in oil (soot resulting from the combustion of the fuel, metal particles due to the wear and the corrosion of the bodies, products resulting from the aging of oil).

The Perspectives and Working Axes

For the qualification of a new oil, engine or transmission applications, based on the data of a series of spots coming from successive samplings during an aging test, it should be possible to:

- Calculate its dispersancy potential.
- To evaluate its potential to resist oxidation and thermal degradation and consequently to know the impact on the oil dispersancy of any pollution and/or biofuel, in particular of the nitrated products or other pollutants according to the mode of use studied.

For in-service oils, engine or transmission applications, it is necessary to control the evolution of their dispersive properties during their use. It is important to be able to define a criterion of acceptance of oil; in other words a criterion making it possible mainly to define the adequate step of maintenance or the wear status of oil.

Several types of pollutants can be identified in an oil:

For gasoline engines—

- The capacity of the additives to disperse the insoluble matters resulting from oxidation.
- The preservation of the additives performance in presence of ethanol biofuels or others.

For diesel engines—

- The capacity of the additives to disperse the soot in addition to the insoluble matters resulting from oxidation.
- The preservation of the additive performances in presence of biodiesel (EMHV).
- This list is not limited.

Based on the parameters reported by the instrument, such spreading surfaces of oil and pollutants, the opacity indices of oil and pollutants, it should be possible to calculate the dispersancy power in an objective way based on a mathematical model. In addition, an analysis of the parameters measured in each ring, should make it possible to recognize the types of pollutants present and their implication on dispersancy. It is on these issues that our team currently works. Many tests are carried out in order to establish the correlations with the known laboratory methods. The use of this new test in conjunction with DV4 engine rig test demonstrates that this long and expensive engine test can be partially replaced by the described procedure. This new approach already lets one foresee a need for specific equipment on which our team currently works:

A laboratory instrument that could report the following parameters:

- The dispersancy power of the oil.

- The type of each pollutant by the detailed analysis of each ring.
- Insoluble matters quantification.

A field instrument that could:

- Indicate the wear level of oil with a simple and automatic test.
- Quantify soot and/or insoluble matters.

Speaker Biographies

Didier Pigeon, President of AD Systems

Didier Pigeon has worked for over 32 years for one of the world leaders in instrumentation for petroleum industry. As Vice President of Marketing in this company, he was responsible for the definition of the product portfolio specifications. Didier created his own company AD systems in 2008. Didier Pigeon is a member of ASTM, EI, BNPé, CEC, and GFC. He wrote several ASTM methods (Automated Pour Point, Automated Cloud Point, Automated Noack, Automatic Freeze Point, Micro distillation, Etc.).

Didier's key achievements include:

- Development of a non-wood metal Noack instrument in 1997.
- Development of a Lubricant Dispersancy tester in 2009.
- Development of a Thermal Deposit Rater for jet fuel thermal oxidation test in 2010.

Gérard Abellaneda, Lubricant Expert, PSA PEUGEOT CITROEN

Gérard ABELLANEDA has been working for 35 years in the development of engine lubricants. He is the Functional Laboratory Manager for the development of lubricant for PSA. Gérard Abellaneda is a member of CEC and GFC and mainly works on lubricant aging methods

His main activities include:

- Development of lubricants in adequacy with the new engine or transmission projects.
- Development of laboratory methods for lubricant evaluation.

Gérard Abellaneda is the author or coauthor of many articles, such as:

- Performance at high temperature of engine oils—1989 CEC Symposium.
- Prediction of bearing corrosion and correlation with the Petter W1 L engine test—1996 Tribotest Journal and 1993 CEC Symposium.
- Coking and Micro coking: Tools for evaluating and developing lubricant additives—1997 Tribotest Journal.

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Andrea Wardlow[1] *and Jo Ameye*[2]

Experiences with ASTM D02 Practices D4378 and D6224 for Turbine Oils and Auxiliary Power Plant Equipment Condition Monitoring Programs

ABSTRACT: Condition monitoring practices ASTM D4378 [2011, "Section Five, Petroleum Products, Lubricants, and Fossil Fuels," *Annual Book of ASTM Standards*, Vol. 05.02, ASTM International, West Conshohocken, PA] and ASTM D6224 [2009, "Standard Practice for In-Service Monitoring of Lubricating Oil for Auxiliary Power Plant Equipment," *Annual Book of ASTM Standards*, Vol. 05.02, ASTM International, West Conshohocken, PA] are important activities at the SC C00 as they are the benchmark today in the power generation industry for condition monitoring and maintenance practices. With the need for increased equipment and plant efficiency in the modern economy, these methods contribute to an improved reliability and better utilization of modern turbine lubricants. In conjunction with the latest ASTM D4303 [*Annual Book of ASTM Standards*, ASTM International, West Conshohocken, PA] (the new turbine oil specification) these methods help the end-users to better understand the condition of their in-service lubricants and by turning them into immediate maintenance actions. This paper will include a few case studies, presenting the different experiences with the integration of these in-service practices, and how they are evolving with the original equipment manufacturers' equipment developments and turbine lube oil developments.

KEYWORDS: power generation, condition monitoring, in-service lubricants, steam turbines, gas turbines, oil analysis parameters

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Introduction

Standard practices ASTM D4378 [1] and ASTM D6224 [2] are important activities at the SC C00 as they are the benchmark today in the power generation industry as part of condition monitoring and maintenance practices. In today's cost-cutting, globally competitive market, there is perhaps no more critical area in terms of plant asset profitability than in the successful execution of a balanced maintenance program. Balancing corrective, preventive (scheduled), and predictive (failure symptom monitoring), and proactive (failure root cause monitoring) maintenance activities will enable a high degree of equipment availability in combination with an optimized lubricating oil interval. A properly executed lubricating oil monitoring (screening) program provides a natural balance of providing information for all these areas of maintenance.

As part of the ASTM D02 Subcommittee C00 on turbine oils, we have active participation from original equipment manufacturers' (OEM's) end-users such as power generation plants, the Navy, as well as oil and additive suppliers. During the last decade, it has become apparent with the introduction of a new generation of high performance turbine lubricants, as well the high installation rate of new power plants, such as gas turbines or combined cycle plants, that new oil monitoring test methods and strategies need to be defined or refined. Questions from end-users such as: "What are the critical lube oil parameters?," "How do I integrate this with the new lube oil parameters (e.g., ASTM D4304 [3])?," "What are the condemning limits?," "How do I adapt the oil sampling frequency as a function of the oils condition?" have convinced the ASTM D02 C00 members that continuously updating the guidance and latest best power industry practices was and still is of high value.

These questions clearly convinced the ASTM D02 C00 members that the need for developing and maintaining an in-service practice would be of high value. This paper will describe the two standards in detail, how the changes in operating conditions and turbine equipment affected the methods, how to integrate the data as part of trending analysis, and how to integrate or interpret the test data.

We will also provide the different experiences that the ASTM Subcommittee SC 00 on Turbine oils has had over the last 10 years, and how other ASTM subcommittees could benefit from these experiences.

Objectives of D4378 and D6224 Practices

All diagnostic, predictive, and proactive maintenance technologies require intimate knowledge of the equipment, its internal design, the system design, and the present operating and environmental conditions for successful execution. The maintenance professionals are in the best position to understand and integrate this information, but need an effective lubricant monitoring program. As a result, oil analysis programs are in the evolution of change. In addition to traditional, laboratory-driven oil analysis programs, more maintenance personnel are starting to perform interim on-site testing as a proactive qualitative indication of the in-service oil condition during interim periods between lab analyses.

This transition requires clear users procedures and the ASTM practices D4378 and D6224 are a very good resource for this.

D4378 and D6224 practices are intended to provide utilities and industries which generate their own electrical power with recommendations on how to establish an effective condition monitoring program for mineral turbine oils in service in steam, gas or combined cycle systems, as well as common auxiliary equipment (e.g., pumps, compressors, gearboxes, etc.) found in power plants. These practices are applicable to an individual coal plant, hydro plant, as well as, combined cycle co-gen plants where a gas and steam turbine is configured in tandem for more efficient power generation. When ASTM D4378, was initially developed [4], the goal of this method was to offer end-users, lubricating oil professionals, as well as OEMs active tools and guidelines on proper sampling procedures, suitable testing schedules for in-service lubricants as well as warning limits, and corrective actions for maintaining lubricants throughout their life cycle. As a result of the use of these practices, lubrication professionals are better equipped with information to help keep the condition of the turbine lubricating oil as close as possible to “fit-for-service” target values, and avoid unnecessary maintenance actions, due to excessive oil degradation. When implemented correctly, the ASTM practice will generate useful data to assist end-users in conducting root cause failure analysis (RCFA) strategies, where the return on investment can be increased significantly. The D4378 practices cover the requirements for the effective monitoring of mineral turbine oils in service in steam and gas turbines, to ensure their long, trouble-free operation of the power plant equipment.

Field experiences confirm that following factors affect the degradation of lubricants:

- Oxidation through oxygen/air contact with the lubricant.
- Oxidation through metal or water contamination.
- Temperature extremes, better known as hot spots.
- Design system of the turbine (oil circulation rate, bearing design, oil reservoir size).

In addition to these, certain industries, such as the gas turbine industry in the USA (with a high increase of installation of gas turbines over the last 10 years), acknowledge the impact of new operating conditions (peak to base-load) to their financial performance. A practice should have the right parameters in order to closely monitor these operating conditions.

Additionally, a new generation of turbine oils is formulated differently than their ancestors. This includes a switch from group I to group II (and beyond) basestocks, coupled with the incorporation of more complex and effective antioxidant chemistries and other additives to balance out performance for varying applications. Reliance on existing analytical techniques has caught many users off-guard, as these tests are no longer the predictive tool that they once were. The ASTM D02.C00 Subcommittee on turbine oils acknowledge the challenges plant operators face, prompting us to rethink how best to monitor this next generation mineral-based turbine oils. Examples of this consideration include what tests may aid in detecting soft contaminants versus hard contaminants, or how to adapt original condemning limits as a function of the type of equipment.

When these ASTM practices have been correctly integrated into a oil monitoring program, the expectation is that relevant data are generated to enable end-users to conduct RCFA strategies, and take the appropriate corrective actions to maximize the return on investment and improve the financial bottom-line.

Description of the Practices

Both practices are intended to assist end-users, particularly power plant operators, in maintaining effective lubrication of turbine machinery (D4378) as well as associated auxiliary power equipment (D6224), and to provide guidance to guard against the onset of problems associated with oil degradation and/or contamination.

Summary of Practice

- Scope or Introduction—gives an overview of the application areas covered by the practice.
- Referenced Documents—lists ASTM and other international organization standards references in the practice.
- Significance and Use—as described in the above paragraph.
- Properties of Turbine Oils—provides some general statements about desirable oil properties and general formula approaches employed to achieve a required performance feature. For more specific recommendations on turbine oil minimum acceptance properties, see also ASTM D4304 [3].
- Operational Factors Affecting Service Life—describes factors that affect the service life of lubricating oils, include type and design of the system, condition of system on start-up, original oil quality, system operating conditions, contamination, oil make-up rate and handling, and storage conditions.
- Sampling—covers best locations to obtain representative samples, appropriate sample bottles, labeling, etc.
- Deterioration of Turbine Oils in Service—gives an overview of the chemical and physical changes an oil will eventually undergo in-service due to thermal and oxidative stresses, contamination, or combinations thereof.
- Monitoring Program—describes the four legs of a sound monitoring program: (1) sampling and testing at appropriate intervals, (2) trending and data interpretation, (3) corrective action, and (4) maintenance follow-up.
- Sludge and Deposits—highlights that deposits in turbines and auxiliary equipment are cause for concern. Analyses can be performed to identify possible root causes; action may need to be taken to clean out the system and replace the oil charge.
- Testing Schedules—recommends inspection tests for new oil receipts as well as periodic in-service oil monitoring test schedules by equipment type, e.g., gas turbine, steam turbine, etc.

Latest Revisions to the Practices

The objective of recent changes and additions was to update the Practices to reflect the latest industrial evolutions from both the lubricant as well equipment viewpoint, and to assure that these standards cover the best condition monitoring practices. A summary of recent changes and proposed changes under consideration within Subcommittee C is given below.

- D4378 scope was expanded to include combined cycle turbines equipment.
- Reference Documents are updated as new standards suitable for condition monitoring are published. For example, the New ASTM D7647 [5] will be included in the next revisions.
- The impact of changes in system operating conditions (whether a unit is operated continuously 24 h/7days or in on/off cycles), has a significant impact on oil degradation rates and tendency to form sludge and varnish deposits. Caution is given on reliance on traditional tests like RPVOT by ASTM D2272 [6] to detect if some oils have undergone sufficient thermal-oxidative degradation to causes operational problems. (See D4378-08 [1], Table III, footnote A).
- Section 6 on sampling has been revised to reflect current best practices. Emphasis is placed on the use of appropriate clean bottles (for meaningful results, any old container really will not do) that are resistant to the material being tested. Proper labeling is critical to track the history of the equipment, sampling date, location, and identity of the lubricant sampled.
- Tables including recommended sampling and testing schedules by turbine or equipment type are being reformatted to consolidate sampling frequency, test methods, warning limits, interpretations, and recommended actions into a single table per application. Footnotes will be used sparingly to ensure important caveats or cautions are not overlooked.

Changes in Operating Conditions and Industry—In this section, we will go deeper in the consequences that have resulted from the change of operational requirements for new generation of turbine technologies, and how it has affected the development of the ASTM D6224 and 4378 practices. In Table 1, new turbine oil systems have higher needs for oil monitoring practices, and are today also differentiated in function of its application, e.g., water, steam and gas (combined cycle). In particular, gas turbines need special attention, as they represent the most demanding application for turbine oils, and are also the largest growing market in the US for power generation. In 2010, 81 % of new power generation production is forecasted to be provided by gas turbines. In 1998, 15 % of US electrical power was provided by gas turbines. In 2020, gas turbines are projected to provide 39 % of the US's electricity [7].

The impact on the oil monitoring practices can be noticed on three levels:

- Temperature—a general trend can be noticed on the increase of the oil sump temperatures, due to different factors such as higher combustion temperatures (gas turbines), smaller oil reservoir size, or higher

TABLE 1—*Summary of operating conditions for hydro, steam, and gas turbines.*

	Water	Steam	Gas
Critical Turbine Components	Bearings Guide Vanes Control System	Bearings Control System	Bearings Gears Control System
Speeds, rpm	50–600	>3000	3000–7000
Oil sump temp., °C	40–60	45-65	50–95
Hot spot peaks, °C	75–90	80-150	150–280
Unfavorable impact	(water) air	(steam) air	air high temp.
Oil service life, thousand hours	100–250	50–150	20–50

circulation rates. The firing temperature of gas turbines is continually rising as more advanced metallurgies are developed. There is a general belief that an increase in firing temperature causes an increase in lubricant stress. Overall, this is a correct trend since the first gas turbine for power generation was installed in the Oklahoma in 1949. However, the stress on turbine oils is more complex than just understanding inlet firing temperatures. Even knowledge of some of the basic lubricant parameters may be insufficient in properly comparing the stress on the lubricant. Table 2 illustrates this point as an older gas turbine technology "class E" appears to have more lubricant stress compared to the more modern "class F."

- This has a direct impact on the bearing and gear temperatures as critical components from the turbine. This does not take into account hot spots.
- Extended oil lifetime—With the introduction of long-life, high performance industrial lubricants, the need for up-to-date oil monitoring methods become critical. By extending the useful life of the lubricants, it will not only be critical to understand how actual oil parameters have evolved, but also how new parameters will be necessary. For example a parameter change might be individual antioxidant monitoring versus total oxidative life of the oil.
- Operating mode of power plants—Except for steam turbines in service at nuclear plants, gas and steam turbine oils have to work from baseload to peaking, which is also creating a need for new monitoring methods (Fig. 1). Most turbine oil formulations on the market and in use

TABLE 2—*Comparison of the turbine oil environment in various gas turbine technologies.*

Model	Lubricant Residence Time (min)	Hottest Bearing Temperature (°F)	Reservoir Temperature (°F)
Class E	5.8	500	120–135
Class F	7.4	250	125–140

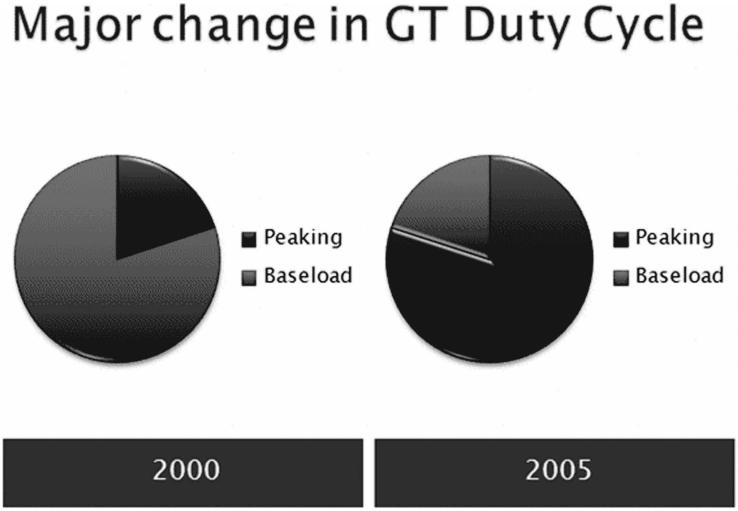

FIG. 1—*Evolution of gas turbine duty cycle between 2000 and 2005.*

today are quite different than those in the recent past. Today's turbine oils are required to perform more functions in increasingly harsh environments. In some of the more unforgiving scenarios, the same reservoir of fluid simultaneously provides lubrication to the turbine bearings, generator bearings, atomizing air compressors, lift oil system, trip oil system, the generator hydrogen seal system, load gears, and a multiplicity of servo valves within the hydraulic circuit. Similarly, monitoring the life of these fluids has been done by well established tests that provide early warning of problems and allow the user plenty of time to take proactive actions prior to any impact on a plant's reliability.

- The time has come to rethink how to monitor turbine oils to provide early warnings of incipient lubricant failure in an effort to optimize the life and performance of these critical fluids. The practices D4378 and D6224 are periodically reviewed and revised to keep pace with the industry's best practices and to educate all interested parties on possible root causes of normal and abnormal oil degradation and the benefits and drawbacks that may be encountered while applying the latest available lab oil analysis tests and on-site tools.

The Importance of Trend Analysis for Turbine Oils: Date Interpretation and Integration

When integrating and using the ASTM D4378 and D6224 Practices, data trending can provide direction into the timeframe of preventive, corrective maintenance actions to be performed when limits or targets are exceeded. The comparison can be easily made between making one picture (1 data point), in comparison to a video (multiple data points), which is presenting the lifetime of the turbine lubricating oil system, knowing that some of these oil systems have a life-time of over 100 000 h. By establishing multiple data points, lubrication professionals will be able to discern accumulated patterns and/or identify acute events occurring within a specific lubricating oil system, or per type of equipment. At any point in a component's life, conditions may exist that will shorten the life if left unattended. The promptness with which these conditions are corrected directly affects the component life. Examples of such conditions are wrong oil and viscosity, fuel contamination, dirt contamination, poor lube condition, and water contamination. This can be of particularly high importance for critical equipment that needs to stay in permanent availability, such as nuclear main feedwater pumps, gas compression stations, etc.

By establishing lubricant trending data, operators can direct corrective maintenance actions to be performed when condemning limits or targets are exceeded. These limits and targets have to be defined in advance, which is also part of the D4378 and D6224 practices. The trending results of the collected oil analysis data will provide direction into optimizing the time frame of preventive maintenance activities and can even be combined with other techniques such as vibration analysis or wear debris analysis.

As part of today's maintenance strategies, where a return on investment is playing an important role for defending investments in maintenance equipment, people and tools, it is critical that users of ASTM practices, such as ASTM D4378 and D6224 are enabling users to take corrective actions from the analysis data and also integrate them into other maintenance programs.

How did we address these issues in the practices D4378 and D6224? By giving guidelines on data sheets recommending the analysis of selected oil properties and the intervals at which this analysis should be carried out. In addition warning limits are indicated for these properties and the measures required restoring the oil quality when these values are reached.

In the various test schedules, you will find a limiting of critical oil parameters, in combination with the following:

1. Warning limit (for example, 0.1 % of water).
2. Application (steam or gas turbine).
3. Oil life (running hours).
4. Interpretation.
    (a) Example: when higher than 0.1 % of water, the oil is contaminated, and could be from a potential water leak.
5. Action steps.
    (a) Investigate and remedy cause.
    (b) Example: improving in-service lube cleanliness through more efficient filtration, or improving cleanliness of new oil delivered to equipment or contaminant exclusion on in-service lubricants.
    (c) It may also involve decreasing the oil drains, if the oil has exceeded service limits at normal drain intervals.

It is clear that condition based oil drains or changing the oil on its condition will benefit the downtime reduction, labor reduction, longer component life from more efficient lubricant, as well oil consumption reduction.

Conclusions

- Modern Maintenance Strategies require methods of determining the condition of the lubricant in service, as an effective method of evaluating maintenance requirements for the lubricated equipment.
- As part of the ASTM D02 C00 subcommittee activities, two existing practices D4378 and D6224 have been developed and are also updated with the latest lubricants developments as well operating conditions.
- The practices recommend the analysis of selected oil properties and the intervals at which this testing should be carried out. In addition warning limits are indicated for these properties and the actions to restore the oil quality to the "fit-to-service" condition.
- Important as part of this practice is the integration of test data through trend analysis in function of properly defined condemning limits and recommended actions.
- During the last decade several original equipment manufacturers that supply power generation equipment to the industry have been integrating the ASTM practices in conjunction with existing ASTM methods as part of their Turbine Maintenance Guidelines and Procedures. OEM's today use and specify the ASTM practices as part of their maintenance procedures, in-service oil practices, in parallel with their existing turbine lube oil developments. Additionally we have also noticed how other important standardization organizations have been using the above practices as part of the in-service oil monitoring practices such as DIN VGB M410 E.

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